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BY

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Detoniruyushchii Shnur. Rus for Detonating Cord. Accdg to Gorst(1957), pp 129-30, one of the varieties contains a core of grained PETN enclosed in a cotton cloth. Another variety contains a core of mixture consisting of PETN 90-97 & lead nitrate 10-3%, to which 0.1-0.2% of red lead oxide (Pb_3O_4) is added

(Compare with info given in Ref 7, p C531-L, Vol 3 of this Encycl under Cord, Detonating)

Detonites. Brit permitted AN-NG expls such as: *Detonit V* - AN 68, NG 4, meal 2, powdered coal 4 & K or Na chloride 22%; Trauzl test 220 cc. *Detonit VI or 14A:* AN 82, NG 4, woodmeal 2, powdered coal 0.5, Nitronaphthalene 1 & NaCl 10.5%; Trauzl test 230 cc. *Detonit XIV* - AN 82, NG 4, K nitrate 10, Nitronaphthalene 2.5, & woodmeal 1.5%; Trauzl test 235 cc (Ref 1)

Cook (Ref 2) states that Detonite, a high-AN expl contg a small amt of Al pdr as a sensitizer, has been used in gas-well shooting (especially when dropped into the dry borehole to obtain a loading density of unity) and in other blasting operations

Refs: 1) Naoúm, NG(1928), 434-35 2) Cook(1958), 282

Detroit Arsenal. An Ord Corps installation, forming part of the Ordnance Tank-Automotive Command, located at Center Line, Mich. It is engaged in the development of tank-automotive materiel & supporting res with respect to armor and ground locomotion

Ref: OrdTechTerm (1962), p 98

Detoxication of Wash Waters Containing DNT.

It was discovered that fish in Picatinny Lake (Picatinny Arsenal, NJ) and its tributaries were being killed by DNT in the effluent waters from the Propellant Water-Dry House. It therefore became necessary to develop a method of detoxifying this effluent since some of the streams led to drinking water supplies. Tomlinson (Ref) developed a process for the purification of the effluent by removing the DNT. The DNT was removed by passing the wash waters thru an absorber contg activated carbon. The DNT content of such waters was reduced from 0.03 to less than 0.002 g/100 cc. It was found unsafe to remove DNT from carbon by superheated steam at temps 180-90°, and below this temp no DNT was removed. Regeneration of the carbon by extraction with solvents was found possible

By the process developed, 140 gals of wash water contg 0.03% DNT were detoxified, yielding water having a DNT content below 0.002%, by passing thru 1.0 lb of carbon. The DNT was removed from the carbon by extracting it with 0.7 gals of benzene

Ref: W.R. Tomlinson, Jr, "Detoxification of Wash Waters Obtained in the Drying of Smokeless Powder", PATR **1324** (12 Aug 1943)

Deuterium or "Heavy Hydrogen". D_2; gas, mw 4.03; fr p 13.95° K, bp 20.57° K, d of liq 169 g/liter; was first isolated in concns sufficient for positive identification by Urey et al at Columbia Univ in 1931. Deuterium is a stable isotope and occurs in natural hydrogen, water and other H-bearing compds in an av abundance of 0.015 mole %. It is of interest to research workers as a tracer in biological processes & in chem reactions. There is now commercial production of Deuterium & Heavy Water (Ref 2). It has found use in military weapons as a constituent of thermonuclear bombs

Refs: 1) Partington(1950), 176 2) Kirk & Othmer **6**, 2nd edit(1965), 895,908 (Uses)

Deuterium Oxide or "Heavy Water", D_2O; mw 20.03; fr p 3.79°, bp 101.41°, d 1.10452 at 25°, max d at temp 11.23° is 1.10602; Q_{form} -70414 cal/mol (liq), $Q_{vaporzn}$ 10846 cal/mol; G.N. Lewis of Univ of Calif concd a large quantity of water to a small amt of nearly pure D_2O by electrolysis in 1931. D_2O is now produced commercially and is used as a moderator for nuclear reactors to make plutonium. The cost of "Heavy Water" for sale by the US Govt is $24.50/lb since 1963

Refs: 1) Partington (1950), 176 2) Kirk & Othmer **6**, 2nd edit (1965), 898-909

Deuterium Peroxide, D_2O_2; mw 36.03; "heavy" hydrogen peroxide; is formed by passing "heavy steam" (D_2O vapor) thru a mixt of deuterosulfuric acid (D_2SO_4) & potassium persulfate ($K_2S_2O_8$) and condensing the vapor. The soln of D_2O_2 in D_2O thus obtained is fractionated, and pure D_2O_2 results

Refs: 1) Partington (1959), 199

Deutsche Geschossfüllung (German Shell Filler): About 100 formulations are listed in PATR **2510** (1958), pp Ger 47 to Ger 49, under FILLER OR BURSTING CHARGE (Füllung oder Füllpulver)

Devarda Metal. A grey alloy consisting of Cu 50, Al 45 & Zn 5%, which is used as a strong reducing agent in alkaline solns. Used in analysis for determining N content of nitrates & nitrites

Refs: 1) Hackh's (1944), 262 2) CondChemDict (1961), 345

Devarda Method of Analysis. The nitrogen in inorganic nitrates or nitrites can easily be determined by using Devarda metal. In this procedure a weighed sample of nitrate (or nitrite) is dissolved in w contained in a special Devarda flask, and, after making the soln strongly alkaline with NaOH, a few grams of Devarda alloy are added. This reduces NO_3^- (or NO_2^-) ion to ammonia which is distilled and titrated as in the Kjeldahl method. The method is described in Ref 1, and in a modified form (Refs 3, 4 & 5)

The method can be applied to detn of N in NC's. A weighed sample of NC is previously hydrolyzed by heating it in a flask at 50-60° with a mixture of aq KOH soln and hydrogen peroxide. After complete dissolution, the liquid, which now contains nitrate ions, is boiled to expel oxygen and then cooled. Further procedure is the same as for inorganic nitrates and nitrites

Refs: 1) Snell & Biffen (1944), 150-02 2) Clift & Fedoroff **2** (1944), p N19 3) R.O. Carter et al, "Modified Devarda Method for the Determination of Nitrogen in Nitrocellulose", OSRD Rept **3875** (July 1944) 4) NDRC Summary Rept of Div 8, Vol 1 (1946), 123 5) StdMethodsChemAnalysis **1** (1962), 748-51

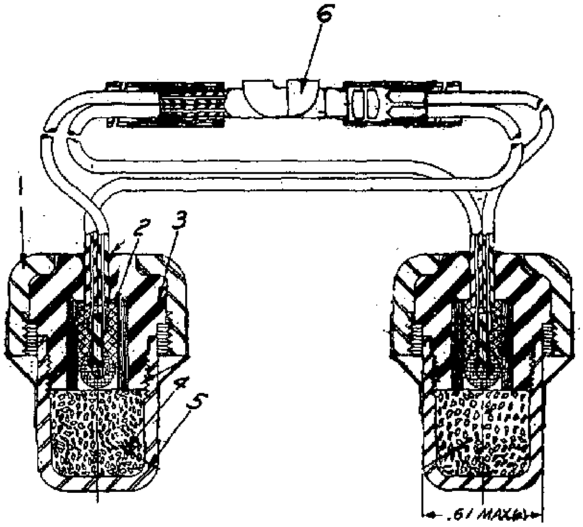
"Development of Combustion and Explosion in Liquids and Solids". Title of a paper by F.P. Bowden in the 4th Symposium on Combustion, Williams & Wilkins, Baltimore (1953), pp 161-72 (41 references)

Development of Explosion (or of Deflagration, or of Detonation). See Deflagrating Explosives and Deflagration in Vol 3, p D38-R of this Encyclopedia and under DETONATION in Volume 4

Device, Arming (Mechanical Safety). It is described by W.V. Moore, "A Subminiature Mechanical Safety and Arming Device", **TM 65-18**, Harry Diamond Laboratories, Washington, DC, 3 May, 1965

Devices for Blasting a Pass Through Wire Obstacle or Through Mine Fields. Various devices were developed during WWII for this purpose. Among them may be mentioned Detonating Cables, Demolition Hoses, Demolition Snakes and Bangalore Torpedoes described in Vol 2 of Encycl, pp B17 to B19, under BANGALORE TORPEDOES

Devices, Special for Use in the Ordnance Industry During World War II. These devices include miscellaneous items, each designed for a particular purpose only. These may



SPOILER ACTUATOR

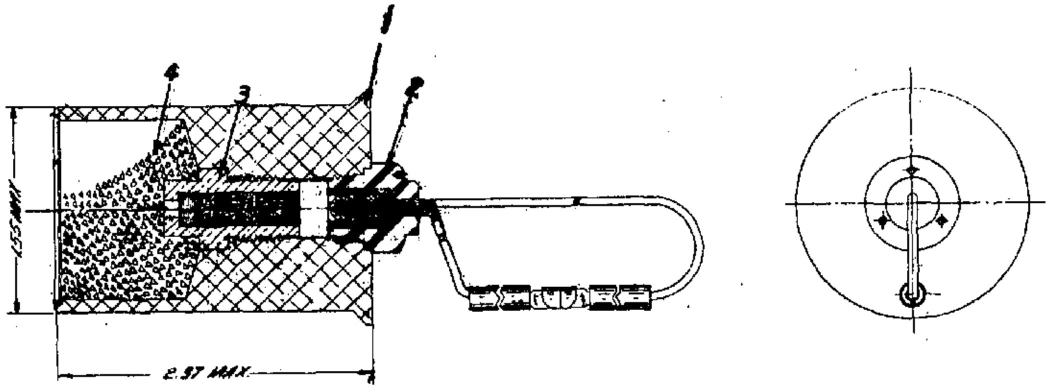
include *destructors*, described in Vol 3, because each destructor serves only for one special purpose. Most of US special devices were developed at PicArsn during WWII

Following are examples taken from the lecture of Robinson (Ref 1)

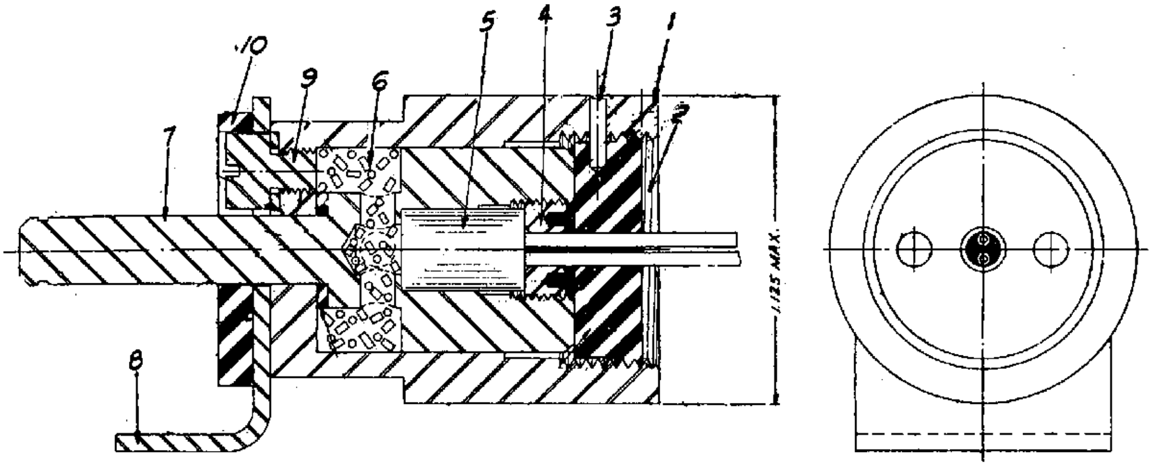
a) *Spoiler Actuator*. An explosive unit required to operate flight controls in the *JB-2 Bomb* [(which was Amer adaptation of the German Buzz Bomb, also known as V-1 (Vergeltungswaffe Eins) (See Vol 2, p B239-L)], at a pre-determined distance from the launching site so that the bomb would dive towards the target

The device developed at PicArsn was less complicated and more reliable than the original Ger device. The PicArsn device consisted of two expl units, each having a threaded cap (1), an electric squib (2), mounted inside of a plastic holder (3), a charge of BkPdr (4) and a cup (5). The leading wires of the two units were connected in parallel by means of splices (6). The latter were connected together to provide a suitable shunt for safety in handling and shipment and to facilitate easy assembly of the leading wires to the utility wires of the JB-2 Bomb. Each unit was assembled in the end of a tube on the bomb and retained in place by the threaded cap (1). In operation, the squibs ignited the BkPdr chges, the gases of which exerted sufficient force against the cups (5) to rupture the plastic holder (3) at the threads and impart a certain forward velocity to the cups. The cups traveled thru the tube and struck against a rectangular shaped steel plate with sufficient force to remove the plate from retaining a guillotine arrangement. When the plate was blown free, spring actuated knives severed the horizontal flight controls, causing the bomb to dive (Ref 1, pp 13-14)

b) An item designed for exerting a definite amount of force against a piston used on a



SPECIAL AIR CORPS DEVICE

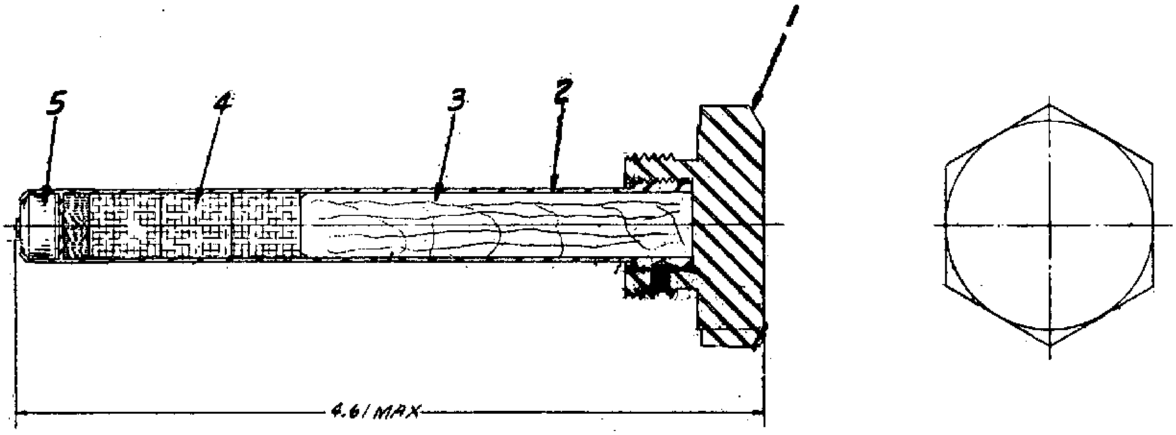


ELECTRICALLY OPERATED EXPLOSIVE PIN

classified item, consisted of a body (1), igniting device (2), delay element (3) and expl charge (4). The squib assembly ignited the delay column in the delay unit which burned thru and ignited the relay, which in turn, ignited the BkPdr. The gases produced exerted pressure on the piston. The squib assembly used in this item was identical with the one used in item(a) (Ref 1, p 14)

c) An *explosive pin* to provide a positive means of releasing large size bombs from bomb shackles, in the event of failure of the shackle to release in the normal manner, consisted of a body (1), blowout plug (2), shear pin (3), piston plug (4), squib (5), BkPdr charge (6), piston (7), clip (8), filling hole screw (9), and a rubber mounting gasket (10). The expl pin was operated by applying an electric current to the squib which ignited the BkPdr chge. The pressure produced by gases blew the piston out of the bomb shackle thus releasing the bomb lug hook which allowed the bomb to fall. The blow-out plug was made of plastic material which provided the required resistance to movement of the piston during handling and shipping of the pin, and while the pin is in use. Further, the threads of the plug readily shear when they are subjected to the shock load applied by the gases of the BkPdr chge thru the piston. This feature was important in the design of the pin since it was desired to maintain as small a release pin as possible so that it would function satisfactorily without permanently damaging the bomb shackle. Two of such expl pins were installed on each bomb shackle, with the grooved end of the piston located in the positions normally occupied by the pivot pins on which the bomb lug hooks are mounted (Ref 1, pp 14-15)

d) A special device (designed at PicArsn) was used for emergency bailing out from captured Ger twin-engine, pusher-type airplane when it was required to fly it from a temporary airfield to a permanent installation for study purposes. Parachuting from this type of plane could be made only after the propellers were blown off because of the probability of the jumper being ensnared in the propellers. The units were capable of assembling into and operated by the propeller releasing mechanism on the plane. The expl force of the units was sufficient to shear five drill rod shear pins without damaging the releasing mechanism. Each unit consisted of a plug (1) for assembly to the releasing mechanism, a tubular container (2), a wood spacer (3), a LA-Tetryl charge (4) and an electric primer (5). The contact post of the primer protruded from the tube so as to contact a spring loaded terminal in the releasing mechanism The primer was grounded thru the tube and plug. For opera-



EXPLOSIVE PROPELLER UNIT

tion of the unit, the pilot closed (prior to bailing out) a switch which functioned the electric primer (5). This initiated the expl chge (4) and the energy of detonation pushed the spacer (3) against the plug (1) and, as a result of this action the pins in the releasing mechanism were sheared, thus allowing the propeller hub to spin off the propeller shaft (Ref 1, pp 15-16)

e) *Bangalore Torpedoes and Demolition Hoses & Snakes*, for use in rapid neutralization of anti-tank mines, timberland and concrete obstacles were described in Vol 2, p B17-R (See also Ref 1, pp 16-17)

f) A special bullet impact fuze for a Bangalore Torpedo, which could be initiated by machine gun fire from the tank situated a safe distance, such as 50 ft, away was also developed. This, rather complicated, fuze is described in Ref 1, pp 17-18

g) *Reddy Fox or Water Snake*, was a device especially designed for destruction of underwater obstructions around Japanese-held islands during WWII. This was necessary in order to prevent heavy losses during landing of Amer troops, such as were caused at Tarawa Island by beach mines prior to introduction of "Reddy Fox". Because of classified nature on the snake itself at the time of the lecture of Robinson, it was not described in Ref. He described only the special fuze, known as the "T1 Demolition Fuze", which was developed at PicArsn (See Ref 1, p 20)

g) Other special devices were developed after WWII, such as the "Button Bomblet", which serves as an intrusion alarm system (Ref 2)

Some of the items already described in this Encyclopedia, may also be called "special devices", as for example, "Cartridge-Actuated Devices" (See Vol 2, pp C70-L to C72-R

Refs: 1) J.H. Robinson, "Destructors and Special Devices", Lecture delivered at PicArsn (16 May, 1947), pp 12-20   2) R.L. Wagner, D.E. Seeger & E.L. Miller, PATR **3385** (June 1966) (Conf) (Not used as a source of info)

"D" Explosive, Dunnite or Explosive "D". It is Ammonium Picrate or Ammonium Salt of 2,4,6-Trinitrophenol and is scheduled to be described under Phenol and Derivatives

Dextrin (Gommeline, Starch Gum, British Gum, Vegetable Gum or Amylin), $(C_6H_{10}O_5)_x$ mw $(162.14)_x$; mp dec, d 1.0384 at 20°. White, or sl yellow amorphous pdr; sol in w & dilute alc; insol in absolute alc or in ether. Can be prepd by the partial hydrolysis of starch, corn, potato, wheat, sago or cassava. For its prepn, any of the above products is moistened with dil nitric or hydrochloric acid and then heated to 140-170°. After the acid & w are evaporated, the dried product is powdered and sieved. It can also be prepd by careful heating of dry starch. (Refs 6, 7, 8 & 10). If starch is hydrolyzed with Bacillus macerans, some crystalline products, such as α-dextrin, $(C_6H_{10}O_5)_6$ are obtd (Ref 7). Crystallized products may also be prepd by decolorizing dextrin soln with animal charcoal and evaporating the product (Ref 10, p 346)

Dextrin is a hydrolysis product intermediate betw starches and sugars, such as dextrose (see next item). It does not give the blue iodine reaction of starch and is not fermentable, but is changed to maltose by the action of enzymes (diastase) and to dextrose by the action of acids

When mixed with w, dextrin forms a sticky glue which is used as an adhesive. It has been applied in expls industry for coating various expls to render them less sensitive. An example is "Dextrinated Lead Azide" (DLA), described in Vol 1 of this Encycl, pp A545-R to A548-R and A557-R to A563-L

Dextrin also has been and is now used as a binder in various pyrotechnic compns (Ref 11) Davis (Ref 5, pp 62ff) gives numerous examples. As a binder & a desensitizer it has been used in primer compns, as for example in a friction primer used for an airplane flare (Ref 5, p 454), or in a compn for electric primers consisting of K chlorate 88, charcoal 10 & dextrin 2% (Ref 9, p 118). As a fuel dextrin was used in some commercial expls, such as "Nobelit" & "Fördit" (Ref 2, p 407). In some gelatine expls, such as "Safety Jelly Dynamite" (Ref, p 407), a paste called "glue-dextrine-glycerine jelly" was used

Mixts of dextrin dust with air are explosive (Ref 3)

US specifications and analytical procedures for technical grade dextrin are given in MIL-D-3994(1957). It covers corn dextrin used in pyrotechnic mixtures

Requirements: Granulation - not less than 99.5% should pass thru No 80 US Std Sieve; Water, uncombined - max 5%; Ash - max 0.15% residue upon ignition; Water - insol material - max 2.5%; Acidity, as AcOH - max 0.18%; Reducing sugar as dextrose

Tests:

1) Granulation. Place 100 g sample on No 80 sieve provided with a bottom pan, cover it and shake in a mechanical shaker to produce 300±15 gyrations and 150±10 taps of a striker per min. After 5 mins stop and weigh the material in bottom pan

2) Water, uncombined. Dry a 12 g sample in a dish at 103°±3° for 4 hrs. Cool in a desiccator and weigh. Save the sample

3) Ash. Ignite in a tared silica or platinum dish at 600° a 5 g sample until the ash is white. Cool in a desiccator and weigh

4) Water-insolubles. Transfer the dried sample of procedure 2 to a 250-ml beaker, add slowly (with agitation) 100 ml of w preheated to 60°±5°. When the sample will be dissolved, filter thru a tared sintered glass crucible into 200-ml volumetric flask. Wash the residue in the crucible with sufficient amt of w to fill the flask to the mark. Save it for tests 5 & 6. Heat residue in the crucible at 103°±3° for 4 hrs, cool and weigh

5) Acidity, as AcOH. Transfer 100 ml of previous filtrate into a conical flask, add few drops of phpht indicator and titrate with 0.1N NaOH soln until a pink color is produced. Run a blank

$$\% \text{ Acidity} = [6.0N(A-B)] / C$$

where:

N = normality of NaOH soln
A = ml of NaOH soln used in the test
B = ml of NaOH soln used in the blank
C = wt of dextrin in 100 ml filtrate

6) Reducing sugars, as dextrose. Prepare Gooch crucibles with a mat of prepared asbestos ca 0.25-inch thick. For prepn of mat digest the amphibole variety of asbestos with 1:3 hydrochloric acid for 2-3 days and wash free of acid. Repeat the digestion for 2-3 days using 10% NaOH soln and then treat the asbestos for a few hrs with hot alkaline tartrate soln (See below). Then digest asbestos for several hrs with 1:3

nitric acid and wash free of acid. Agitate with water until a fine pulp is obtd

Prepare alkaline tartrate soln by dissolving 170 g of Rochelle salt and 50 g of NaOH in w, allowing to stand for 48 hrs and filtering thru the Gooch crucible, prepd as above

Prepare copper sulfate soln by dissolving 34.63 g of $CuSO_4.5H_2O$ in w, diluting to 500 ml and filtering thru Gooch crucible

Prepare potassium iodide soln by dissolving 42 g of KI in 100 ml of w and making the soln slightly alkaline to avoid the formation of hydriodic acid and its oxidation

Prepare a satd soln of bromine by agitating 2 to 3 ml of bromine with 100 ml of cold w in a bottle, closed with a glass stopper lubricated with petrolatum. Preserve in a cool dry place

Procedure. Transfer 25ml of the copper sulfate soln and 25 ml of the alkaline tartrate soln to a 400-ml beaker and add 50 ml of the filtrate saved in 200-ml flask of procedure 4. Cover the beaker with a watch glass and heat on an asbestos gauze over a Bunsen burner, regulating the flame so that boiling will begin in exactly 4 mins. Continue the boiling for exactly 2 mins and filter the hot soln at once thru the prepd Gooch crucible (See above). Wash the ppt thoroughly with w at 60°, cover the crucible with a watch glass and dissolve the residue (copper oxide) by means of 5 ml of 1:1 nitric acid introduced under the watch glass with a pipette. Collect the filtrate in a 250-ml flask and wash the watch glass and crucible free of Cu nitrate. Boil the filtrate to expel the red fumes, add a slight excess of bromine soln (See above) and boil until bromine is completely driven off. Cool, and add 25% NaOH soln with agitation until a faint turbidity of cupric hydroxide appears. Discharge the turbidity with a few drops of AcOH and add 2 drops in excess. Add 10 to 12 ml of potassium iodide soln (See above) for each 100 ml of total soln present and titrate with 0.1N Na thiosulfate soln using starch indicator. Run a blank

Calculation:

% Reducing sugars as dextrose = 100A/B and
Grams of copper reduced = 0.0636N(C-D)

where: A = g of dextrose equivalent to g of Cu reduced as obtd from table published in JACS **28**, 663 (1906) and listed in Lange and ChemRubberPubgCo Handbooks;
B = wt of dextrin in 50-ml aliquot;
C = ml of thiosulfate soln used for test;
D = ml of thiosulfate soln used for blank;
N = normality of thiosulfate soln

Refs: 1) Beil - not found 2) Naoum, NG (1928), 407 3) G.J.O. Zonnenberg, ChemZbl **31**, 722-26(1934) & CA **29**, 3162(1935) (Investigation of causes of dust explns, particularly of dextrin dust) 4) Hackh's (1944), 262 5) Davis (1943), 64 & other pp listed on p 200; also 425 & 454 6) Pérez Ara (1945), 355 7) Karrer (1947), 350 8) Ott, **5**, Part 1 (1954), 46 9) TM **9-1910**(1955), 94 & 118 10) CondChemDict (1961), 345-46 11) AMC Pamphlet **706-187**, "Properties of Materials Used in Pyrotechnic Compositions", pp 114-15, Dextrin (Oct 1963)

Dextrin Nitrate or Nitrodextrin. Composition could not be found and it is not listed in Beil; N content 13.90 - 14.01 (Ref 4). Daniel (Ref 2, pp 5 & 522) stated that A.Nobel patented it in 1891 as an ingredient of some smokeless proplnts. It is a substance resembling in appearance and properties *Nitrostarch* and can be prepd by nitrating dextrin with mixed concd nitric-sulfuric acid, as described in Ref 2, p 457 and in Ref 4

Perrott et al (Ref 3) proposed to use it for sensitization of AN crysts

It has been used in Belgian expls *Casteau*, described in Ref 2, p 117 and this Encycl, Vol 2, p C83-R

Refs: 1) Beil - not found 2) Daniel (1902), 5, 117, 457 & 522 3) G.St.J. Perrott et al, USBurMines RI **2987**(1930) & CA **24**, 1983 (1930) 4) G.V. Caesar & M. Goldfrank, JACS **68**, 374(1946) & CA **40**, 3634(1946) (Nitration of dextrin & starches with N_2O_5 in presence of Na fluoride) 5) G.V. Caesar et al, JACS **69**, 617-21(1947) & CA **41**, 3680(1947) (MW detns & intrinsic viscosities of nitric triesters of dextrins & corn starches)

Dextrose, d-Glucose, Dextroglucose, Corn Sugar or Grape Sugar, $HOCH_2.(CHOH)_4.CHO$; mw 180.16; col crysts, d 1.544 at 25°, mp 146°(anh); in Ref 2, d is 1.562 & mp 147°; v sol in w, sl sol in alc, insol in eth. It is a monosaccharide or carbohydrate constituent of many animal and vegetable fluids (especially sweet fruits). Can be obtd by incomplete hydrolysis of starch, cane sugar and glucosides. Used as a reagent, as a reducing agent (such as for Fehling's soln), for production of beer and alcoholic liquors and pharmaceuticals

Can be nitrated to produce an expl pentanitrate

Refs: 1) Beil **31**, 879 2) Hackh's (1944), 379-80 3) CondChemDict (1961), 346 (Dextrose) 4) Kirk & Othmer **6**, 2nd edit (1965), 919

Dextrose- or d-Glucose Pentanitrate, known also as **Nitroglucose,** $(C_6H_7O(ONO_2)_5$; mw 405.14, N 17.35% (theory); 16.96 (found); col viscous liq, which hardens at 0° to an amorphous mass; it is unstable above 50° and decomp rapidly at 135°; insol in w & ligroin; sol in alc; reduces Fehling's soln on warming. It can be prepd, accdg to Daniel (Ref 2) by nitrating glucose with equal parts of concd nitric and sulfuric acid; its expl props are weak

Nitroglucose was used as an ingredient of some expls, such as *Ammonia Nitrate Powder* (AN 80, Nitroglucose 10, K chlorate 5 & coal tar 5%) (Ref 2, p 24) and in Keil (Explosif) (Mixt of Nitroglucose with K nitrate, K chlorate and vegetable fibers) (Ref 2, p 391)

Refs: 1) Beil **31**, 146 2) Daniel (1902), 523 (Nitroglucose) 3) Davis (1943), 241

Dextrose Polymers and Their Nitrates. Dextrose (d-glucose) polymers can be obtd by heating dextrose or polysaccharides composed of dextrose units, in the presence of catalysts, such as boric anhydride, or boric acid. The resulting solid products are light in color and have a high & wide degree of polymerization. By composition they are synthetic glucose polyanhydrides (Ref 1)

Power (Ref 2) nitrated some of these polymers and recommended their use as base chges in detonators. The best results were claimed to be obtd with polymers having an average of 7 to 10 glucose units in the molecule. Nitration was conducted by adding slowly, with stirring, over a period of 7 mins, 10 g dry powdered dextrose polymer to 175 g of mixed acid (HNO_3 35, H_2SO_4 58 & H_2O 7%). The temp of nitration was 23°. After stirring for addnl 10 mins the slurry was poured into 1 liter of cold w and the resulting ppt transferred into a Büchner funnel. After applying suction the nitrate was washed with two portions of w at 70-80°, then dried and dissolved, with stirring, in 25 ml acetone contg 1 g of Ca hydroxide. After filtering, the clear soln was fed, with vigorous agitation, into 500 ml of w at RT, and the ppt collected in a Büchner funnel. The dried product was about 12.6 g. Its nitrogen content was not specified in abstract of patent (Ref 2)

Refs: 1) G.J. Leuck, USP 2375564 (1945) & CA **39**, 4508 (1945) 2) J.D. Power, USP 2495868 (1950) & CA **44**, 7540 (1950)

DGO (Propellant). A modified double-base proplnt, developed by ADL, for rocket motors. Its compn and props are given as Unit No 598 in conf Proplnt Manual SPIA/M2 (1962)

DGV or **DDP(70).** A modified double-base proplnt, developed by ADL, for rocket motors. Its compn and props are given as Unit No 634 in conf Proplnt Manual SPIA/M2 (1962)

A Hercules cast rocket proplnt by the same description is described in conf Propellant Manual SPIA/M2 (1969), Unit No 1138

DI or Di. Ger abbr for DNT

Di-. A prefix meaning two or twice. A compd not found under di- should be looked for under bi- or bis-; since bi-, bis- & di- are nearly equiv prefixes, assigned with sl differences in meaning for particular compds, or accordg to customary usage
Ref: CondChemDict(1961), p346

Diacetaldehyde Peroxide. See Diethylidene Diperoxide

Diacetato(or Oxalato)-plumbo Complexes. The following complexes, some of them expl, were prepd by Weinland & Paul (Ref 1) and also studied by others (Refs 2, 3 & 4): *Diacetato-diplumbo-bromate*, $(Pb_2ac_2)(BrO_3)_2$; mw 788.35, O 20.29%; thin lfts, mp deflgr at 165±5°. Weinland & Paul (Ref 1) observed that during the prepn of lead bromate, $Pb(BrO_3)_2.H_2O$, from lead acetate & potassium bromate in the presence of acetic acid there was formed an extremely expl complex salt. Günzel & Marcus (Ref 2) obtd the same complex and gave a method for its prepn. They also reported this expl to be very sensitive to impact & friction, and so powerful that even 50 mg was sufficient to shatter a strong glass vessel. See also Victor (Ref 3)
Diacetato-diplumbo-picrate,
$(Pb_2ac_2)[C_6H_2(NO_2)_3O]_2.H_2O$; mw 974.73, N 8.62%; lemon-yel prisms, deflgr on heating
Diacetato-triplumbo-nitrate, $(Pb_2ac_2)(NO_3)_4$; mw 987.75, N 5.67%; granular cryst aggregates
Oxalato-diplumbo-perchlorate,
$(Pb_2ox)(ClO_4)_2.3H_2O$; mw 755.40, O 31.77%; deflgr weakly (Ref 1)
Oxalato-diplumbo-nitrate,
$(Pb_2ox)(NO_3)_2.2H_2O$; mw 662.50, N 4.23%; leaflets
Trioxalato-tetraplumbo-perchlorate,
$(Pb_4ox_3)(ClO_4)_2.5H_2O$; mw 1381.90, O 28.94%; six-sided prisms

NOTE: ac stands for CH_3COO- and ox stands for -O.OC.CO.O-

Refs: 1) R. Weinland & F. Paul, ZAnorgChem **129**, 243(1923) & CA **17**, 3293(1923)
2) E. Günzel & E. Marcus, ZAnorg Chem **38**, 929(1925) & CA **20**, 718(1926) 3) DipIng Victor, ZAngChem **40**, 841(1927) & CA **21**, 3324(1927) 4) Dr Eibel, SS **22**, 221(1927) & CA (Not found)

Diacetin. See under Acetins, Vol 1, p A31-R

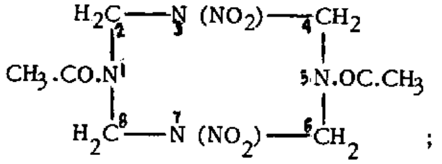
Diacetone; Diacetone Alcohol; 4-Hydroxy-4-methyl-pentanone-2; or 4-Hydroxy-2-keto-4-methylpentane [called Diacetonalkohol; Dimethyl-acetonyl-carbinol; 2-Methyl-pentanol-(2)-on-(4); or Methyl-(β-oxy-isobutyl)-keton in Ger], $CH_3COCH_2C(CH_3)_2OH$; mw116.16, col, pleasant-odored liq, fr p -42.8°, bp 169° at 760mm, flash p 170°F, d 0.9406 at 20°, vapor pressure 1.1mm at 20°, viscosity 0.032 poise at 20°, n_D 1.42416 at 20°, coeff of expansion 0.00097 at 20 . Diacetone is derived by condensation of acetone. It is miscible with alcohols, aromatic & halogenated hydrocarbons, esters & water. A const boiling mixt with water has a bp 99.6° and contains approx 13% diacetone (Refs 1, 3, 4 & 5)

Diacetone is used as a solv for NC, cellulose acetate, oils, resins, waxes, fats, dyes, tars, lacquers, etc. Fassnacht (Ref 2) patented the use of diacetone or other aliphatic monohydric alc contg a carbonyl group, as an additive to NG, which acts as an accelerant in the gelatinization of NC
Refs: 1) Beil **1**, 836, (424), [876] & {3234}
2) H.H. Fassnacht, USP 2194544(1940) & CA **34**, 4908(1940) 3) Kirk & Othmer 1(1947), 93; **8**(1952), 117 & 134-36; **10**(1953), 205 and **12**(1954), 668, 670, 682, & 729 4) Sax(1957), 538-39 5) CondChemDict(1961), 347

Diacetone Diperoxide. See Acetoneperoxide, Dimeric or Diacetone Diperoxide in Vol 1, p A41-R

Diacetophenone Diperoxide. See Acetophenoneperoxide, Dimeric in Vol 1, p A48-R

Diacetotetrazacycloöctane, $C_8H_{16}N_4O_2$, may be considered the parent compd of its dinitro deriv but is not used to prepare it: *1, 5-Diaceto-3, 7-dinitro-1, 3, 5, 7-tetrazacycloöctane,*

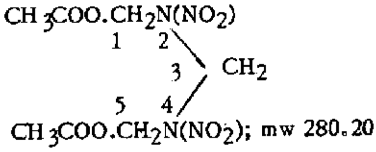


mw 290.24, N 28.96%, crysts (from boiling nitromethane), mp 265-66°; was obtd as one of the products of hexamine nitrolysis, but more specifically by treating of the 3-nitroso-7-nitroderiv with HNO_3 & $(NH_4)_2SO_4$ at 2-3°. The dinitroderiv was converted in 25% yield to HMX by overnight treatment with 99% HNO_3

Ref: E.E. Aristoff et al, CanJRes **27 B**, 539-40(1949) & CA **43**, 9075(1949)

1, 9-Diacetoxy-2-(4-)acetyl-4-(2-), 6, 8-trinitro-2, 4, 6, 8-tetrazanonane. See 2-(4-) Acetyl-1, 9-diacetoxy-4-(2-), 6, 8-trinitro-2, 4, 6, 8-tetrazanonane in Vol 1, p A57-R

Diacetoxy-diazapentane, $C_7H_{14}N_2O_4$, may be considered the parent compd for its dinitro deriv but is not used to prepare it: *1, 5-Diacetoxy-2, 4-dinitro-2, 4-diazapentane* or *1, 5-Dicetoxy-1, 3, 5-trimethylene-2, 4-dinitramine,*



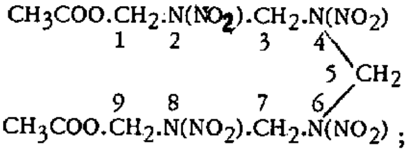
mw 280.20, N 20.00%, crysts (from acet + alc), mp 90-92° & 103-04°, was obtd from a mixt of methylenedinitramine in formalin, anhyd Na acetate & aceticanhydride warmed to 95° for 1 hr, drowning in ice water & neutralizing with NH_3. This compd could not be detonated by striking with a hammer, but it burst into flame on being heated on a spatula and burned like cordite (Refs 1 & 2)

Ref: 1) E.E. Aristoff et al, CanJRes **27B**, 533(1949) & CA **43**, 9075(1949) 2) D. Woodcock, JCS **1949**, 1635-36(1949) & CA**44**, 1411(1950)

1, 9-Diacetoxy-1, 3, 5, 7, 9-pentamethylene-2, 4, 6, 8-tetranitramine. See under Diacetoxy-tetrazanone

1, 7-Diacetoxy-1, 3, 5, 7-tetramethylene-2, 4, 6-trinitramine. See under Diacetoxy-triazaheptane

Diacetoxy-tetrazanonane, $C_9H_{20}N_4O_8$, may be considered the parent compd for its tetranitro deriv but is not used to prepare it: *1, 9-Dacetoxy-2, 4, 6, 8-tetranitro-2, 4, 6, 8-tetrazanonane* or *1, 9-Diacetoxy-1, 3, 5, 7, 9-pentamethylene-2, 4, 6, 8-tetranitramine* (Code name **AcAn**),



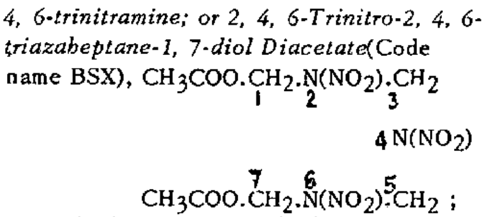
col crysts (from NM or acetone), mp 186.5-187.2°; was prepd by reaction of DPT (1, 5-*endo*methylene-3, 7-dinitro-1, 3, 5, 7-tetrazacycloöctane), acetic anhydride & 99.6% HNO_3 over 40min at 44°. This compd can also be prepd from 1, 9-dinitroxy-2, 4, 6, 8-tetranitro-2, 4, 6, 8-tetrazanonane and Na acetate in acetic acid. It is destroyed by 1 hr boiling with aq NH_3 (Ref 1). Other props & methods of prepn are given in Refs 2 to 7

This compd gives on nitration a powerful expl 1, 9-Dinitroxy-2, 4, 6, 8-tetranitro-2, 4,-6, 8-tetrazanonane (qv)

Refs: 1) A.F.McKay et al, CanJRes **27B**, 465 -66(1949) & CA **43**, 9073(1949) 1a) HACISR, BritP 615793(1949), pp 1-3 (Prepn & props) 2) R.N. Jones & G.D. Thom CanJRes **27B**, 832, 835, 857(1949) & CA **44**, 2848(1950)(UV absorption spectra) 3) W.E. Bachmann et al, JACS **73**, 2772, 2774(1951) & CA **46**, 2084(1952)(Prepn & props) 4) W.E. Bachmann & E.L. Jenner, JACS **73**, 2774(1951) & CA **46**, 2085(1952) 5) E.W. Malmberg et al, AnalChem **25**, 901(1953) & CA **47**, 12095(1953)(chromatographic props) 6) R.A. Marcus & C.A. Winkler, CanJChem **31**, 602-14(1953) & CA **47**, 12219(1953)(Prepn & reaction mechanism)

7) G.F. Wright & W.J. Chute, USP 2678927 (1954) &CA **49**, 7606-07(1955)(Prepn) 8) H.J. Matsuguma et al, PATR **2442**(July 1957)(Conf)

Diacetoxytriazaheptane, $C_8H_{17}N_3O_4$, may be considered the parent compd of its trinitro deriv but is not used to prepare it: *1,7-Diacetoxy-2,4,6-trinitro-2,4,6-triazaheptane; 1, 7-Diacetoxy-1, 3, 5, 7-tetramethylene-2, 4, 6-trinitramine; or 2, 4, 6-Trinitro-2, 4, 6-triazaheptane-1, 7-diol Diacetate*(Code name BSX),



$$\begin{array}{l} CH_3COO.\underset{1}{C}H_2.\underset{2}{N}(NO_2).\underset{3}{C}H_2 \\ \qquad\qquad\qquad 4\,N(NO_2) \\ CH_3COO.\overset{7}{C}H_2.\overset{6}{N}(NO_2).\overset{5}{C}H_2 \end{array}$$

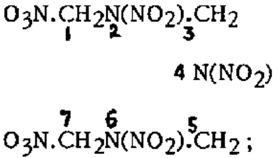
mw 354.24, N 23.73%; col plates (from CCl_4) mp 154-55°, was obtd as one of the products of nitrolysis of hexamine. It can be prepd by treating an acetic acid soln of hexamine with a mixt of HNO_3, AN & acetic anhydride in the cold (rather than at 75° when RDX is formed). BSX may also be prepd under the same conditions w/o the use of AN (Refs 1, 2, 3, 4, 6, 7, 8, & 9). Other props of BSX are given in Refs 5, 10 & 11)

BSX is an expl compd about 75% as powerful as TNT and slightly less sensitive

It can be nitrated to give a *dinitroxy deriv* which is a very powerful & very sensitive expl

Refs: 1) W.E. Bachmann, "Studies on the Preparation and Properties of RDX", OSRD **820**(Aug 1942)(Structure of HMX & BSX 2) W.E. Bachmann, "Studies on RDX(B) and Related Compounds", OSRD **5186**(June 1945) p 156 3) Anon, Summary Technical Report of Division 8, NDRC, "The Preparation and Testing of Explosives", Vol 1(1946), pp 8 & 153 4) W.J. Chute et al, CanJRes **27B**, 511, 514-15 (1949) & CA **43**, 9074(1949) 5) R.N. Jones & G.D. Thorn, CanJRes **27B**, 832, 834(1949) & CA **44**, 2848(1950)(UV spectra) 6) W.E. Bachmann & J.C. Sheehan, JACS **71**, 1842-45(1949) & CA **43**, 8137(1949) 7) K.W. Dunning, JCS **1950**, 2925-32 & CA 45, 6644(1951) 8) W.E. Bachmann et al, JACS **73**, 2772(1951) & CA **46**, 2084(1952) 9) A.D. Little, Synthesis HE's 2nd Rept (March 1951), pp 58-59 10) E.W. Malmberg et al, AnalChem **25**, 901(1953) & CA **47**, 12095 (1953)(Chromato-graphic props) 11) H.J. Marsuguma et al, "Behavior of Nitramide and Linear Polynitramines as Intermediates in the Synthesis of HMX", PATR **2442** (July 1957) (Conf, not used as a source of info) 12) Urbański 3(1967), 91, 114

Dinitroxy-BSX or 1, 7-Dinitroxy-2, 4, 6-trinitro-2, 4, 6-triazaheptane,



$$\begin{array}{l} O_3N.\underset{1}{C}H_2\underset{2}{N}(NO_2).\underset{3}{C}H_2 \\ \qquad\qquad 4\,N(NO_2) \\ O_3N.\overset{7}{C}H_2\overset{6}{N}(NO_2).\overset{5}{C}H_2\,; \end{array}$$

mw 360.16, N 31.12%; crysts (1/1:dioxane-CCl_4), mp 153-54°; was prepd by nitrating BSX with 99% HNO_3 at 0°, one hr at 20°, and drowning the soln in water. It was obtd also by nitrating 1-acetoxy-7-ethoxy-2, 4,6-trinitro-2, 4, 6-triazaheptane with 99% HNO_3 at 0°, 20min at 20°, and drowning in water (Refs 1, 2 & 3)

Dinitroxy-BSX is a powerful expl which is very sensitive to impact when pure(Ref 1)

Other props are given in Refs 4 & 5

Refs: 1) W.J. Chute et al, CanJRes **27B**, 513-14(1949) & CA**43**, 9074(1949) 2) K.W. Dunning & W.J. Dunning, JCS **1950**, 2925-28 & CA **45**, 6643(1951) 3) A.D. Little, Synthesis HE's 2nd Rept (March 1951), pp 58-59 4) E.W. Malmberg et al, AnalChem **25**, 901(1953) & CA **47**, 12095(1953) (Chromato-graphic props) 5) R. Reed, Jr, JACS **78**, 801(1956) & CA **50**, 12864(1956)

1, 9-Diacetoxy-1, 3, 5-trimethylene-2, 4-dinitramine. See under Diacetoxy-diazapentane

Diacetylaminophentetrol, $C_{10}H_{11}NO_6$, may be considered the parent compd of its mononitro deriv but is not used to prepare it: *Diacetylnitroaminophentetrol or 3-Amino-6-nitro-1, 2, 4, 5-benzenetetrol Diacetyl Deriv,* $(CH_3CO.O)_2C_6(OH)_2NH_2(NO_2)$; mw 286.20, N 9.79%; lt yel ndls (from alc), mp darkens at 208-10° & deflgr at 214°; was prepd by treating nitraminophentetrol

with water & acetic anyydride
Refs: 1) Beil-not found 2) G. Heller et al, JPraktChem **129**, 252-53(1931) & CA **25**, 2129(1931)

Diacetylaminophenol, $C_{10}H_{11}NO_3$, may be considered as the parent compd of its tetranitro deriv but is not used to prepare it: *X-Tetranitrodiacetylaminophenol,* $(O_2N)_4.C_6(OH).N(OC.CH_3)_2$; mw 373.56, N 18.85%; col short ndls (from benz or petr eth), mp 147-47.5°; was obtd when benzene-free pentanitroaniline was boiled with acetic anhydride & two drops of H_2SO_4, followed by cooling, removing the excess anhydride, and mixing with snow (Refs 1 & 2)
Refs: Beil **12**, [429] 2) B. Flürscheim & E.L. Holmes, JChemSoc **1928**, 3045-46 & CA **23**, 823(1929)

Diacetylene or Butadiyne (called Diacetylen or Butadiin in Ger), $HC{\vdots}C.C{\vdots}CH$; mw 50.06; gas at RT having a pronounced odor, forms lfts on cooling with liq air, fr p -36°; bp 10° at 760mm press, d 0.7364 at 0°, n_D 1.4386 at 0.8°; can be prepd by the action of cupric chloride soln on cuprous acetylide, followed by treatment of the Cu diacetylide, thus formed, with dil HCl; and by action of excess alcoholic KOH at 150-60° on butadiene tetrabromide; and by other methods (Refs 1 & 2)

The critical pressure of expln of Diacetylene is 30-33mm; the critical concn (by vol %) of it in mixts at 700mm with other gases is 22-25% with N, 37% with NH_3, 16.5-17% with acetylene, 39% with natural gas, 35% with CO_2, 30% with CO, and 30% with H(Ref 4)

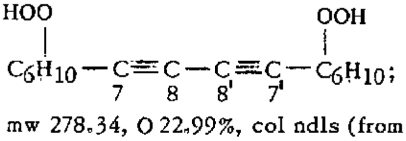
It readily yields expl metallic derivs, such as *Silver salts,* Ag_2C_4, citron-yel ppt, very expl; and $AgC_4H+AgNO_3$, citron-yel ppt, explodes by friction; and with iodine in KI it forms the very expl compd *Diiododiacetylene* (qv)(Ref 1). See also Ref 3
Refs: 1) Beil **1**, 266, [245] & {1056}
2) Thorpe **3**(1939), 571 3) Sax(1963), 665
4) R.Ya. Mushii et al, KhimProm **1963** (2), 109-11 & CA **59**, 7309(1963)

Diacetylenedicarbonic Acid or Hexadiynedioic Acid (called Hexadiindisäure; Butadiindicarbonsäure or Diacetylen-dicarbonsäure in Ger), $HOOC.C{\vdots}C.C{\vdots}C.COOH$; mw 138.08, O 46.35%; rhombic tablets (from eth + petr eth) with $1H_2O$, mp darkened at 100°, exploded at 177°, changed in light to purple mass; was prepd by oxidation of the Cu compd of propiolic acid with alkaline ferricyanide (Refs 1 & 3)

Dunitz & Robertson (Refs 2 & 4) prepd the compd after the method of Baeyer (Ref 3) and obtd the *Dihydrate,* It yel monoclinic ndls (from w or aq eth & petr eth), mp darkened at 95-100°, decompd on further heating w/o expln. The crysts became purple on exposure to light and almost black on exposure to X-rays (Refs 2 & 4)

Other props are given in Beil (Ref 2)
Refs: 1) Beil **2**, 809-10 2) Beil **2**, {2013}
3) A. Baeyer, Ber **18**, 676, 2270(1885)
4) J.D. Dunitz & J.M. Robertson, JChemSoc **1947**, 1146, 1153

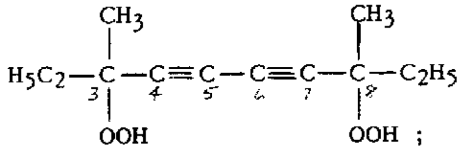
Diacetylenic Dihydroperoxides. Organic peroxides which contain two acetylenic groups in conjugation with two hydroperoxy groups in the same molecule had not been previously prepd. Milas & Mageli synthesized four of these peroxides and detd some of their props, including IR spectra. These compds are relatively stable at RT, but they explode on heating with great violence. They are sensitive to initiation by shock and, when detonated by a blasting cap, explode with tremendous brisance:
7, 7'-Dicyclohexyl-7, 7'-butadiene-6, 6'-dihydroperoxide,



$$\underset{}{\overset{HOO}{\overset{|}{C_6H_{10}}}}-\underset{7}{C}\equiv\underset{8}{C}-\underset{8'}{C}\equiv\underset{7'}{C}-\overset{OOH}{\overset{|}{C_6H_{10}}};$$

mw 278.34, O 22.99%, col ndls (from benzene + pentane), mp 96-97° dec; was prepd by reacting in the cold (0°) H_2O_2, H_2SO_4, benzene & anhydrous ether, adding 7, 7'-dicyclohexyl-7,7'-butadiyndiol-6,6 (mp 173°) and allowing the temp to rise to 25°

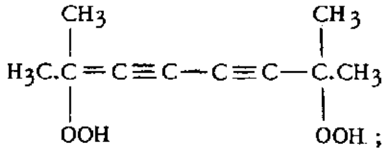
3, 8-Dimethyl-4, 6-decadiyn-3, 8-dihydroperoxide,



$$H_5C_2-\underset{OOH}{\overset{CH_3}{C}}-C\equiv C-C\equiv C-\underset{OOH}{\overset{CH_3}{C}}-C_2H_5$$

; mw 226.26, O 28.29%; col crysts, mp 44-45.5°; was obtd by adding to the above mixt (at5-10°) 3, 8-dimethyl-4, 6-decadiyn-3, 8-diol (mp 73-76°) in a single portion and allowing the temp to rise to 25° during 1hr while stirring. The yel oil crystd after standing 2-3 days at 4°

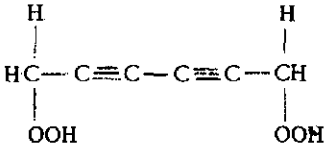
2, 7-Dimethyl-3, 5-octadiyn-2, 7-dihydroperoxide,



$$H_3C.\underset{OOH}{\overset{CH_3}{C}}=C\equiv C-C\equiv C-\underset{OOH}{\overset{CH_3}{C}}.CH_3$$

; mw 198.21, O 32.29%; crysts (from w + alc), mp 95-96.5°; was prepd by adding in small portions 2, 7-dimethyl-3, 5-octadiyn-2, 7-diol (mp 131°) to a cold (0°) soln of H_2O_2 & H_2SO_4

2, 4-Hexadiyn-1, 6-dihydroperoxide,



$$H\underset{OOH}{\overset{H}{C}}-C\equiv C-C\equiv C-\underset{OOH}{\overset{H}{C}}H$$

; mw 142.11, O 45.04%; gum, diffc to purify; was obtd by adding 2, 4-hexadiyndiol-1, 6 (mp 112-13°) in small portions to a cold (0°) well-stirred mixt of H_2O_2 & H_2SO_4. Attempts to purify the viscous oily product failed because of its instability

Ref: N.A. Milas & O.J. Mageli, JACS **75** 5970-71(1953) & CA**49**, 8090(1955)

Diacetyl-orthonitric Acid (called Diacetyl-ortho-salpetersäure in Ger), $(CH_3COO)_2N(OH)_3$; mw 183.12, N 7.65%; col liq, bp 45° at 15mm, d 1.189 at 23°, n_D^{23} 1.37542; formed by the action of concd HNO_3 on acetic anhydride or by the action of acetic acid on fuming HNO_3. It is decompd by water into acetic & nitric acids

Diacetyl-orthonitric acid reacts with acetic anhydride to form tetranitromethane, [$C(NO_2)_4$ or $(NO_2)_3C.O.NO$]; and with acetanilide to give mainly o-nitroacetanilide, $O_2NC_6H_4NHCOCH_3$

It has found use as a nitrating agent. A. Pictet used it to prep *Tetranitroacetyldiphenylamine* (See Vol 1, p A58)

Its Ag salt, called *Diargentic Acetonitrate,* $(AgO)_2N(OH)(OOC.CH_3)$, col crysts, explodes about 172°, was prepd by dissolving $AgNO_3$ in glacial AcOH

Refs: 1) Beil **2**, 271, [175] & {382} 2) G. Bacharach & W. Breckstone, Ber **64B**, 2136(1931) & CA **26**, 110(1932) 3) Sidgwick, OrgChem of N(1937), 12 4) B. Reichert & W. Turkewitsch, ArchPharm **276**, 397 (1938) & CA **33**, 1336(1939)(Reactions of diacetylorthonitric acid) 5) A.S. Naúmova, ZhurObshKhim **19**, 1228(1949) & CA **44**, 2359(1950)(Elec conductivity & viscosity of the system acetic acid-nitric acid)

Diacetyl Peroxide or Acetyl Peroxide (called Diacetylperoxyd or Acetylperoxyd in Ger), $CH_3CO.O.O.COCH_3$; mw 118.09, O 54.20%; col ndls (from ether), mp 30°, bp 63 at 21mm, flash p (OC) 113°F; explodes on further heating & on shock; sol in alc & eth; sl sol in w; can be prepd by action of acetic anhydride or acetyl chloride on Na_2O_2, BaO_2 or H_2O_2 in ethereal soln. A relatively safe procedure for its prepn is given by Shanley (Ref 8). Its toxicity, fire hazard, expln hazard & shipping regulations are discussed in Refs 14, 15 & 17

Diacetyl peroxide is an extremely sensitive & powerful compd, exploding even in storage at RT. Kuhn (Ref 6) and Shanley (Ref 7) warn about the danger involved during its prepn and report a shattering expln of a small sample. Dr M. Szwarc (Ref 16) reports that rigorous removal of traces of acid stabilizes acetyl peroxide and reduces the danger of expln

Solid acetyl peroxide is not accepted for shipping but its 25% soln in dimethyl phthlate, the commercial product which serves as an oxidizer, is acceptable for shipment by commercial carriers (Refs 14, 15 & 17)

Refs: 1) Beil **2**, 170,(78), [174] & {380} 1a) Pepin Lehalleur(1935), 137 2) O.J. Walker & G.L.E. Wild, JCS **1937**, 1132-36 & CA **31**, 8508-09(1937)(Thermal & photochem decompn of acetyl peroxide) 3) L.F. Fieser & A.E. Oxford, JACS **64**, 2060-65(1942)(Prepn of diacetyl peroxide and the alkylation of para quinones) 4) M.S. Kharasch et al, JACS **65**, 15-17(1943) & CA **37**, 1385(1943)(Decompn of acetyl peroxide in aliphatic acids) 5) M.S. Kharasch et al, JOC **10**, 386-93(1945) & CA **40**, 1783(1946)(Decompn of acetyl peroxide and other peroxides in aliphatic acids & substituted aliphatic esters) 6) L.P. Kuhn, C & EN **26**, 3197(1948) & CA **43**, 405(1949) (Expln of acetyl peroxide) 7) E.S. Shanley, C & EN **27**, 175(1949) & CA **43**, 3199(1949) (Warning as to expln hazard of acetyl peroxide with recommended precautions) 8) E.S. Shanley, JACS **72**, 1419(1950)(Safe method for isolation of acetyl peroxide) 9) M.S. Kharasch et al, JOC **16**, 533, 905 (1951) & CA**46**, 1484-85(1952)(Expln of acetyl peroxide, and its decompn in alcohols) 10) Kirk & Othmer **10**(1953), 73 11) A.Krug & J. Sixt, GerP 764109(1954) & CA **50**, 12100(1956)(Prepn) 12) Tobolsky & Mesrobian(1954), 39-40, 169 & 179 13) C. Luner & M. Szwarc, JChemPhys **23**, 1978-79(1955) & CA **50**, 2302-03(1956)(Photosensitized decompn of acetyl peroxide) 14) CondChemDict(1956), 13 15) Sax(1957), 240 16) Anon, C & EN **38**, 47(1960)(Danger in handling acetyl peroxide) 17) CondChem Dict(1961), 13 18) E.G.E. Hawkins, "Organic Peroxides", Van Nostrand, Princeton NJ(1961), 300ff

Dialkylacetylenes. A convenient method for the prepn of dialkylacetylenes, $RC\equiv CR$, is described in which the process involves interaction of sodium acetylide, sodamide & alkyl halides in liq ammonia. Intermediate products are not isolated, thereby saving substantial time as compared with older methods. The following pure compds were prepd:

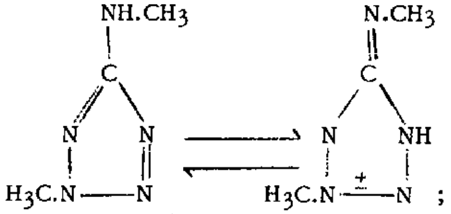
Compound	°C bp	mm	d at 25°	n_D^{25}
Diethylacetylene	74.5	600	0.7263	1.4112
Dipropylacetylene	116.7	498	0.7474	1.4225
Dibutylacetylene	106.3	80	0.7673	1.4311
Diamylacetylene	110	23	0.7816	1.4374
Ethylbutylacetylene	118.5	498	0.7501	1.4230

Details of the methods of prepn are given in the Ref

Ref: E.A. Bried & G.F. Hennion, JACS **59**, 1310-11(1937)

Dialkyliminotetrazoles (Dimethyliminotetrazoles). A new class of tetrazole derivs has been reported of which the following are examples:

1, 3-Dimethyl-5-iminotetrazole,



mw 113.13, N 61.91%; hygr crysts (from benz, and methylene chloride-petr eth), mp 41-43°, bp 88° at 1mm; was obtd by dissolving the hydrochloride (mp 202-05°) deriv in boiling methanol and a soln of methanol in 97% NaOH, evaporating to dryness and extracting the residue with chlf. Also prepd were the *Picrate*, $C_9H_{10}N_8O_7$, crysts (from alc), mp 186-87°; *Hydrobromide*, $C_3H_8N_5Br$, crysts (from 98% isopropyl alc), mp decompd 188.5-89°; and *Nitrate*, $C_3H_8N_6O_3$, crysts (from alc), mp 153.5-54.5° (Refs 1 & 2)

1, 4-Dimethyl-5-iminotetrazole, crysts (from benz), mp 108.5-09.5°; was obtd by saturating with anhyd K carbonate the aq filtrate from the HCl salt resulting from the methylation of 1-methyl-5-aminotetrazole. Also prepd were its *Picrate*, fine ndls (from 95% alc), mp 211.5-12.5° dec; *Hydrochloride*, long thin ndls (from 90% isopropyl alc), mp 242-44° dec; and *Hydrobromide*, crysts (from abs alc), mp dec 190-91° (Ref 2)

Refs: Beil-not found 2) J.H. Bryden et al, JACS **75**, 4863(1953) & CA **49**, 6242(1955) 3) R.A. Henry et al, JACS **76**, 2894-8(1954) & CA **49**, 10274(1955)

Dialkylphthalates, Determination in NG Propellants. A boiling soln of 20% NaOH does not saponify dialkyl-phthalates; they can be recovered with a loss of only 3-4% by extraction with ether, and this loss can be corrected for experimentally. However, a warm alc soln of NaOH saponifies dialkylphthalates completely. These facts form the basis for an analysis of proplnts contg dibutyl- or diamyl-phthalates, centralite & possibly phthalide

Procedure: Extract a sample of proplnt with ether and weigh the NC residue. Treat another sample with 20% aq NaOH soln and extract with ether. Save the aq layer (1), which contains any Na hydroxymethylbenzoate. (2). Dry the ether extract, which contains phthalates & centralites, and weigh the residue. Treat this mixed residue with hot alc NaOH to saponify the phthalates. Add water to dissolve Na phthalate. Extract centralite from the liq in the usual manner and weigh. Determine phthalate by difference. Acidify (1) with 50% H_2SO_4 and heat to reconvert (2) to phthalide. Extract the phthalide with ether. Determine NG present by difference

Ref: M. Tonegutti & E. Brandimarte, AnnChimAppliata **29**, 485-89(1939) & CA **34**, 5662(1940)

N^1, N^4-Dialkyl-triethylene-tetranitramines. These are compds of the general formula R-N(NO_2)-CH_2.CH_2-N(NO_2)-CH_2.CH_2-N(NO_2)-CH_2.CH_2-N(NO_2)-R. They were patented by Blomquist and Fiedorek (Ref 3) for use in propellants as explosive, practically non-volatile, plasticizers for NC. The dialkyltriethylenetetranitramines are obtained, together with compds of general formula R-N(NO_2)-CH_2-N=N(:O)-O-CH_2.CH_2-N(NO_2)-CH_2.CH_2.N(NO_2)-R, when N-alkylethylenedinitramines are treated with ethylene dibromide as described by Franchimont & Klobbie (Ref 2)

As an example of such tetranitramines may be cited N^1, N^4-dimethyl-triethylene-tetranitramine (qv)

Refs: 1) Beil **4**, 572-73 2) A.P. Franchimont & E.A. Klobbie, Rec **7**, 346-47(1888) 3) A.T. Blomquist & F.T. Fiedorek, USP 2, 481, 283(1949) & CA **44**, 4925(1950)

Diallylbenzidine and Derivatives

Diallylbenzidine or 4, 4^1- Diallyldiaminobiphenyl (called Diallylbenzidin or 4, 4^1-Diallyldiamino-diphenyl in Ger), CH_2:CH.CH_2.HNC_6H_4-C_6H_4.NH.CH_2CH:CH_2; mw 264.36, N 10.60%; may be considered the parent compd of its nitroderivs but is not used to prep them

N, N'-Diallyl-3, 5, 3', 5'-tetranitrobenzidine, C_3H_5.$HNC_6H_2(NO_2)_2$-$C_6H_2(NO_2)_2$NH.C_3H_5; mw 444.36, N 18.91%; orn-red ndls (from phenol or salicylic acid methyl ester), mp 205° (dec); was prepd by heating at 100° in a sealed tube 3, 5, 3^1, 5^1-tetranitro-4, 4^1-dimethoxy (or diethoxy) -diphenyl with allylamine in alcohol (Refs 1 & 3)

N, N'-Diallyl-N, N', 3, 5, 3', 5'-hexanitrobenzidine, $[C_6H_2(NO_2)_2.N(NO_2).CH_2.CH:CH_2]_2$; mw 534.35, N 20.97%; nearly col crysts, mp dec ca 100°; was obtd by treatment of the tetranitro deriv with concd HNO_3 (Refs 2 & 3)

Refs: 1) Beil **13**, [109] 2) Beil **13**, [110] 3) G. van Romburgh, Rec **41**, 43(1922) & CA **16**, 1238(1922)

Diallylether or Allyl Ether (called Diallyläther in Ger), CH_2:CH.CH_2.O.CH_2.CH:CH_2; mw 98.14, O 16.30%; col liq, bp 94.3°, d 0.805, vap d 3.38, n_D 1.4163 at 20°; can be prepd from the reaction of allyl iodide & Na alloxide, or from allyl iodide & mercuric oxide (Refs 1 & 3). Diallylether is also obtd, along with other products, by hydrolysis of allyl chloride in a glass-packed tube (Ref 4). Niederl et al (Ref 2) reported that diallylether reacted with m-cresol, in the presence of a small quantity of H_2SO_4, with explosive violence

Refs: Beil **1**, 438, [477] & {1883} 2) J.B. Niederl et al, JACS **53**, 3393-94 (1931) 3) Thorpe **1**(1937), 258 4) Kirk & Othmer **7**(1951), 751 5) Sax (1957), 256 (Allyl Ether) 6) CondChemDict (1961)-not found

Diallylpentaerythritol Ether. See Bis(allyloxymethyl)-propanediol in Vol 2, p B128-L, where this compd is called erroneously Diallylpentaerythritol. The Dinitrate of this Ether was suggested as a possible gelatinizer for NC

Diallylphosphite, $(CH_2:CH.CH_2O)_2$ P.OH; mw 162.05, O 29.62%; yel oil, bp 58-62° at 0.5mm & 98° at 2mm, d 1.0793 at 20°, n_D 1.4430 at 20°; can be prepd by reaction of allyl alcohol & PCl_3 in the presence of a tertiary amine (Refs 2, 4, 5, 6 & 8). The product when carefully distilled in CO_2 usually exploded (Ref 5). A prepn from dimethyl phosphite, allyl alcohol & Na heated for 12hrs on a water bath is also reported (Ref 9)

Its nuclear magnetic resonance spectra were detd by Marvel (Ref 10). Also reactions are reported by Steinberg (Ref 3), and its addn to unsaturated compds by Pudovik & Khlyupina (Ref 7)

Craig & Hester (Ref 2) found that diallylphosphite dissolved in acetone and diluted with water to a 1% soln has fungicidal props

Refs: 1) Beil-not found 2) W.E. Craig & W.F. Hester, USP 2405958(1950) & CA **44**, 3202-03(1950) 3) G.M. Steinberg, JOC **15**, 637-47(1950) & CA **44**, 9339(1950) 4) G. Kamaĭ & E.I. Shugurova, Doklady **72**, 3014(1950) & CA **45**, 542(1951) 5) E.I. Shugurova & G. Kamaĭ, ZhurObshKhim **21**, 658-62(1951) & CA **45**, 8970(1951); JGenChem(Rus) **21**, 727-30(1951)(English translation) & CA **46**, 10099(1952) 6) G.E. Walter et al, USP 2574516(1951) & CA **46**,9580(1952) 7) A.N. Pudovik & N.I. Khlyupina, ZhurObshKhim **26**, 1672-77 (1956) & CA **51**, 3439(1957); JGenChem(Rus) **26**, 1877-80(1956)(English translation) & CA **51**, 14583(1957) 8) J. Kennedy, BritP 778077(1957) & CA **51**, 17980(1957) 9) G.L. Quesnel & R. Marqué, FrP 1162199(1958) & CA **55**, 1533(1961) 10) G Marvel, CR **248**, 3699(1959) & CA **54**, 4158-59(1960)

Diallyl-o-phthalate, $C_6H_4(CO_2CH_2CH:CH_2)_2$; mw 246.25, O 25.99%; syrupy liq, bp 183° at 15mm, d 1.118 at 20°, n_D 1.5189 at 25°, Q_{comb} at C_v 1663.5 kcal/mol, Q_{form} at C_v 132.2 kcal/mol; miscible with alc & eth; insol in w (Refs 3 & 4) are manufd by esterizing phthalic anhydride with allyl alcohol in the presence of H_2SO_4 (Ref 3)

According to Kirk & Othmer (Ref 2), diallyl-o-phthalate may find use as a possible replacement for styrene in laminating & potting applications. We suggest its use as a possible gelatinizer for NC

Refs: 1) Beil-not found 2) Kirk & Othmer **13**(1954), 175 3) L. Medard & M. Thomas, MP **37**, 132 & 138(1955) 4) P. Tavernier & M. Lamouroux, MP **13**, 202 & 206(1955) 5) Faith, Keyes & Clark(1957), 310 6) Sax(1957), 540 7) CondChemDict (1961), 348

Dialysis, Electrolysis and Electrodialysis.
Dialysis may be described as the fractional diffusion of solids from one side of a semipermeable membrane to the other side under a concentration gradient

Electrolysis is the process of local or spatial separation of the ions of an electrolyte and the transfer of their respective charges, ie the decompn of a compd by an elec current

Electrodialysis is the combined process of dialysis & electrolysis using semipermeable membranes to confine the soln under treatment

Dialysis is used for separating colloids from non-colloids. Electrodialysis is used for complete removal of small amts of electrolytes, usually on a lab scale

Refs: 1) P.H. Prausnitz & J. Reitstötter, "Elektrophorese, Elektro-osmose, Elektrodialyse in Flüssigkeiten", Steinkopf, Dresden und Leipzig (1931) 2) A. Weissberger, "Physical Methods of Organic Chemistry", Interscience, NY, Vol **3**(1950), pp 313-61 (R.E. Stauffer, "Dialysis and Electrodialysis") 3) Kirk & Othmer, **5**(1950), pp 1-26 (F.K. Daniel & P Stamberger, "Dialysis and Electrodialysis") 4) Perry (1963), Sect 17-42ff ("Dialysis and Electrodialysis")

Diameters, Limiting (of Explosives). According to Bobolev (Ref), the min diameters of expl charges are largely detd by the temp of detonation. The following min diameters of various expls are reported:

TNT (of 0.05-0.01mm grain size)	5.4mm
PA	2.28mm
K picrate	6mm
Pb picrate	1.5mm

PETN (of 0.25mm grain size) 2.2mm
(of smaller than 0.25mm grain size) 0.86mm
NG (of particles smaller than 0.4mm grain size) <2mm
Ref: V.L. Bobolev, DokladyAkadNauk **57**, 789-92(1947) & CA **46**, 4797(1952)

Diamide Powder No 1. A safety expl, based on nitrates, manufd in England during 1899 and then replaced by *Aphosite* (See Vol 1; p A273-R)
Ref: Daniel(1902), 204

Diamides. These compds are the dibasic acid derivs of ammonia or amines, and contain two -$CONH_2$ groups. Numerous combinations of diacids, diamines, and amino acids have been interacted. Copolymers contg various proportions of two or more diacids have been prepd (Ref 2). The diamide of carbonic acid is called urea, H_2NCONH_2. It is a product of animal metabolism and is prepd industrially by reaction of CO_2 with excess NH_3 at high temp, or by an older method involving the hydrolysis of cyanamide (Ref 5)

Frankel & Klager (Ref 3) patented the use of *Polynitrodiamides* of the general structure

$$RC(O)NHCH_2[A.N(NO_2)]_nCH_2A'.NHC(O)R'$$

(where R & R' are alkyl radicals
A & A' are alkylene radicals, and
n is 1-4)
as suitable main expl chges in shells, missiles & blasting devices, or as O donators & modifiers for proplnt fuels. These nitrodiamides are prepd by condensing the appropriate polynitrodiamines, derived from nitraza diisocyanates & strong mineral acids, with acid anhydrides. The polynitrodiamides prepd & cited are as follows:
N, N'-diacetyl-3,6-dinitraza-1, 8-octanediamine, mp 178-80°;
N, N'-diacetyl-3-nitraza-1, 5-pentanediamine, mp 197-98°;
N, N'-dihexanoyl-4-nitraza-1, 7-heptanediamine;
N, N'-dipentanoyl-4, 7-dinitraza-1, 10-decanediamine; and
N-propionyl-N'-butanoyl-3-nitraza-1, 5-pentanediamine

Compds of the general structure $RC(O)N(NO_2)A.CH_2.C(NO_2)_2CH_2.A.N(NO_2).C(O)R$ where R is alkyl and A is alkalene, were also patented by Frankel & Klager (Ref 4) for use as HE chges or proplnt additives. These nitramides are prepd by heating the appropriate dinitrodiamines with acyl anhydrides and treating the product with HNO_3. Thus, the following N-nitro nitramides were prepd & cited:
N, N'-dinitro-N, N'-dipropionyl-4, 4-dinitro-1, 7-heptanediamine;
N, N'-dinitro-N, N'-dipropionyl-5, 5-dinitro-1, 9-nonanediamine; and
N, N'-dinitro-N, N'-dihexanoyl-6, 6-dinitro-1, 11-undecanediamine
Refs: 1) Sidgewick, OrgChem of N(1937), p 148ff 2) Kirk & Othmer **10**(1953), 916ff 3) M.B. Frankel & K. Klager, USP 2967198 (1961) & CA **55**, 7842(1961) 4) M.B. Frankel & K. Klager, USP 2967199(1961) & CA **55**, 7843(1961) 5) Kirk & Othmer **2**, 2nd ed (1963), 69ff

Diamidides. These are compds having the structure N-C-N-C-N, such as $NR:CPh.NR'.CPh:NR''$ and $NRR'.CPh:N.CPh:NR''$, prepd from the appropriate imidochloride & amidine. The derivs of triazapentadiene & tetraazaheptatriene were prepd by Cooper et al (Ref 1). A systematic study showed that the diamidides $NRR.CPh:N.CPh:NR''$ yield normal salts, but di- and tri-amidides derived from $NR:CPh.NR'.CPh:NR''$ are readily decompd by acids (HX) to $NHR.CPh:NR$ & $X.CPh:NR''$. The bond structures of the diamidides are discussed in the light of their UV absorption structure (Ref 1)

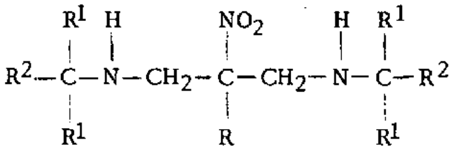
Peak (Ref 2) prepd conjugated diamidides of the type $NH_2.CAr:N.CAr':NH$ in which the N atoms carry no aryl or alkyl substituents. A number of such diamidides were prepd and isolated as fairly stable hydrated hydrochlorides
Refs: 1) F.C. Cooper et al, JCS **1951**, 391-404 & CA **46**, 1985(1952) 2) D.A. Peak, JCS **1952**, 215-26 & CA **47**, 2134(1953)

Diamin. Ger designation for Ethylene Dinitrate (EDD)

Diamines. *Aliphatic diamines* may be regarded as derived from hydrocarbons by replacement of two H atoms by two amino ($-NH_2$) groups. The most important aliphatic diamines are the ω-diamines of the normal hydrocarbons, $NH_2.[CH_2]_n.NH_2$, some of which are produced by putrefaction of animal matter. They are also prepd by a) heating alkylene dibromides with alc ammonia at 100° b) reduction of dicyanides, dioximes, and dihydrazones of dialdehydes or diketones, or dinitroparaffins c) from dicarboxylic acids thru Hofmann or Curtius degradations and d) hydrolysis of alkylene-diphthalimides. Cyclic diamines are produced by distilling the dihydrochlorides of aliphatic diamines, the nature of the product depending upon the number of C atoms separating the amino groups (Ref 1)

Aromatic diamines are usually prepd either by reduction of dinitro- or nitramino-compds or by reduction of aminoazo-compds. The o-diamines, unlike the m- & p-diamines, readily form cyclic compds (Ref 1)

Senkus (Ref 2) patented the prepn of *Nitro-Diamines* of the structure



$$R^2-C(R^1)_2-NH-CH_2-C(NO_2)(R)-CH_2-NH-C(R^1)_2-R^2$$

where R may be aryl, alkyl, H, or X(halogen); R^1 may be H, CH_2OH, or alkyl; and R^2 may be aryl, H, aralkyl, alkyl, or CH_2OH by treating nitroglycols with primary amines. These compds were unobtainable by interaction of nitro alkanes & methylol amines

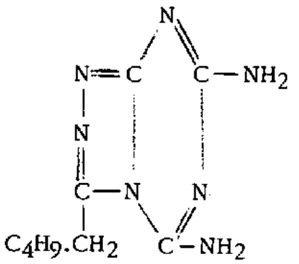
Refs: 1) Thorpe 1(1937), 314(Aliphatic); 315 (Aromatic) 2) Sidgewick, OrgChem of N(1937), 43 (Aliphatic); 79 (aromatic) 3) Kirk & Othmer **13**(1954), 185 (Reactions with succinic acid & succinic anhydride); 827 (Aromatic as solv for polyacrylonitride)

Diamino-1-alkenes. Doss & Bost (Ref 1) describe the prepn of 1, 2-bis(dimethylamino) ethylene (bp 38-40° at 15mm & n_D 1.4648), its hydrogenation in a Parr bomb at 40lb/sq inch to the ethane (n_D 1.4170) and the use of the *ethane deriv* as a component of hypergolic & fast-burning rocket fuels

Mahan (Ref 2) also prepd 1, 2-diamino-1-alkene derivs and tested their rocket fuel props of hypergolicity & ignition delay. The product *N, N, N', N'-tetramethyl-1, 2-diaminoethylene* was found to have an ignition delay of 3.6 millisec at 75°F with white, fuming HNO_3 as compared to 6.9 millisec for *N, N, N', N'-tetramethyl-1, 2-propanediamine*, the best of many hypergols similarly tested. Also prepd and tested are the hypergols *N, N, N', N'-tetraethyl-1, 2-diaminoethylene* and *N, N'-dimethyl-N, N'-diethyl-1,2-diaminoethylene*

Refs: 1) R.C. Doss & H.W. Bost, USP 2881217 (1959) & CA **53**, 19886(1959) 2) J.E. Mahan, USP 2941005(1960) & CA **54**, 17890(1960)

5, 7-Diamino-3-amyl (or 3-pentyl)-s-triazolo-[4, 3-a]-s-triazine,



; mw 221.27, N 44.32%; plates, mp 315-16° dec; was prepd by refluxing 5, 7-diamino-3-amyl-s-triazolo-[4, 3-a]-s-triazine.HNO_3 with dicyandiamide in H_2O for 4hrs (Refs 2, 3 & 4)

This compd and other 3-substituted derivs, similarly prepd, are claimed to be useful intermediates in the manuf of plastics, dyes & pharmaceuticals

Refs: 1) Beil-not found 2) D.W. Kaiser, USP 2473797(1949); CA **43**, 7975(1949) & CA **44**, 8382(1950) 3) D.W. Kaiser et al, JOC **18**, 1610(1953) & CA **48**, 13687(1954)

DIAMINOANISOLE AND DERIVATIVES

Diaminoanisole or Methoxyphenylenediamine (called Diamino-phenol-methyläther or Diamino-anisol in Ger), $(H_2N)_2C_6H_3.O.CH_3$; mw 138.17, N 20.28%. Three isomers are described in Beil, sometimes as the HCl salt: *2, 3-Diamino-anisole*, $C_7H_{10}N_2O$ + 2HCl, crysts (from alc + eth), mp ca 250°, to grn liq (Ref 1); *2, 4-Diamino-anisole*, ndls (from eth), mp 67-68° (Ref 2); $C_7H_{10}N_2O$ + 2HCl, crysts, sol in alc + eth (Ref 3); and *3, 5-Diamino-anisole*, $C_7H_{10}N_2O$ + 2HCl, crysts, mp dec ca 220°; its addn compd with 1, 3, 5-TNB, $C_7H_{10}N_2O$ + $C_6H_3N_3O_6$, dk red-brn ndls, mp 130° (Ref 4)
Refs: 1) Beil **13**, [308] 2) Beil **13**, (204) & [308] 3) Beil **13**, [315] 4) Beil **13**, 567 & (211)

Mononitrodiaminoanisole, $(H_2N)_2C_6H_2(NO_2).O.CH_3$; mw 183.17, N 22.94%. Three isomers are found in Beil: *5-Nitro-2, 3-deriv*, dk-red ndls (from w), mp 131-32° (Ref 1); *x-Nitro-2, 4-deriv*, yel ndls (from w), mp 162° (Ref 2); and *6-Nitro-3, 4-deriv*, dk-red ndls (from w), mp 184° (Ref 3)
Refs: 1) Beil **13**, (204) 2) Beil **13**, (206) 3) Beil **13**, [317]

Dinitrodiaminoanisole, $(H_2N)_2C_6H(NO_2)_2.O.CH_3$; mw 228.17, N 24.56%. Two isomers are described in the literature:
3, 5-Dinitro-2, 4-diaminoanisole, red-violet crysts (from alc or Nitrobenz), mp 250-55° & 260-61°; sol in alc or Nitrobenz; diffc sol in alc or AcOH; was prepd by heating on a water bath 2, 3, 5-trinitro-4-aminoanisole with alcoholic NH_3 and by other methods (Refs 1, 3, 4 & 5)
3, 5-Dinitro-2, 6-diaminoanisole, grn-red ndls (from ethyl acet), mp 237°; diffc sol in most org solvs; was prepd, as above, using the 3, 4, 6-trinitro deriv (Ref 2)

The expl props of these dinitro derivs were not detd. The 2, 4-deriv was found by Allen & Kennard (Ref 4) to be an effective antifoggant in photographic Ag halide emulsions. This use is also the subject of a Belgian patent (Ref 5)
Refs: 1) Beil **13**, 552, (206) & [311] 2) Beil **13**, (209) 3) H.F.J. Lorang, Rec **46**, 639(1927) & CA **22**, 230(1928) 4) C.F.H. Allen & K.C. Kennard, USP 2776211(1957) & CA **51**, 4187(1957) 5) KodakSocAnon, BelgP 553885(1957) & CA **54**,134(1960)

Trinitrodiaminoanisole, $C_7H_7N_5O_7$, not found in Beil or in CA thru 1961

DIAMINOANTHRAQUINONE AND DERIVATIVES

Diaminoanthraquinone (called Diamino-anthrachinon in Ger), $H_2N.C_6H_3(CO)_2C_6H_3.NH_2$ or $C_6H_4(CO)_2C_6H_2(NH_2)_2$; mw 238.24, N 11.76%. Ten isomers are described in Beil
Ref: Beil **14**, 197, 203, 212, 215, 216, (459, 461, 467, 470, 471, 473) & [112, 113, 116, 119, 120]

Dinitrodiaminoanthraquinone, $H_2N.C_6H_2(NO_2)(CO)_2C_6H_2(NO_2).NH_2$; mw 328.24, N 17.07%. The following isomers are found in Beil:
2, 6-Dinitro-1, 5-diaminoanthraquinone, red flakes; sl sol in pyridine with a red color (Ref 1)
4, 8-Dinitro-1, 5-diaminoanthraquinone, dk-red pltlts (from Nitrobenz) , mp above 330°; diffc sol in all solvs (Ref 2)
4, 5-Dinitro-1, 8-diaminoanthraquinone, yel crysts, mp above 300°; sl sol in hot NB or pyridine; v sl sol in AcOH; insol in w, alc or eth (Ref 3)
1, 5-Dinitro-2, 6-diaminoanthraquinone, brn crysts (from NB), mp above 300° (Ref 4)

Other props & methods of prepn of the dinitro derivs are given in the Refs
Refs: 1) Beil **14**, 210, (470) 2) Beil **14**, 210, (470) & [119] 3) Beil **14**, 215 4) Beil **14**, (473)

Dinitrodinitraminoanthraquinone, $O_2N.HN.C_6H_2(NO_2)(CO)_2C_6H_2(NO_2).NH.NO_2$; mw 418.24, N 20.10%;
4,8-Dinitro-1, 5-dinitraminoanthraquinone, cryst yel powder, mp explodes ca 134°; sl sol in concd H_2SO_4 giving a red color; insol in w; can be prepd by treating 1, 5-dinitramino-anthraquinone with HNO_3 at 0°. Its dibromo & some other salts are also expl (Ref 1)

X, X-Dinitro-1, 5-dinitraminoanthraquinone, grn-yel powder, mp explodes ca 121° on rapid heating, dec on prolonged slow heating at 116°, deflgr in presence of a little H_2SO_4; sol in excess H_2SO_4 giving a green color; insol or diffc sol in cold org solvs; obtd with other products by action of HNO_3 on 1, 5-diaminoanthraquinone at -12° (Refs 2 & 3)

Refs: 1) Beil **16**, 680 2) Beil **16**, [348] 3) E. Terres, Monatsh **41**, 610-611(1921) & CA **15**, 3835(1921)

Tetranitrodiaminoanthraquinone,
$H_2N.C_6H(NO_2)_2(CO)_2C_6H(NO_2)_2NH_2$; mw 418.24, N 20.10%;

2, 4, 6, 8-Tetranitro-1, 5-diaminoanthraquinone, dk-brn violet powd, mp-not given; sol in pyridine with a crimson-red color; sol in 40% fuming H_2SO_4 with an olive-grn col; sol in concd H_2SO_4 with a crimson-red col, becoming violet red on warming with boric acid; was prepd by heating 2, 4, 6, 8-tetranitroanthraquinonylene-1, 5-diurethane with concd H_2SO_4, and by other methods (Ref 1)

2, 4, 5, 7-Tetranitro-1, 8-diaminoanthraquinone, compd not described; sol in pyridine giving a blue-red color; sol in 40% fuming H_2SO_4 giving a yel-brn col; in concd H_2SO_4 with a reddish-brn color, becoming claret red on warming with boric acid; was obtd as above by heating the appropriate urethane deriv with concd H_2SO_4 at 80° (Ref 2)

Refs: 1) Beil **14**, 211 2) Beil **14**, 215 3) Bayer & Co, GerP 171588(1906) & CR **1906** II, 468

2, 4, 6, 8-Tetranitro-1, 5-dinitraminoanthraquinone,
$O_2N.HN.C_6H(NO_2)_2(CO)_2C_6H(NO_2)_2.NH.NO_2$; mw 508.24, N 22.05%; yel cryst powd (from acet + MeOH), mp explodes on gentle heating; readily sol in glacial AcOH & acet; sol in benz giving a brn-red color; in alc giving a brn color; sol in Na carbonate giving olive-brn; sol in caustic soda giving brn; sol in H_2SO_4 giving brick-red, becoming reddish-brn on adding boric acid; mod sol in water giving a brn color; was prepd by treating 1, 5-diamino-anthraquinone with an excess of HNO_3 (d 1.52) and cooling (Refs)

Refs: 1) Beil **16**, 680 2) R. Scholl et al, Ber **37**, 4446(1904)

NOTE: See also Aminoanthraquinone and Derivatives in this Encycl, Vol 1, p A183ff

DIAMINOAZOBENZENE AND DERIVATIVES

Diaminoazobenzene, Phenylazophenylene or Azodianiline, $C_{12}H_{12}N_4$, mw 212.25, N 26.40%. Several isomers are known:

2, 2′; 3, 3′; and 4, 4′-Diaminoazobenzene (called Diamino-azobenzol or Azoanilin in Ger), $H_2N.C_6H_4.N{:}N.C_6H_4.NH_2$. See Azoaniline in Vol 1 of this Encycl, p A646-L

2, 4-Diamino-azobenzene, $C_6H_5.N{:}N.C_6H_3(NH_2)_2$. Called 2, 4-Diamino-azobenzol; 4-Benzolazo-phenylendiamin or Base des Chrysoidins in Ger. See Chrysoidine, Vol 3, p C321-R

4-Amino-diazoaminobenzene, $C_6H_5.N{:}NH.C_6H_4.NH_2$; brn-yel ndls (from dil alc), mp 157° deflgr; sl sol in cold alc; diffc sol in eth; insol in petr eth & w; was prepd by treating 4-acetamino-diazobenzene with Na ethylate (Refs 1 & 2)

Refs: 1) Beil **16**, 732 2) R. Willstätter & M. Benz Ber **39**, 349(1906)

Azido, $C_{12}H_{11}N_7$, and *Diazido,* $C_{12}H_{10}N_{10}$, derivs of Diaminoazobenzene were not found in Beil

Mononitrodiamino-azobenzene, $C_{12}H_{11}N_5O_2$; mw 257.25, N 27.23%. Four derivs are found in the literature:

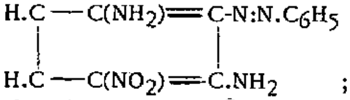
3′-Nitro-2, 4-diamino-azobenzene, $O_2N.C_6H_4.N{:}N.C_6H_3(NH_2)_2$; yel crysts (from dil alc), mp 204° (Ref 1)

4′-Nitro-2, 4-diamino-azobenzene, above formula, red-grn shiny ndls (from Nitrobenz or benzene homologes), mp 230°; readily sol in acet, pyridine & glacial AcOH; sl sol in alc & eth (Ref 2). Kul'berg & Ivanova (Ref 5) detd the color reaction of this compd in Mg salts

6-Nitro-2, 4-diamino-azobenzene or Nitrochrysoidine,
$C_6H_5.N{:}N.C_6H_2(NO_2).(NH_2)_2$; obtd as HCl salt, brn crysts having a metallic luster, mp-not reported; its aq soln is orn which turns

red on addg excess HCl (Refs 3 & 4). See also Chrysoidine, Vol 3, p C321-R

4-Nitro-2-phenylazo-1, 3-phenylenediamine,



$$\begin{array}{lll} H.C & - C(NH_2) = & C\text{-}N{:}N.C_6H_5 \\ | & & | \\ H.C & - C(NO_2) = & C.NH_2 \end{array}$$

; no description given, mp 193.5°; was prepd by coupling PhN_2Cl with 4-nitro-1, 3-phenylenediamine (Ref 6)

Refs: 1) Beil **16**, 384 2) Beil **16**, [204] 3) Beil **16**, [205] 4) A. Korczyński & S. Piasecki, AnzAkadKrakau [A] **1917**, 180 & CA **16**, 2123(1922) 5) L.M. Kul'berg & Z.V. Ivanova, ZhObshKhim **17**, 601-12 (1947) & CA **43**, 597(1949) 6) Z.J. Allan & F. Mužik, ChemListy **52**, 474-85(1958) & CA **53**, 4173(1959)

Dinitrodiamino-azobenzene, $C_{12}H_{10}N_6O_4$; mw 302.25, N 27.81%. Two isomers are found in the literature:

5, 4'-Dinitro-2, 4-diamino-azobenzene, $O_2N.C_6H_4.N{:}N.C_6H_2(NO_2)(NH_2)_2$; orn-colored powd, colors acetate rayon a golden yel; was prepd by coupling 4-nitro-1, 3-diaminobenzene with 4-nitroaniline (Ref 1)

3, 3'-Dinitro-4, 4'-diamino-azobenzene, $H_2N.(O_2N).C_6H_3.N{:}N.C_6H_3(NO_2).NH_2$; red plates or orn ndls (from phenol + ethanol), mp 340-41°; was obtd by hydrolysis with H_2SO_4 of 4, 4'-diacetamido-3, 3'-dinitroazobenzene, itself obtd by coupling of 2-nitro-4-nitrosoacetanilide & 4-amino-2-nitroacetanilide in acetic acid (Ref 2)

Refs: 1) Beil **16**, [205] 2) C.M. Atkinson et al, JCS **1954**, 2025 & CA **49**, 5487(1955)

NOTE: Higher nitrated derivs of Diaminoazobenzene were not found in Beil or in CA thru 1961

Diaminoazoxybenzene. See Azoxyaniline, Vol 1, p A665-L

DIAMINOBENZENE AND DERIVATIVES

Diaminobenzene, Phenylenediamine, or Aminoaniline (called Diamino-benzol or Phenylendiamin in Ger), $H_2N.C_6H_4.NH_2$; mw 106.12, N 26.40%. Three isomers are known:

1, 2-Diaminobenzene, col monoclinic crysts, mp 102-04°, bp 252-58°; sol in alc, eth & water (Refs 1,4 & 5). Some salts & addn compds are expl (Ref 1);

1, 3-Diaminobenzene, col ndls, unstable in air; usually obtd as the stable chloride, mp 63°, bp 282-87°; sol in alc, eth & w (Refs 2, 4 & 5). It forms numers salts & addn compds. The **Perchlorate salt,** $C_6H_2N_2 + 2HClO_4$, crysts, explodes by impact or friction (Ref 2)

1, 4-Diaminobenzene, col to lt purple crysts, sensitive to light, mp ca 147°, bp 267°; sol in alc & eth; sl sol in cold w & chlf (Refs 3, 4 & 5). It forms numerous salts & addn compds, some of which are unstable (Ref 3)

Of the three isomers, the 1, 4- or para form is an especially powerful skin irritant. It will cause kerato-conjunctivitis, swollen conjunctiva & eczema of the eyelids. Systemic poisoning is uncommon, but at least one fatal case of liver damage is reported. The m- & o- diaminobenzenes are somewhat less toxic than the p- isomer (Ref 4). Other props and methods of prepn are given in the Refs

Refs: 1) Beil **13**, 6, (5) & [8] 2) Beil **13**, 33, (10) & [23] 3) Beil **13**, 61, (18) & [34] 4) Sax (1957), 1003-04 5) CondChemDict (1961), 880-81

Azido, $C_6H_7N_5$, and *Diazido,* $C_6H_6N_8$, derivs of Diaminobenzene were not found in Beil or in CA thru 1961

Mononitrodiaminobenzene, $H_2N.C_6H_3(NO_2).NH_2$; mw 153.14, N 27.44%. The following isomers are described in the literature:

3-Nitro-1, 2-diaminobenzene, dk-red ndls (from dil alc), mp 158-59°; diffc sol in w (Ref 1);

4-Nitro-1, 2-diaminobenzene, dk-red ndls, mp 199-200° (Ref 2);

4-Nitro-1, 3-diaminobenzene, orn-red ndls (from w), mp 161°; sol in w; much more sol in alc & eth (Ref 3);

5-Nitro-1, 3-diaminobenzene, purple-red crysts (from w), mp 139-41°; sol in hot w, alc & acet (Ref 4); and

2-Nitro-1, 4-diaminobenzene, almost blk ndls having a brilliant luster (from w), mp 137° (Ref 5)

All of the mononitro derivs form salts & addn compds. Other props & methods of prepn are given in the Refs
Refs: 1) Beil **13**, (10) 2) Beil **13**, 29 & [21] 3) Beil **13**, 57, (16) & [30] 4) Beil **13**, 58, (16) & [31] 5) Beil **13**, 120, (38) & [59]

1, 3-Dinitraminobenzene [called 1,3 Dinitraminobenzol or m-Phenylendinitramin (?) in Ger], $C_6H_4(NH.NO_2)_2$ or $C_6H_4[N:N(:O).OH]_2$ (?); mw 198.14, N 28.28%; dk-yel oil, having a strong unpleasant odor, bp 90°, explodes very violently; sol in eth; insol in w; was obtd by warming a mixt of an alc soln of m-phenylenediamine nitrate with $AlCl_3$
Ref: Beil **16**, 676. No later refs found

1, 4-Dinitrosodihydroxylaminobenzene [Called 1,4-Bis-nitrosohydroxylamino-benzol in Ger] [called N, N -Dihydroxy-N, N'-dinitroso-phenylenediamine and N, N' -p-phenylene-bis-(N-nitrosohydroxylamine) in CA], $ON.(HO)N.C_6H_4.N(OH)ON$; mw 198.14, N 28.28%; compd blackens at 90° w/o fusion; was obtd by reaction of p-nitrophenylnitrosohydroxylamine & NONa (Ref 2). Its *Na salt*, $NaON:N(:O).C_6H_4.N(:O):N.ONa$, brn-yel crysts, deflgr on heating on Pt foil (Ref 3). Danzig et al (Ref 4) also prepd the Na salt & others and found them to be expl in nature. Its *Fe salt*, dk-red ppt, explodes ca 215°; almost insol in pyridine & acet (Ref 3)
Refs: 1) Beil **16**, [347] 2) D. Bigiavi, AttiAccadLincei [6] **4**, 457-59(1926) & CA **21**, 904(1927) 3) D. Bigiavi & F. Franceschi, GazzChimItal **57**, 380-81(1927) & CA **21**, 3048(1927) 4) M.J.Danzig et al, JOC **25**, 1071(1960) & CA**54**, 20827(1960)

Dinitrodiaminobenzene, $H_2N.C_6H_4(NO_2)_2.NH$; mw 198.14, N 28.28%. The following isomers are known:
3, 5-Dinitro-1, 2-diaminobenzene, red ndls (from alc), mp 210-15°; almost insol in cold w, sl sol in hot w; mod sol in glacial AcOH; very sol in alc (Ref 1);
2, 4-Dinitro-1, 3-diaminobenzene, orn-yel ndls (from alc) or lt yel-brn ndls (from glac AcOH), mp 260-64°; diffc sol in alc, eth, benz, w & glac AcOH (Ref 2);
4, 6-Dinitro-1, 3-diaminobenzene, yel prisms (from AcOH); sl sol in alc; somewhat more sol in glac AcOH (Ref 3);
2, 3-Dinitro-1, 4-diaminobenzene, brn-yel ndls (from benz or alc), mp 241-50° (dec); diffc sol in alc, eth, benz or chlf (Ref 4); and
2, 5-Dinitro-1, 4-diaminobenzene, reddish-brn ndls (from alc), mp 295-300° (dec); sol in acet & H_2SO_4; sl sol in chlf, CCl_4 & eth; insol in Benz (Ref 5)

Other props & methods of prepn are given in Refs.
Refs: 1) Beil **13**, 32 2) Beil **13**, 59 & [31] 3) Beil **13**, 59, (16) & [31] 4) Beil **13**, [59] 5) Beil **13**,[60]

2, 4, 6-Trinitro-1, 3-diaminobenzene; 2, 4, 6-Trinitro-m-phenylenediamine; or 1, 3-Diamino-2, 4, 6-trinitrobenzene (abbrd DATB or DATNB) [called 2,4,6-Trinitro-1,3-diaminobenzol or 2,4,6-Trinitro-phenylendiamin-(1,3) in Ger; and 2, 4, 6-Trinitrometaphenylenediamine in Fr]; $H_2N.C_6H(NO_2)_3.NH_2$; mw 243.14, N 28.81%; yel crysts (glac AcOH), mp 287-301° dec; sl sol in glac AcOH; v sl sol in other solvs; can be prepd by nitrating m-dichlorobenzene to 2, 4, 6-trinitro-1, 3-dichlorobenzene and aminating in methanol soln; or by the amination of 2, 4, 6-trinitro-3-aminoanisole (Ref 1); and by other methods (Refs 2, 3, 4, 5, 6, 7 & 9). Warman & Siele (Ref 10) describe an improved procedure, leading to an 89% yield, in which phosphorous oxytrichloride & dipyridinium styphnate react directly at steam-bath temp, and a suspension of the resulting 2, 4, 6-trinitro-1, 3-dichlorobenzene in methanol is treated with gaseous NH_3

The expl props of DATNB are given in Ref 8. DATNB is used as a pressed HE, and also used as a ballistic modifier in some rocket proplnts (Ref 11)
Refs: 1) Beil **13**, 60, (17) & [33] 2) C.F. van Duin & B.C.R. van Lennep, Rec **39**, 151, 165, 170, 174(1920) & CA **14**, 2708(1920) (Prepn, stability & impact sensitivity) 3) H.F.J. Lorang, Rec **46**, 649-50(1927) & CA **22**, 231(1928)(Prepn) 4) H.B. Hass et al, JACS **72**, 2282-83(1950) & CA **44**, 9929 (1950)(Prepn) 5) H. Feuer & A.A. Harban,

USP 2679538(1954) & CA **49**, 4715(1955) (Prepn from chloropicryl chloride by treatment with gaseous NH_3) 6) M. Blais et al, PATR **2524**(1958)(conf, not used as a source of info) 7) K.G. Shipp & M.E. Hill, NAVORD Rept **6016**(1958)(conf, not used as a source of info) 8) O.E. Sheffield, "Properties of Explosives of Military Interest", PATR **1740**, Supplement **1**(1958); Reprinted as Ord Corps Pamphlet ORDP **20-178**(1960), 7-10; as US Army Materiel Command Pamphlet AMCP **706-188**(1963)(Conf); and as AMCP **706-177** (March 1967)(Unclassified) 9) M. Blais et al, PATR **2587**(1959)(conf, not used as a source of info) 10) M.Warman & V.I Siele, JOC **26**, 2997-98(1961); PA Monograph 104 & CA **56**, 364-65(1962) 11) Proplnt Manual SPIA/M2, Unit 644 (1962)(Conf) 12) W. Selig et al, Explosivst **19**, No 1, 1–13(1971) (Charge-transfer complexes of DATNB)

Tetranitrodiaminobenzene, $H_2N.C_6(NO_2)_4.NH$ or $H_2N.C_6H(NO_2)_3NH.NO_2$; mw 288.14, N 29.17%. This compd is listed in CA Collective Formula Index **14-40**(1920-46), p 111 as Tetranitrophenylenediamine because it is incorrectly listed in Ref 2 as Aminotrinitrophenylnitramine. The orig article shows this compd to be 2,4,6-Trinitro-1-methylnitramino-3-aminobenze (called l' aminotrinitrophenylmethylnitramine in Fr)

The *Amm salt,* called 2,4,6-Trinitro-1-ammoniumnitramino-3-aminobenzene or NH_3+ 2,4,6-Trinitro-3-aminophenylnitramine (in CA), $H_2N.C_6H(NO_2)_3.N(NO_2).NH_4$, canary-yel crysts (from boiling alc), mp dec at 180° when heated rapidly and at 300° when heated slowly, explodes when heated in air, was prepd by Macciotta & Orani (Ref 3) by dissolving 2,3,4(6-tetranitrophenylnitramine, $(O_2N)_4C_6H.N(NO_2)H$, in w, filtering, and making sl alk with NH_4OH. With excess NH_4OH *sym-Trinitrophenylenediamine* (DATNB) is formed

Refs: 1) Beil-not found 2) C.F. van Duin & B.C.R. van Lennep, Rec **39**, 153-54(1920) & CA **14**, 2708(1920) 3) E. Macciotta & Z. Orani, Gass **60**, 412-13(1930) & CA **24**, 4280(1930)(Compd called Sale ammonico della 2:4:6-trinitro-3-amminofenilnitrammina in Ital)

DIAMINOBENZIL AND DERIVATIVES

Diaminobenzil, $H_2N.C_6H_5CO.CO.C_6H_5.NH_2$; mw 240.25, N 11.66%. The p,p'-Diamino deriv, yel ndls (from boiling water or alc), 166-69°, was patented by Anderson (Ref 3) by reducing p, p'-dinitrobenzil using $FeSO_4$, $(NH_4)_2S$ or $Fe(OH)_2$ as reducing agents, or using catalytic reducing agents. A similar method of prepg the 4,4'-Diamino deriv had been reported earlier by Kuhn et al (Ref 2). An improved prepn of the 4,4'-Diamino deriv from p-aminobenzaldehyde which, after acetylation, is converted to 4,4'-diacetamidobenzoin which is oxidized to the benzil deriv and hydrolyzed with HCl is reported by Gee & Harley-Mason(Ref 4)

4,4 -Diaminobenzil was found by Kuhn et all (Ref 5) to act as an anesthetic. Its tuberculostatic activity was tested by van der Stelt et al (Ref 6)

Refs: 1) Beil-not found 2) R. Kuhn et al, Ber **76B**, 405-12(1943) & CA **37**, 6256(1943) 3) G.W. Anderson, USP 2359280(1944) & CA **39**, 785(1945) 4) H.L. Gee & J. Harley-Mason, JCS **1947**, 251 & CA **41**, 5123(1947) 5) R. Kuhn et al, Naturwissenschaften **38**, 12(1951) & CA **46**, 2239(1952) 6) C. van der Stelt et al, Antonie Van Leeuwenhoek JMicrobiolSerol **20**, 285(1954) & CA **49**, 10866(1956)

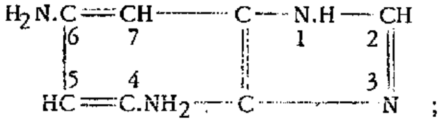
Dinitrodiaminobenzil, $C_{14}H_{10}N_6O_6$, not found in Beil or in CA thru 1961

3,3',5,5'-Tetranitro-4,4 -diaminobenzil
$H_2N.C_6H_2(NO_2)_2.CO.CO.C_6H_2(NO_2)_2.NH_2$; mw 420.25, N 20.00%; brn-yel crysts (from NB), mp 271°; insol in most org solvs; was prepd by heating of 3,3' 5,5'-tetranitro-4,4-dimethoxybenzil with aq alc NH_3 in a tube at 100°

Refs: 1) Beil **14**, [98] 2) J. van Alphen, Rec **48**, 1118(1929) & CA **24**, 844(1930)

Hexanitrodiaminobenzil, $C_{14}H_6N_8O_{14}$, not found in Beil or in CA thru 1961

4,6-Diaminobenzimidazole,

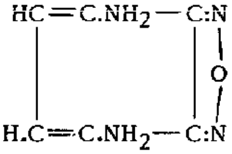


mw 148.17, N 37.82%. The *Dihydrochloride* deriv, $C_7H_{10}N_4Cl_2$, wh ndls (from HCl), mp 335° dec; was prepd by addg formic acid to freshly prepd 1,2,3,5-tetra-aminobenzene trihydrochloride monohydrate in 2N HCl and heating the mixt on a steam bath for 1hr. The *Picrate*, $C_{13}H_{11}N_7O_7$, dk-orn crysts (from Me cellosolve) becoming deep red on drying at 100°, mp 231-32° dec; was obtd by passing an aq soln of the dihydrochloride thru Amberlite (IRA 400) and immediately addg an ethanoic soln of PA. It was purified by dissolving repeatedly in Methyl Cellosolve & pptg with water

A number of other derivs with substituents in the 4 & 6 positions, their hydrochlorides & picrates were also prepd by Hoover & Day

Refs: 1) Beil-not found 2) J.R.E. Hoover & A.R. Day, JACS **77**, 4324-27(1955) & CA **50**, 5645(1956)

Diaminobenzofurazan,



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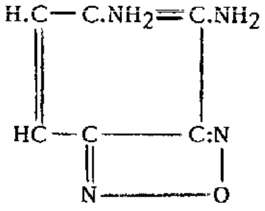


I II

mw 150.14, N 37.32%. The *4,5-Diamino* deriv (Formula II), dk-yel crysts & red ndls when closely packed, mp 151°; results from catalytic reduction of benzodifurazan in MeOH. The *4,7-Diamino* deriv, red crysts (from hot w), mp 193-94°; sol in methanol, acet, chlf & toluene giving a red colored soln; results from the catalytic reduction of 4,7-benzofurazanquinone dioxime in MeOH. Several other derivs and salts were also prepd, some of which are probably mild expls (Ref 2)

Refs: 1) Beil-not found 2) W. Borsche & H. Weber, Ann **489**, 287-88(1931) & CA **26**, 708(1932)

DIAMINOBENZOPHENONE AND DERIVATIVES

Diaminobenzophenone (called Diaminobenzophenon in Ger), $H_2N.C_6H_4.CO.C_6H_4.NH_2$; mw 212.24, N 13.20%. The possible isomers are described in Beil: *2,2'-Diamino;* lt-yel pltlts (from dil alc) or yel rods (from benz), mp 132-35°; forms salts & addn compds (Ref1);
2,3'-Diamino-, golden-yel pltlts or ndls (from dil alc), mp 80°; readily sol in alc; less sol in w; forms a hydrochloride salt (Ref 1);
2,4'-Diamino-, straw colored ndls (from w), mp 128-29°; readily sol in alc; forms a hydrobromide salt (Ref 1);
3,3'-Diamino-, lt-yel ndls (from alc), mp 170-74°; sol in alc & eth; v sl sol in w; also forms salts (Ref 2);
3,4'-Diamino-, ndls + H_2O (from dil alc), mp 98-100° on rapid heating, 125-26° when dehydrated & 131-32° by std method; readily sol in alc (Ref 2); and
4,4'-Diamino-, prismatic ndls (from dil alc), mp 241-45°; readily sol in alc; mod sol in hot w & is decompd by boiling w; insol in cold w (Ref 3)

Other props & methods of prepg Diaminobenzophenones are given in the Refs

Refs: 1) Beil **14**, 87 2) Beil **14**, 88 & (390) 3) Beil **14**, 88, (391) & [56]

3,3'-Dinitro-4,4'-diaminobenzophenone, $H_2N.C_6H_3(NO_2)_2CO.C_6H_3(NO_2)_2.NH_2$; mw 302.24, N 18.54%; crysts (from abs alc), red ndls becoming yel on heating, red ndls (from pyridine), mp 289-93°; almost insol in all org solvs, except pyridine; was prepd by reaction of alc NH_3 with 3,3'-dinitro-4,4'-dichlorobenzophenone at 150° or in the presence of Na acetate at 125°; and by reaction of alc NH_3 on the dibromo or di-iodo-dinitrobenzophenone deriv at 150°. Its expl props are not reported

Refs: 1) Beil **14**, 100, (394) 2) D. Maron & C. Fox, Ber **47**, 2781(1914) 3) P.J. Montagne, Ber **48**, 1033(1915) & Ber **51**, 1488 (1918)

3,5,3',5'-Tetranitro-4,4'-diaminobenzophenone, $[H_2N.C_6H_2(NO_2)_2]_2CO$; mw 392.24, N 21.43%; lt-yel, wooly crysts (from NB), mp dec 250°, mp 324° by Maquenne block); sol in alc, benz, AcOH & H_2SO ; was prepd by heating the 4,4'-dichlorotetranitrobenzophenone deriv with alc NH_3; and by reaction of 4,4'-dimethylamino-3,5,3',5'-tetranitro-diphenylmethane or -benzophenone with chromic acid & AcOH (Ref 1). The expl props of this compd are not reported (Ref 2)

Refs: 1) Beil **14**, 100 2) J. van Alphen, Rec **49**, 160-61(1930) & CA **24**, 3007(1930)

NOTE: Higher nitrated derivs of Diaminobenzophenone were not found in Beil or in CA thru 1961

4,'4 -Diaminobiphenyl. See Benzidine in Vol 2, p B63-R

Diaminobiphenyl, Nitrated Derivatives. In addn to the nitrated derivs of Benzidine or 4,4'-Diaminobiphenyl given in Vol 2, p B64 of this Encyclopedia, the following nitrated derivs of Diaminobiphenyl are described in the literature:

Dinitrodiaminobiphenyl, $H_2N.C_6H_3(NO_2).C_6H_3(NO_2).NH_2$; mw 274.23, N 20.43%;

5,5'-Dinitro-2,2'-diaminobiphenyl; golden-yel prisms having a violet glaze (from phenol + alc), mp-becomes brn & sinters at 285° & 303°, sol in hot phenol; diffc sol in alc; v sl sol in chlf; insol in eth, benz & petr eth; was prepd by nitration of diacetyl-2,2'diaminobiphenyl and hydrolysis of the dinitro deriv with HCl & alc (Refs 1 & 2);

4,4'-Dinitro-2,2'-diaminobiphenyl, formula as above, orn crysts (from alc), mp 249-50°; was obtd by heating of disuccinyl-2,2'-diamino-4,4'-dinitrobiphenyl with 10% KOH (Ref 2);

4,5'-Dinitro-2,2'-diaminobiphenyl formula as above, rhmb crysts + AcOH (from AcOH), mp 180°, on heating in air for several hrs or on washing with alc the crysts lose AcOH; exists in two forms: yel mp 143-44° & orn, mp 179-80° & 170-72° (Ref 5), the labile form is obtd by addg w to its soln in HCl, by washing the crysts of the acetate with alc, and by hydrolyzing the disuccinyl & di-Ac compds with KOH. Dil acid or boiling with MeOH changes the lower-melting form to the higher-melting form (Ref 2);

4,3'-Dinitro-2,4'-diaminobiphenyl, formula as above; bright-red crysts (from alc), mp 199-200°; sol in concd but not in dil acids; was formed on saponification of 2,4'-diacetylamino-4,3'-dinitrobiphenyl by boiling in a mixt of concd H_2SO_4 & 50% alc for 6hrs, pouring into w and washing the ppt with aq HCl (Ref 3);

5,3'-Dinitro-2,4 -diaminobiphenyl, formula as above, red crysts, mp 242-43°; was obtd on saponification of 2,4'-di(acetylamino)-3',5-dinitrobiphenyl with a mixt of dil H_2SO_4 & alc for six hrs (Ref 4); and

6,6'-Dinitro-2,2'-diaminobiphenyl, formula as above, orn blades (from alc), mp 241° & 242-44° (Ref 2); was obtd by boiling 2,6,2',6'-tetranitrobiphenyl with an excess of Na sulfide & sulfur in hot alc and extracting the amines with eth (Ref 6)

The expl props of these dinitro derivs were not reported

Refs: 1) Beil **13**, 210 2) S. Sako, MemCollEngKyushuImpUniv 6, 327-51(1932) & CA **26**, 3245-46(1932) 3) C. Finzi & A. Mangini, Gazz **62**, 676(1932) & CA **27**, 78(1933) 4) Ibid, **62**, 1191(1932) & CA **27**, 2948(1933) 5) W. Baker et al, JCS **1958**, 2664 & CA **53**, 1281(1959) 6) R.S.W. Braithwaite et al, JCS **1958**, 4076-77 & CA **53**, 11379(1959) 7) P.F. Holt & A.N. Hughes, JCS **1960**, 3219 & CA **55**, 1626(1961)

4,6,4',6';Tetranitro-3,3'-diaminobiphenyl, $H_2N.C_6H_2(NO_2)_2.C_6H_2(NO_2)_2.NH_2$; mw 364.23, N 23.08%; red-brn crysts, mp 297°; was prepd by heating 5,5'-dichloro-2,4,2',4'-tetranitrobiphenyl with alc NH_3 at 110° (Ref 1 & 2). The expl props of this tetranitro deriv were not reported

Refs: 1) Beil **13**, 58 2) W. Borsche & B.G.B. Scholten, Ber **50**, 610(1917)

2,4,6,2',4',6'-Hexanitro-3,3'-diaminobiphenyl or 2,4,6,2',4',6'-Hexanitro-3,3'-biphenyldiamine; also called Dipicramide (by Dacons et al), $H_2N.C_6H(NO_2)_3.C_6H(NO_2)_3.NH_2$;

mw 454.23, N 24.67%; microcryst-yel powd (from acet + alc), mp 299° dec; explosive compd; was prepd by addg portionwise during 1hr at a temp below 12° 3,3'-dichloro-2,4,6,2',4',6'-hexanitrobiphenyl to abs ethanol saturated with dry NH_3; anhyd NH_3 slowly bubbled thru the blood-red soln for an addnl 15mins and the soln slowly heated to reflux; refluxed for 30mins with loss of NH_3 & alc; cooled to 10° and the ppt washed with 3% aq HCl, alc & eth; the compd was purified by treating a hot acetonic soln with charcoal, addg hot alc & concentrating to a small vol (Ref 2)

Refs: 1) Beil-not found 2) J.C. Dacons et al, Tetrahedron **19**, 797-98(1963) & CA **59**, 8626(1963) 3) C. Dickinson et al, ProcChemSoc (London) **1964**, 232-33 & CA **61**, 14050(1964)(UV absorption spectra of Dipicramide & other ring-substituted picramides in dioxane)

1(5-Diaminobiuret)(called Diamino-biuret; Imino-dicarbonsäure-dihydrazid; Iminodiameisensäure-dihydrazid; or Ammoniakdicarbonsäuredihydrazid in Ger), $HN(CO.NH.NH_2)_2$; mw 133.12, N 52.61%; col prisms (from w + alc) or ndls (from glac AcOH), mp 200-05° dec; readily sol in w; mod sol in hot glac AcOH; sl sol in alc & eth; was prepd by reaction of N-tricarboxylic ester & 42% hydrazine hydrate (Refs 1 & 2). Its relative basicity was detd by Rochlin et al (Ref 3)

Refs: 1) Beil **3**, 101 & {189} 2) C.F.H. Allen & A. Bell, OrgSynth **24**, 58-59(1944) & OrgSynthColl Vol **3**, 404(1955) 3) P. Rochlin et al, JACS **76**, 1451-53 (1954) & CA **49**, 3951(1955)

NOTE: Nitrated deriv of Diaminobiuret were not found in Beil or in CA thru 1961

DIAMINOBUTANE AND DERIVATIVES

Diaminobutane or Butanediamine, $C_4H_{12}N_2$; mw 116.17, N 48.23% All possible isomers are known:

1,2-Diaminobutane, $CH_3.CH_2.CH(NH_2).CH_2.NH_2$ exists in the form of salts, many of which are unstable on heating; and as an oil having a fish-like odor, bp 135-40° (Ref 1);

1,3-Diaminobutane, $CH_3.CH(NH_2).CH_2.CH_2.NH_2$ forms numerous salts, some of which are unstable on heating; and exists as an oil, bp 140.5-41.5° at 738mm Hg (Ref 2);

1,4-Diaminobutane, $H_2N.CH_2.CH_2.CH_2.CH_2.NH_2$ forms many salts, some of which are unstable on heating; exists as pltlts, mp 26-28° (Ref 3); and

2,3-Diaminobutane, $CH_3.CH(NH_2).CH(NH_2).CH_3$ forms many salts which are unstable on heating; exists as meso form, bp 59-60° at 60mm Hg, and as racem form, bp 57-58° at 60mm Hg (Ref 4)

Other props & methods of prepn of the various derivs are given in the Refs

Refs: 1) Beil **4**, 264, [701] & {567}
2) Beil **4**, 264, (419), [701] & {569}
3) Beil **4**, 264, (420), [701] & {570}
4) Beil **4**, 265, [707] & {579}

Azido, $C_4H_{11}N_5$, and *Diazido,* $C_4H_{10}N_8$, derivs of Diaminobutane were not found in Beil or in CA thru 1961

Mononitrodiaminobutane, $C_4H_{11}N_3O_2$, not found in Beil or in CA thru 1961

Dinitroaminobutane, $C_4H_{10}N_4O_4$; mw 178.15, N 31.45%. The following derivs are reported in the literature:

1,3-Dinitraminobutane, $CH_3.CH(NH.NO_2).CH_2.CH_2.NH.NO_2$; col crysts (from benz), mp 81.6-82.2°; was prepd by dissolving 1,3-dinitro-4-methyl-1,3-diazacyclohexanone-2 in 10% aq NaOH and acidifying this soln with concd HCl (Ref 3);

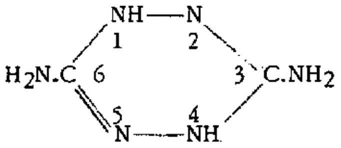
1,4-Dinitraminobutane, $O_2N.NH.CH_2.CH_2.CH_2.CH_2.NH.NO_2$; col crysts (from w), mp 162.2° dec; mod sol in hot w; decompd by boiling with H_2SO_4; was prepd from N,N'-dinitro-N,N'-dicarbomethoxy-tetramethylene & aq NH_3 (Refs 1 & 2); and by hydrolysis of 1,3-dinitro-1,3-diazacycloheptone-2 (Ref 3)

Refs: 1) Beil **4**, 573 2) M.P.J. Dekkers, Rec **9**, 97(1890) 3) A.F. McKay & D.F. Manchester, JACS **71**, 1972-73(1949) & CA **43**, 9065-66(1949)

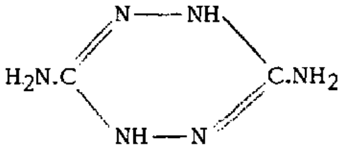
NOTE: Higher nitrated derivs of Diaminobutane were not found in Beil or in CA thru 1961

DIAMINODIHYDRO-s-TETRAZINE AND DERIVATIVES

Diaminodihydro-s-tetrazine, $C_2H_6N_6$; mw 114.12, N 23.65%. The *3,6,-Diamino-1.4-dihydro-s-tetrazine,*



or

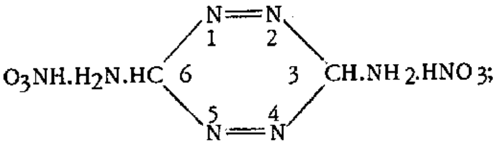


is known, dk-red amorphous solid, mp 360°; was obtd when S-methylisothiosemicarbazide hydroiodide, $H_2N.NHC(:NH).S.CH_3.HI$, was condensed in a heterocyclic amine, such as piperidine, pyrrolidine or morpholine (Ref 3). Its sulfate & picrate were also prepd

Walter (Ref 2) patented the use of this compd, in the manuf of *aminoplasts* by its reaction with aldehydes, prepd by recrystallization from H_2O of the reaction of $H_2N.COOC_2H_5$ and an aq soln of 85% $N_2H_4.H_2O$

Refs: 1) Beil-not found 2) H.A. Walter, USP 2475440(1949) & CA **43**, 9088(1949) 3) F.L. Scott & J. Reilly, Chem & Ind **1952**, 908 & CA **47**, 6886(1953) 4) F.L. Scott, Chem & Ind **1954**, 158-59 & CA **49**, 2446(1955)

3,6-Diamino-3,6-dihydro-s-tetrazine Dinitrate (called Nitrate de diaminotétrazène in Fr),

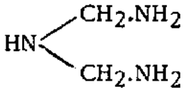


mw 240.14, N 46.67%; deflgr weakly in a flame, explodes at 150° when heated in a tube; was prepd and examined along with several other compds as to solubility, hygroscopicity, corrosive effects, effect of heat, shock and behavior on ignition. Only Pb-trinitroresorcinol and tetracene were considered useful as primer compns (Ref 2)

Refs: 1) Beil-not found 2) H. Ficheroulle & A. Kovache, MP **31**, 14(1949) & CA **46**, 11687-88(1952)

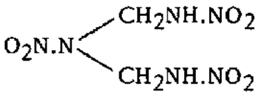
NOTE: See also 3,6-Diamino-s-tetrazine

Diaminodimethylamine,



$HN(CH_2.NH_2)_2$

Its trinitro deriv, *1,1′-Dinitramino-N-nitro dimethylamine,*



$O_2N.N(CH_2NH.NO_2)_2$;

mw 210.12, N 40.00%; was found as a secondary by-product during the nitration of hexamine, and its presence explains the low yield of RDX obtd (Ref 2)

Refs: 1) Beil-not found 2) L.A. Di Cerrione, AnnChimApplicata **38**, 255-71(1948) & CA **43**, 4633(1949)

Diaminodimethylanthraquinone, $C_{16}H_{14}N_2O_2$; mw 266.29, N 10.52%. Two isomers are found in the literature:

2,4-Diamino-1,3-dimethylanthraquinone, $C_6H_4(CO)_2C_6(CH_3)_2(NH_2)_2$, dk-red crysts (from glac AcOH), mp 230° dec; obtd by reduction of 2,4-dinitro-1,3-dimethylanthraquinone with hot aq Na sulfide soln (Ref 2)

1,8-Diamino-2,7-dimethylanthraquinone, $H_2N.C_6H_2(CH_3)(CO)_2C_6H_2(CH_3).NH_2$; grn-red crysts, mp 208-09°; obtd by reduction of 1,8-dinitro-2,7-dimethylanthraquinone with $ZnCl_2$ & KOH (Refs 1 & 3)

Refs: 1) Beil **14**, 225 & [126] 2) Beil **14**, 479 3) G.T. Morgan & E.A. Coulson, JCS **1929**, 2213

NOTE: Dinitro, $C_{16}H_{12}N_4O_6$, and *Tetranitro,* $C_{16}H_{10}N_6O_{10}$, derivs of Diaminodimethylanthraquinone were not found in Beil or in CA thru 1961

Compare with the nitrated derivs of Diaminoanthraquinone in this Vol

DIAMINODIMETHYLBENZENE AND DERIVATIVES

Diaminodimethylbenzene or Diaminoxylene (called Diamino-dimethyl-benzol; Diamino-xylol and Dimethyl-phenylendiamin in Ger); $C_6H_2(NH_2)_2(CH_3)_2$; mw 136.19, N 20.57%. All possible isomers are described in Beil:
3,4-Diamino-1,2-dimethylbenzene, pltlts, mp 89° (Ref 1);
3,5-Diamino-1,2-dimethylbenzene, ndls (from alc), mp 66-67° (Ref 1);
3,6-Diamino-1,2-dimethylbenzene, yel nds or prisms (from benz), mp 116° (Ref 2);
4,5-Diamino-1,2-dimethylbenzene, pltlts + H_2O, mp 125-26° (Ref 2);
2,4-Diamino-1,3-dimethylbenzene, col ndls (from petr eth), mp 64-66° (Ref 3);
2,5-Diamino-1,3-dimethylbenzene, lfts (from benz + petr eth), mp 103-04° (Ref 4);
4,5-Diamino-1,3-dimethylbenzene, ndls (from petr eth) or lfts (from benz), mp 75-78° (Ref 5);
4,6-Diamino-1,3-dimethylbenzene, ndls (from petr eth), mp 104-05.5° (Ref 6);
2,3-Diamino-1,4-dimethylbenzene, nds, mp 75° (Ref 7);
2,5-Dimethyl-1,4-dimethylbenzene, ndls (from benz) or lfts (from w), mp 147-50° (Ref 8); and
2,6-Diamino-1,4-dimethylbenzene, yel prisms (from benz + petr eth), mp 102-03° (Ref 9)

Other props & methods of prepn are given in the Refs

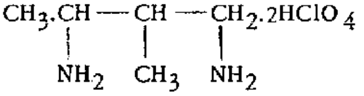
Refs: 1) Beil **13**, 178 2) Beil **13**, 179
3) Beil **13**, 181 4) Ibid 181 & [70]
5) Ibid, 182 6) Ibid, 183, (49) & [70]
7) Ibid, 187 8) Ibid, 187 & [71]
9) Ibid, 188

Mononitrodiaminodimethylbenzene, $(CH_3)_2C_6H(NO_2)(NH_2)_2$; mw 181.19, N 23.19%. Three isomers are described in the literature:
6-Nitro-2,4-diamino-1,3-dimethylbenzene, or-yel nds (from w), mp 151-52°; readily sol in alc; diffc sol in w; was prepd by reduction of 4,6-dinitro-2-amino-m-xylene with Amm hydrosulfide (Ref 1);
2-Nitro-4,6-diamino-1,3-dimethylbenzene, ruby-red prisms (from alc or boiling w), mp 212-13°; sol in alc; mod sol in boiling w; v sl sol in cold w; was obtd by heating 2,6-dinitro-4-amino-m-xylene with alc Amm sulfide in a sealed tube at 100°; compd forms numerous addn salts (Ref 2); and
5-Nitro-2,3-diamino-1,4-dimethylbenzene, lt-red ndls (from benz or dil aq alc), mp 169°; mod sol in eth & glac AcOH; sl sol in hot w; v sl sol in benz; was obtd by reduction of either 3,5-dinitro-2-azido-1,4-dimethylbenzene or 3,5-dinitro-2-amino-p-xylene with Na sulfide in dil alc (Ref 3)

Refs: 1) Beil **13**, 181 2) Beil **13**, 185
3) Beil **13**, (50)

NOTE: Higher nitrated derivs of Diaminodimethylbenzene were not found in Beil or in CA thru 1961. Compare with Dimethylphenylenediamine

1,3-Diamino-1,2-dimethylpropane Diperchlorate or 1,2-Dimethyl-1,3-propanediamine Diperchlorate,



$$\underset{NH_2}{CH_3.\underset{|}{CH}} - \underset{CH_3}{\underset{|}{CH}} - \underset{NH_2}{\underset{|}{CH_2}}.2HClO_4$$
;

an expl listed as Compd No 212 in ADL PureExplCompds, Part 1(1947, pp 62, 69, 70 & 149

DIAMINOETHANE AND DERIVATIVES

1,2-Diaminoethane or Ethylenediamine (called 1,2-Diamino-äthan or Äthylen-diamin in Ger), $H_2N.CH_2.CH_2.NH_2$; mw 60.10, N 46.62%; col liq, fr p 10.8°, bp 117.2° at 760mm, d 0.8995 at 20°, n_D 1.4565 at 20°, flash p (COC) 150°F; completely miscible with w; forms azeotropes with w, toluene & methyl cellosolve; was prepd for the first time in 1871 by heating ethylene dichloride with alc NH_3 in a closed tube (Refs 1 & 2). This same reaction, only slightly altered in detail, is now used commercially

Diaminoethane is remarkable for its ability to form coordination compds. Its reactions with fatty acids & their derivs lead to amides, either mono- or diamides. With many inorganic acids, it forms salts, some of which are expl

Diaminoethane is irritating to the eyes & skin. Prolonged or repeated breathing of the vapor should also be avoided

This compd & its derivs find wide applications in the chemical industry as intermediates, in the rubber industry & in a wide variety of miscellaneous applications

Refs: 1) Beil **4**, 230, (398), [676] & {478}
2) Kirk & Othmer **5**(1950), 898-905(Ethylene diamine & other ethylene amines)
3) Sax (1957), 678 (Toxicity & props)

N-Nitro-diaminoethane or N-Nitro-ethylenediamine (called β-Aminoethylnitramine by Hall & Wright), $H_2N.CH_2.CH_2NH.NO_2$; mw 105.10, N 39.98%; col crysts (from w), mp 240-45° dec; insol in alc; was obtd by treatment of propyl-β-chloroethyliminonitrocarbonate with alc KOH or by soln of β-aminoethylnitramine in aq NaOH, refluxing, neutralizing with concd HCl & evaporating the filtrate to dryness (Ref 2). McKay et al (Ref 3) prepd the compd by aq hydrolysis of 1-nitro-2-nitriminoimidazolidine

Refs: 1) Beil-not found 2) R.H. Hall & G.F. Wright, JACS **73**, 2215-16(1951) & CA **46**, 1989(1952) 3) A.F. McKay et al, JACS **76**, 6373(1954) & CA **49**, 15862(1955)

N,N^1-Dinitro-diaminoethane; N,N^1-Dinitroethylenediamine or Ethylenedinitramine (abbrd EDNA) and HALEITE (called 1.2-Dinitramino-äthan;N,N^1-Dinitro-athylendiamin; and Äthylenedinitramin in Ger), $O_2N.NH.CH_2.CH_2.NH.NO_2$; mw 150.10, N 37.33%; col ndls, mp 174-78° dec; d 1.75 at 20°;Q_{comb} 2506 cal/g at const press; readily sol in alc & acet; mod sol in w; sl sol in eth, chlf & benz; was prepd by aq hydrolysis of N,N^1-dinitroethylene urea (Refs 1 & 3). Hall & Wright (Ref 4) converted β-aminoethylnitramine to N,N^1-Dinitrodiaminoethane via the dichloramine deriv

The expl & other props of EDNA are reported in Ref 6 and by Rinkenbach (Refs 5 & 7):

Booster sensitivity, 2.09inches wax for 50% detonations (TNT value 1.68inches)
Brisance by sand test, 52.3g sand crushed (TNT = 48)
Compatibility with metals, most metals are unaffected in contact with dry expl; most are heavily corroded by wet expl, Al & stainless steel are unaffected
Detonation rate, 7570m/sec at d 1.49g/cc
Explosion temperature, 216° in 1sec, dec 189° in 5secs, and 178° in 10secs
Flammability index, 138 (TNT = 100)
Friction pendulum, unaffected by either fiber or steel shoe
Gas evolved on explosion, 908cc/g
Heat of combustion, 2477cal/g at const vol
Heat of explosion, 1276cal/g
Heat of formation, 134cal/g
Heat test at 100° C, 0.2% loss 1st 48hrs, 0.3% loss 2nd 48hrs, no expln in 100hrs
Hygroscopicity, 0.1%
Impact sensitivity, with 2kg wt, 14inches for 17mg sample by PA App; 48cm for 20mg sample by BM App
International heat test at 75°C, 0.01% loss in 48hrs
Power by ballistic mortar test, 139% of TNT; and by Trauzl test 122% TNT
Rifle bullet impact test, 60% partials, 20% burned and 20% unaffect in 5 trials
Sensitivity to initiation, 0.21g MF or 0.13g LA reqd for 0.4g sample
Stability in storage, withstands storage at 50°C
Vacuum stability test, 0.5cc gas evolved in 40hrs at 100°C, 1.5cc at 120°C snd 11+ at 150°C
Volatility, nil at RT
Uses: Haleite (named for the late Dr G.C. Hale of PA in recognition of his studies on EDNA) is a HE used as pressed chge in boosters; and in 55/45 mixt with TNT as a cast HE chge, called EDNATOL, for projectiles & bombs. Hale (Ref 2) also patented its use with oxidg agents such as $KClO_3$ & NH_4ClO_4

The lead salt of Haleite is as sensitive to impact as MF, but it cannot be detonated by flame or stab action. It is therefore not suitable for use in blasting caps or detonators. The Ag, Na & K salts are also known (Ref 7)

Refs: 1) Beil **4**, 572 & (569) 2) G.C. Hale, USP 2011578(1935) & CA **29**, 6762(1935) 3) G.V. Caesar & M. Goldfrank, USP 2400288(1946) & CA **40**, 4526(1946) 4) R.H. Hall & G.F. Wright, JACS **73**, 2215 (1951) & CA **46**, 1989(1952) 5) W.H. Rinkenbach, in Kirk & Othmer **6**(1951), 23-24 6) W.R. Tomlinson & O.E. Sheffield, "Properties of Explosives of Military Interest", AMCP **706-177**(1963), 121-25 7) W.H. Rinkenbach, in Kirk & Othmer **8**(1966), 626-27

Diaminoethane Dichlorate, $(H_2N.CH_2.CH_2.NH_2).2HClO_3$, an expl listed as Compd No. 16 in ADL PureExplCompds, Part 1(1947), p 72. No props of this compd are reported

Di(aminoethyl)-amine or Diethylenetriamine. See Bis(aminoethyl)-amine and Derivatives in this Encycl, Vol 2, p B128-L

N,N'-Di(β-aminoethylamino)-ethane or N,N'-Di(β-aminoethyl)-ethylenediamine. See 1,2-Bis(2^1-aminoethylamino)-ethane, Vol 2, p B129-L

Diaminoethylene Dichlorate or Ethylenediaminodichlorate, $O_3ClH.H_2N.CH_2.CH_2.NH_2.HClO_3$, an expl listed as Compd No. 16 in ADL PureExplCompds, Part 1(1947), p 72. No props of this compd are reported

N,N'-Di(β-aminoethyl)-ethylenediamine; 1,4,7,10-Tetrazadecane or Triethylenetetramine abbr as **TETA.** See Bis(aminoethylamino)-ethane in Vol 2, p B129-L

sym-Di(aminoethyl)-urea. See 1,3-Bis(1-aminoethyl)-urea in Vol 2, p B130-L

N,N'-Di(β-aminoethyl)-propanediamine. See Bis(aminoethylamino)-propane in Vol 2, p B130-L

1,3-Diaminoguanidine (called N.N'-Diaminoguanidin in Ger), $H_2N.NH.C(:NH).NH.NH_2$; mw 89.11, N 78.60%; known in the form of its salts, ie *Hydrobromide*, $CH_7N_5 + HBr$, pltlts (from w), mp 162-67° dec; *Hydrochloride*, $CH_7N_5 + HCl$, crysts (from alc), mp 176-85° dec; and *Nickel*, $Ni(CH_6N_5)$, dk-red crysts, very unstable. Other props & methods of prepn are given in Beil
Ref: Beil **3**, 122, (57), [97] & {232}

Diaminoguanidine Azide, $N_3.C(:NH).NH.NH_2$; mw 100.09, N 83.97%; compd predicted by reduction of azidonitroaminoguanidine, $N_3C(:NH).NH.NO_2$, using Na hydrosulfite, however, nitroguanidine, $H_2N.C(:NH).NH.NO_2$, always resulted (Ref 2)
Refs: 1) Beil-not found 2) F.L. Scott et al, JApplChem(London) **2**, 368(1952)

Di-(aminoguanidinium)-1,6-dinitro-2-(aminoguanyl)-biguanidine. See Bis-(aminoguanidinium)-1,6-dinitro-2-(aminoguanyl)-biguanidine in Vol 1, p A214-R

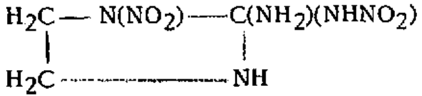
Di(aminoguanidinium)-1,6-dinitrobiguanidine. See Bis(aminoguanidinium)-1,6-dinitroguanidine in Vol 1, p A214-R

Diaminohydroxypropane. See Diaminopropanol

Diaminohydroxytriazine Picrate. See Ammeline Picrate in Vol 1, p A274-R

Diaminoimidazolidine, $C_3H_{10}N_4$, may be considered as a parent compd of the following substance:

(2-Amino-2-nitramino-1-nitro)-imidazolidine,



$$\begin{array}{l} H_2C-N(NO_2)-C(NH_2)(NHNO_2) \\ \;| \qquad\qquad\qquad\quad | \\ H_2C-\!\!-\!\!-\!\!-\!\!-\!\!-\!\!-\!\!-NH \end{array}$$

; mw 192.15 N 43.68%. Cryst, mp 184.8-185.3° with decompn. Was prepd from 1-nitro-2-nitriminoimidazolidone-2 (Ref 3) by the method described in Ref 2

Refs: 1) Beil-not found 2) A.F. McKay & G.F. Wright, JACS **70**, 3990(1948) & CA **43**, 2203(1949) 3) R.H. Hall, A.F. McKay & G.F. Wright, JACS **73**, 2205 & 2207(1951); CA **46**, 1988(1952)

DIAMINOMETHANE AND DERIVATIVES

Diaminomethane or Methylenediamine (called Diaminomethan or Methylendiamin in Ger), $H_2N.CH_2.NH_2$; mw 58.08, N 48.23%; exists in the form of salts, ie *Dihydrochloride*, $CH_6N_2 + 2HCl$, prisms, dec on soln in w; *Dinitrate*, $CH_6N_2 + 2HNO_3$, prisms expl mildly on heating w/o melting; *Stannic Chloride*, $CH_6N_2 + 2HCl + SnCl_4$, prisms, dec above 300° w/o melting; and *Sulfate*, $CH_6N_2 + H_2SO_4$, crysts, dec 183-93°
Ref: Beil **1**, (306), [648] & {2596}

Mononitrodiaminomethane, $CH_5N_3O_2$, was not found in Beil or in CA thru 1961

N,N'-Dinitro-diaminomethane; N,N'-Dinitromethanediamine; Dinitraminomethane or N,N'-Dinitromethylenediamine (called Dinitraminomethan or Methylendinitramin in Ger), $O_2N.HN.CH_2.NH.NO_2$; mw 104.07, N 53.84%; crysts (from eth), mp 98-106° dec; was obtd from methylene-bis-N-acetamid by nitration & subsequent hydrolysis (Refs 1 & 2). Alternate procedures are given by Sauer & Follett (Ref 8) & Sauer (Ref 12). Its decompn in aq soln s was studied by Lamberton et al (Ref 3) and by Barrott et al (Ref 6); its dissociation constant measured by Lindley & Speakman (Ref 4). Other props are reported by Jones & Thorn (Ref 5), Tobin et al (Ref 7), Krc (Ref 10), Reed (Ref 12) and Pristera et al (Ref 13)

Sauer (Ref 9) stabilized Dinitrodiaminomethane at a temp below its mp by coating with 0.1 to 2.0% polynitrophenol or polynitro aromatic carboxylic acid by means of absorption, recrystn or evapn from a nonsolvent. Thus, 98% of the pure compd & 2% Picric Acid were mixed & benz added to form a slurry. After the mixt was dried under vacuum, tests at 75° showed improved stability. Other examples using 9/1 ethylene chloride/iso-propanol mixt as a solv; and styphnic acid, 2,4-dinitrophenol & 3,5-dinitrobenzoic acid as stabilizers gave similar results

Glowiak (Ref 15) reported the following expl & other props of Dinitrodiaminomethane:
Detonation rate 8864m/sec
Explosion temp 217°
Impact sensitivity 100% (PA = 100%)
Minimum detonating chge 0.005g LA
Weight loss at 60° 4% in open vessel
after 500hrs, 2.5% in closed vessel
This compd is considered similar to PETN as regards its expl characteristics

Glowiak (Ref 15) also detd some props of the Pb salt of Dinitrodiaminomethane:
Explosion temp 195°
Impact work 0.0584kg m/sq cm,
fired from a distance
of 2-3cm
Melting point 40°

Water absorption, % at	80%RH	100%RH
hrs 24	0.11	0.14
48	0.16	.37
76	0.20	0.50
100	0.23	0.68
200	0.30	0.80
500	0.38	0.84
1000	0.52	0.93

Weight loss at 60°, %	Open Vessel	Sealed Vessel
hrs 24	0.24	0.0
48	0.38	0.05
76	0.48	0.10
100	0.52	0.15
200	0.54	0.15
300	0.56	0.15
400	0.58	0.15
500	0.60	0.15

It was also found that the Pb salt showed no absorption band, unless acidified, while the dinitro compd had an absorption band at $1582cm^{-1}$

Some props of the Pb salt & other salts of the dinitro deriv were detd by Piskorz & Urbański (Ref 14) as follows:

Explosion by 2kg falling wt, Salt	Height, cm, for 50% explns
Pb(II)	12
Hg	10
Ag	10
Ba	insensitive
Na	insensitive
Ignition point	**°C**
Pb(II)	238°
Hg	195 deflgr
Ag	195 explodes
Ba	162 slow decomp
Na	198 dec

Data similar to the above were obtd and compared to the props of the salts of methylenediisonitramine, $HO.N:N(:O).CH_2.N(:O):N.OH$

Refs: 1) Beil **1**, {2606} 2) F. Chapman, JCS **1949**, 1634 & CA **41**, 1411(1950) 3) A.H. Lamberton et al, JCS **1949**, 1650-56 & CA **44**, 1413(1950) 4) C. Lindley & J.C. Speakman, JCS **1949**, 1657-58 & CA **44**, 1413(1950) 5) R.N. Jones & G.D. Thorn, CanJRes **27B**, 829, 836, 838, 844(1949) & CA **44**, 2848(1950) (UV absorption spectra) 6) J. Barrott et al, JCS **1953**, 1998 & CA **48**, 10541(1954) 7) M.C. Tobin et al, JACS **76**, 3249-53(1954) & CA **49**, 8806(1955)(Thermal decompn & IR spectra 8) C.W. Sauer & R.P. Follett, JACS **77**, 2560-61(1955) & CA **50**, 4008(1956) 9) C.W. Sauer, USP 2786078(1957) & CA **51**, 9163(1957) 10) J. Krc, Jr, AnalChem **30**, 1301(1958) & CA **52**, 14276(1958)(Crystallographic data) 11) C.W. Sauer, USP 2856429(1958) & CA **53**, 5128(1959) 12) R. Reed, Jr, JOC **23**, 496(1958) & CA **53**, 8161(1959)(Reactions with diaminoalkanes) 13) F. Pristera et al, AnalChem **32**, 495(1960) & CA **54**, 12585(1960)(IR absorption spectra) 14) M. Piskorz & T. Urbański, BiulWojskowejAkadTechn im Jaroslawa Dabrowskiego (Warsaw) **8**, No 84, 112-15(1959) & CA **54**, 17012(1960) 15) B. Glowiak, ibid **9**,77-82 (1960)(Engl summary) & CA **54**, 21761(1960)

NOTE: Higher nitrated derivs of Diaminomethane were not found in Beil or in CA thru 1961

Di(aminomethyl).propanediamine and Derivatives. See Bis(aminomethyl)-propanediamine in Vol 2, p B130-R

Its nitrated derivative:

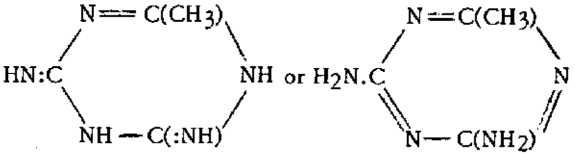
2,2-Di(nitraminomethyl)-1,3-propanedinitramine or **Pentaerythrityltetranitramine,** $C(CH_2.NH.NO_2)_4$; mw 312.21, N 42.29%, OB to CO_2 -41%. This compd is listed by ADL (Ref 2) as compd No 232 and is called *Tetra(nitraminomethyl)methane*. The following props are reported by ADL, now declassified (Ref 2):

Melting point 167°
Heat of combustion (calcd) 812.6 kcal/mol
Heat of explosion (calcd) 303.4 kcal/mol
Impact sensitivity (FI) = Tetryl
Power by ballistic mortar 129% TNT
Power by Trauzl test 137% TNT
Temp developed on expln (calcd) 3247°K
Thermal stability satisfactory

This expl compd is not found in Beil or in CA

Refs: 1) Beil-not found 2) ADL, PureExplCompds, Part 1(1947), pp 35, 148, 178 and Part 4(1952), p 586

2,4-Diamino-6-methyl-s-triazine (called 4,6-Diamino-2-methyl-1,3,5-triazin; 4,6-Diimino-2-methvl-tetrahydro-1,3,5-triazin or Acetoguanamin in Ger),



mw 125.14, N 55.97%; shiny wh pltlts or ndls (from w), mp 265-74°; readily sol in hot w or alc; less sol in cold w; can be prepd by heating cyanoguanidine & acetamidine hydrochloride or acetonitrite in a tube at 225-30°; or by heating biguanide with acetanhydride and by other methods given in the Refs

Many addn compds & salts have been prepd, some of which are unstable on heating. For example, the *Nitrate*, $C_4H_7N_5 + HNO_3$ prisms, explodes on heating. Many other reactions & props of the Diamino-methyl-s-triazine are found in the Refs listed

Refs: 1) Beil **26**, 229, (66) & [121] 2) A. Ostrogovich & G. Gheorghiu, Gazz **60**, 659(1930) & CA **25**, 958(1931)(Prepn) 3) W.Zerweck & W. Brunner, USP 2302162 (1942) & CA **37**, 2016(1943)(Prepn & also of other guanamine derivs) 4) J.K. Simmons & W.I. Weaver, USP 2408694 (1946) & CA **41**, 1240(1947)(Prepn) 5) D.E. Nagy, USP 2446980(1948) & CA **42**, 8216(1948)(Prepn) 6) C. Grundmann & E. Beyer, ChemBer **83**, 452(1950) & CA **45**, 2489(1951)(Prepn) 7) J.S. Mackay, USP 2527314(1950) & CA **45**, 2514(1951)(Prepn) 8) Amer Cyanamid, BritP 642409(1950) & CA **46**, 146(1952)(Prepn) 9) J.R. Dudley, JACS **73**, 3007(1951) & CA **46**, 1005(1952) (Dissociation constant) 10) S. Birtwell, JCS **1952**, 1279 & CA **47**, 1135(1953) (Prepn) 11) R.C. Hirt & D.J. Salley, JChemPhys **21**, 1181(1953) & CA **47**, 10353 (1953)(UV absorption spectra) 12) K. Rehnelt, Monatsh **86**, 653(1955)(Prepn & paper-chromatographic separation of a series of triazine derivs) 13) B. Bann et al, ComptRend27^{e}CongrInternChimInd, Brussels **1954**, 3; IndustrieChimBelge **20**, Spec No, 342-45(1955)(English & CA **50**, 11053(1956) (Improved prepn)

Diamino-oxalic Acid Dihydrazide; Oxalhydrazone Oxamide (called Oxaldiimidsäure-dihydrazid; "carbohydrazimin" or "cyanhydrazin" in Ger), $(H_2N.N:)(H_2N)C.C(NH_2)(:N.NH_2)$ or $(H_2N.NH)(HN:)C.C(:NH)(NH.NH_2)$ mw 116.13, N 72.37%; col ndls (from dil alc), mp dec & becomes orn-yel at 140° and does not melt up to 250°; readily sol in w; sl sol in alc; insol in eth; obtd by combining cyanogen with hydrazine hydrate (Refs 1, 2 & 3). This compd is also obtd from dithioxamide in 20% alc & $N_2H_4.H_2O$ (Ref 4)

Salts of the compd were prepd by Dedichen (Ref), some of which are expl:
Hydrochloride, $C_2H_8N_6.2HCl$, yel ndls, explodes by impact but not by heating
Sulfate, $C_2H_8N_6.H_2SO_4$, wh powder
Nitrate, $C_2H_8N_6.2HNO_3$, mother-of-pearl crysts. Many other salts, similar to those of NH_3, having characteristic colors are also formed

Refs: 1) Beil **2**, 560 2) A. Angeli, Gaz **23** II, 103(1893) & JCS **66** I, 150(1894) 3) T. Curtius & G.M. Dedichen, JPraktChem **50**, [2], 254(1894) & JCS **68** I, 30(1895) 4) G.M. Dedichen, AvhandlNorskeVidenskaps-AkadOslo I, Mat-Naturv Klasse **1936**, No 5, 42 pp & CA **31**, 4985(1937)

DIAMINOPHENOL AND DERIVATIVES

Diaminophenol or Diaminohydroxybenzene (called Diamino-oxy-benzol or Diamino-phenol in Ger), $(H_2N)_2.C_6H_3.OH$; mw 124.14, N 22.57%. The following isomers are known:
2,3-Diaminophenol, brown lfts (Ref 1)
2,4-Diaminophenol, lfts, mp 78-80° dec (Ref 2,
2,5-Diaminophenol, as Hydrochloride $C_6H_8N_2O + 2HCl$, ndls, (Ref 3)
2,6-Diaminophenol, as Hydrochloride, $C_6H_8N_2O + 2HCl$, ndls, or Sulfate, $C_6H_8N_2O + H_2SO_4$, lt-yel nds (Ref 4)
3,4-Diaminophenol, mp 167-68°, dec under rapid heating (Ref 5)
3,5-Diaminophenol, prisms, mp 168-70° (Ref 6)

Other props & methods of prepn of Diaminophenols are given the Refs

Refs: 1) Beil **13**, (203) & [308] 2) Beil **13**, 549, (204) & [308] 3) Beil **13**, 553, (208) & [312] 4) Beil **13**, 563, (209) & [315] 5) Beil **13**, 564 & (210) 6) Beil **13**, 567

Mononitrodiaminophenol, $(H_2N)_2C_6H_2(NO_2).OH$ mw 169.14, N 24.85%. Two isomers are known:
4-Nitro-2,6-diaminophenol, dk-yel ndls or thin lfts + H_2O (from water), mp 169° dec (Ref 3) 260° (Ref 1); sol in acet, ethyl acetate, benz, alc, water, chlf, dil acids & dil alk; was prepd by passing H_2S into Picric Acid in hot dil NH_4OH until no ppt was given by addn of dil HCl
6-Nitro-2,4-diaminophenol, compd mentioned as an ingredient of hair dye. No other info is reported (Ref 4)

Addnl info on the mononitro deriv may be found in the Refs

Refs: 1) Beil **13**, 563, (209) & [316] 2) A. Korczyuski & S. Plasecki, AnzAkadWiss-Krakau **1917**, 176 & CA **16**, 2124(1922) 3) J. English, Jr et al, JACS **62**, 354(1940) & CA **34**, 2348(1940) 4) Société Monsavon-l'Oréal, FrP 1137922(1957) & CA **53**, 18405(1959)

Dinitrodiaminophenol, $C_6H_6N_4O_5$, derivs were not found in Beil or in CA thru 1961

2,4,6-Trinitro-3,5-diaminophenol, $(H_2N)_2C_6(NO_2)_3.OH$; mw 259.14, N 27.03%; yel crysts (from glac AcOH), mp 270°; was obtd by Blanksma (Refs 1 & 2) by treating pentanitrophenol, tetranitroresorcin-monomethylether, or 5-chloro-2,3,4,6-tetranitrophenol (Ref 3) with alcoholic ammonia

No information is given in the literature as to the expl nature of this compd

Refs: 1) Beil **13**,568 2) J.J. Blanksma, Rec **21**, 263(1902) 3) J.J. Blanksma, Rec **27**, 36(1908)

DIAMINOPROPANE AND DERIVATIVES

Diaminopropane; Propanediamine; N-Methylethylenediamine and Trimethylenediamine; $C_3H_{10}N_2$, mw 74.13, N 37.80%. Two isomers are known:

1,2-Diaminopropane (called 1,2-Diaminopropan or Propylendiamin in Ger), $CH_3.CH(NH_2).CH_2.NH_2$; exists as d-form, l-form & dl-form all of which form numerous cryst salts, some of which are unstable on heating (Ref 1)

1,3-Diaminopropane (called 1,3-Diamino-propan or Trimethylendiamin in Ger), $H_2N.CH_2.CH_2.CH_2.NH_2$; liq, fr p 23.5° bp 131°; forms numerous salts, the perchlorate, $C_3H_{10}N_2 + 2HClO_4$, explodes when heated to 310° (Ref 2)

N-Methyl-ethylenediamine [called N-Methyl-äthylendiamin or Methyl-(2-amino-äthyl)-amin in Ger] $CH_3.NH.CH_2.CH_2.NH_2$; liq, bp 115-117°; forms numerous salts, such as the Cu perchlorate, $[Cu(C_3H_{10}N_2)_2](ClO_4)_2$, blue-violet crysts, defglr on heating (Ref 3)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **4**, 257, 417, [697] & {547-51}
2) Beil **4**, 261, (419), [699] & {552-54}
3) Beil **4**, (415), [689] & {512}

Mononitrodiaminopropane, mw 119.13, N 35.28%:

N^1-Nitro-1,2-diaminopropane [called N^1-Nitro-1,2-Propanediamine in CA and 2-Amino-3-nitraminopropane by McKay & Viron (Ref 2)] $CH_3.CH(NH_2).CH_2.NH.NO_2$, crysts(from w + alc), mp 239.5-40.9° dec; prepd by refluxing 1-nitro-2-nitramino-4-methyl-2-imidazolidine with water & hydrolyzing completely; or by refluxing of 1-nitro-4-methyl-2-imidazolidone and 1-(1-methyl-2-nitraminoethyl)-3-nitrourea with water until gassing ceases (Ref 2)

N-Nitro-N-methyl-ethylenediamine Salts [called 3-Nitrazabutylammonium Nitrate or Chloride by Frankel & Klager (Ref 3)], $CH_3.N(NO_2).CH_2.CH_2.NH_2.HX$, where X = ONO_2 or Cl, have been reported in the literature (Ref 3)

Refs: Beil-not found 2) A.F. McKay & S.J. Viron, JACS **72**, 3965-66(1950) & CA **45**, 2936(1951) 3) M.B. Frankel & K. Klager, JACS **78**, 5429(1956)

1,2-Dinitraminopropane or Propylenedinitramine, $CH_3.CH(NH.NO_2).CH_2.NH.NO_2$; mw 164.13, N 34.14%; crysts (from w), mp 109.8-12.0°; was first prepd by Bachmann in poor yield by hydrolysis of propylene dinitrourethane (Ref 2), and in 61% yield by Bloomquist using dry NH_3 as the hydrolytic agent (Ref 3); McKay & Manchester (Ref 4) prepd it by hydrolysis of 1,3-dinitro-4-methyl-1,3-diazacyclopentane

Bloomquist (Ref 3) reported the following props:

Explosion temp - ignited at 355°
Hygroscopicity -- 0.76% at 30°C & 90%RH; 2.42% at 30°C & 100%RH
Impact sensitivity - 63cm vs 48-50 for RDX
Power - 116% TNT
Stability tests, International at 75° - 2.0% loss in 24 hrs

Some addnl props & info on this compd may be found in the following Refs

Refs: 1) Beil-not found 2) W.E. Bachmann, "EDNA and Related Compounds", OSRD 819 (1942) 3) A.T. Bloomquist et al, "Certain Aliphatic Nitramines and Related Compounds" OSRD 4134 (1944) pp 15-16, 70-89
4) A.F. McKay & D.F. Manchester, JACS **71**, 1970(1949) & CA **43**, 9065(1949) 5) A.F. McKay et al, JACS **72**, 3659(1950) & CA **45**, 2888(1951)(Prepn)

1,3-Dinitraminopropane; N,N -Dinitrotrimethylenediamine; or N,N' -Dinitro-1,3-propanediamine, $O_2N.HN.CH_2.CH_2.CH_2.NH.NO_2$, prisms (from w), mp 67.2-68°; readily sol in w & alc; less sol in eth & chlf; obtd by hydrolyzing 1,3-dinitro-1,3-diazacyclohexanone-2 (Ref 2). Its decompn in aq soln was studied by Lamberton et al (Refs 4 & 8); its dissociation constant was reported by Lindley & Speakman (Ref 5), and its UV absorption spectra reported by Jones & Thorn (Ref 6) and McKay & Sandorfy (Ref 7). The expl props of this dinitro deriv were not found in the literature

Refs: 1) Beil **4**, 573 2) A.F. McKay & D.F. Manchester, JACS **71**, 1973(1949) & CA **43**, 9066(1949) 3) D. Woodcock, JCS **1949**, 1637 & CA **44**, 1411(1950)(Prepn) 4) A.H. Lamberton et al, JCS **1949**, 1654 & CA **44**, 1413(1950) 5) C. Lindley & J.C. Speakman, JCS **1949**, 1657 & CA **44**, 1413(1950) 6) R.N. Jones & G.D. Thorn, CanJRes **27B**, 828(1949) & CA **44**, 2848 (1950) 7) A.F. McKay & C.Sandorfy, CanJChem **31**, 42(1953) & CA **47**, 6765(1953) 8) J. Barrott et al, JCS **1953**, 1998 & CA **48**, 10541(1954) 9) L. Fishbein & J.A. Gallaghan, JACS **76**, 3217(1954)& CA **49**, 8991(1955)(Prepn)

N,N -Dinitro-N-methyl-ethylenediamine; N-Methyl-ethylenedinitramine; or 1,4-Dinitro-1,4-diazapentane, $CH_3.N(NO_2).CH_2.CH_2.NH.NO_2$; col crysts (from w), mp 121-22°; readily sol in w; less sol in eth & chlf; was first obtd by Franchimont & Klobbie (Refs 1 & 2), in addn to dimethyl-ethylenedinitramine, by action of methyl iodide on ethylenedinitramine. Some other methods of prepn & props are given in the Refs. The expl props were not reported

Refs: 1) Beil **4**, 573 2) A.P.N. Franchimont & E.A. Klobbie, Rec **7**, 347(1888) & JCS **56**, 492(1889) 3) R.N. Jones & G.D. Thorn, CanJRes **27B**, 830, 837(1949) & CA **44**, 2848(1950)(UV absorption spectra) 4) N. Allentoff & G.F. Wright, ActaCryst **6**, 8(1953) & CA **47**, 6733(1953)(Prepn) 5) M.W. Kirkwood & G.F. Wright, JOC **18**, 640(1943) & CA **48**, 6968(1954)(Prepn) 6) L.W. Kissinger & M. Schwartz, JOC **23**, 1344(1958) & CA **53**, 15968(1959)(Prepn)

NOTE: Higher nitrated derivs of Diaminopropane were not found in Beil or in CA thru 1961

DIAMINOPROPANOL AND DERIVATIVES

Diaminopropanol or Diaminohydroxypropane, $C_3H_{10}N_2O$; mw 90.13, N 31.08%. Three isomers are listed in the literature:

1,3-Diamino-1-propanol [called 1,3-Diaminopropanol-(1) or α-Hydroxy-trimethylenediamin in Ger], $H_2N.CH_2.CH_2.CH(OH).NH_2$, used to remove org sulfur compds of mercaptan type from mineral oils (Refs 3 & 4)

1,3-Diamino-2-propanol [called 1,3-Diaminopropanol-(2); 1,3-Diamino-2-hydroxy-propan; or β-Hydroxy-trimethylenediamin in Ger], $H_2N.CH_2.CH(OH).CH_2.NH_2$; known in the form of salts, some of which are unstable (Ref 1)

2,3-Diamino-1-propanol [called 2,3-Diaminopropanol-(1); 2,3-Diamino-1-oxy-propane; β.γ-Diamino-propyl-alcohol; or γ-Oxy-propylendiamin in Ger], $H_2N.CH_2.CH(NH_2).CH_2.OH$; exists as dihydrobromide salt in 3 forms (Ref 2)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **4**, 290, [739] & {766} 2) Beil **4**, (436) & [736] 3) Beil **4**, {875} 4) A.Y. Mottlau, USP 2287118(1942) & CA **37**, 254(1943)

Mononitro, $C_3H_9N_3O_3$, and *Dinitro,* $C_3H_8N_4O_5$, derivs of Diaminopropanol were not found in Beil or in CA thru 1961

1,3-Dinitramino-2-propanol Nitrate or 1,3-Dinitramino-2-nitroxy-propane [called 1,5-Dinitro-3-nitroxy-1,5-diazapentane by Jones & Thorn (Ref 4)], $O_2N.NH.CH_2.CH(ONO_2).CH_2.NH.NO_2$; mw 225.49, N 31.22%, OB to CO_2 -17.8%; crysts (from Nitromethane), mp 160.5° (Ref 3) & 164-65° dec (Ref 5), defgr at 230°; can be prepd by reaction of 1,3-diamino-2-propanol with $ClCO_2C_2H_5$ followed by nitration & ammonolysis (Ref 5)

This compd is a very powerful explosive (142% of TNT by Ballistic Mortar Test); Impact Sensitivity, using Bruceton No 3

Machine - 50% positive with 5kg weight at 24cm drop (vs 20cm for PETN & 50cm for RDX); Thermal Stability - not acid in 300 minutes at 100°, but acid in 105 mins at 135°; Vacuum Stability at 100° - 1.99 to 4.36cc for 5g in 48hrs; Hygroscopicity at 100%RH - gains 0.02% (Ref 1a)

Refs: 1) Beil-not found 1a) Blatt, OSRD **2014**(1944) 2) ADL, PureExplCompds, Part 1(1947) pp 125 & 148 (Listed as Compd No 42) 3) A.F. McKay & D.F. Manchester, JACS **71**, 1970-73(1949) & CA **43**, 9065(1949) (Prepn) 4) R.N. Jones & G.D. Thorn, CanJRes **27B**, 836(1949) & CA **44**, 2848(1950) (UV absorption spectra) 5) A.T. Bloomquist & F.T. Fiedorek, USP 2485855(1949) & CA **44**, 3517(1950)(Prepn & props) 6) Sax(1957), 541(Toxicity details unknown)

*NOTE:*Higher nitrated derivs of Diaminopropanol were not found in Beil or in CA thru 1961

DIAMINOTETRAMETHYLBENZENE AND DERIVATIVES

Diaminotetramethylbenzene, Tetramethyldiaminobenzene or Tetramethylphenylenediamine (called Tetramethyl-phenylenediamine in Fr and Tetramethyl-phenylendiamin in Ger), $C_6H_4[N(CH_3)_2]_2$; mw 164.24, N 17.06%. Three isomers are known:

1,2-(Diamino-N,N,N′,N′-tetramethyl)-benzene, stable oil when dry, having a camphor-like odor, bp 215-16° at 759mm press; forms cryst salts (Ref 1)

1,3-(Diamino-N,N,N′,N′-tetramethyl)-benzene, liq, fr p -2°, bp 266.7° at 761mm press, d 0.992 at 15°; forms cryst salts (Ref 2)

1,4-(Diamino-N,N,N′,N′-tetramethyl)-benzene, lfts (from dil alc or petr eth), mp 52°, bp 260°, d 0.8795 at 99.3°; forms cryst salts (Ref 3)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **13**, 16 2) Beil **13**, 40 & (12) 3) Beil **13**, 74, (22) & [40]

Azido, $C_{10}H_{15}N_5$, and *Diazido,* $C_{10}H_{14}N_8$, derivs of Diaminotetramethylbenzene were not found in Beil or in CA thru 1961

Mononitrodiaminotetramethylbenzene, $O_2N.C_6H_3[N(CH_3)_2]_2$; mw 209.24, N 20.08%. Two isomers are reported in the literature:

4-Nitro-1,2-(diamino-N,N,N′,N′-tetramethyl)-benzene, crysts (from alc), mp 62-63°; was prepd from $2,5\text{-}Cl(O_2N)C_6H_3N(CH_3)_2$ and $NH(CH_3)_2$ in alc in an autoclave at 150° (Ref 4)

4-Nitro-1,3-(diamino-N,N,N′,N′-tetramethyl)-benzene, orn-red pltlts, mp 79-81°; was prepd from 3,4-dinitrodimethylaniline & dimethylamine in alc under pressure (Refs 1, 2 & 3)

Refs: 1) Beil **13**, [30] 2) P. van Romburgh, Rec **42**, 806(1923 CA **18**, 49 (1924) 3) A. Weissberger & J.R. Thirtle, USP 2652331(1953) & CA **48**, 12595(1954) (Use as a developer in color photography) 4) I.I. Levkoev et al, ZhObshchKhim **24**, 280(1954) & CA **49**, 4628(1955); JGenChem (Russia) **24**, 283(1954) & CA **49**, 8248(1955) (English translation)

Dinitrodiaminotetramethylbenzene, $(O_2N)_2C_6H_2[N(CH_3)_2]_2$; mw 254.24, N 22.04% Three isomers have been reported:

X,X-Dinitro-1,3-(diamino-N,N,N′,N′-tetramethyl)-benzene, crysts (from dil alc or glac AcOH); was obtd by nitration of N,N,N′,N′-tetramethyl-phenelene-1,3-diamine using dil H_2SO_4 & HNO_3 (Ref 1)

4,6-Dinitro-1,3-(diamino-N,N,N′,N′-tetramethyl)-benzene, yel nds (from chlf + alc), mp 191°; sol in chlf; sl sol in hot alc; was prepd by heating 1,3-dinitro-4,6-dichlorobenzene with dimethylaniline in alc; or from 2,4-dinitro-5-chloro-N,N-dimethylaniline & $NH(CH_3)_2$ (Ref 2)

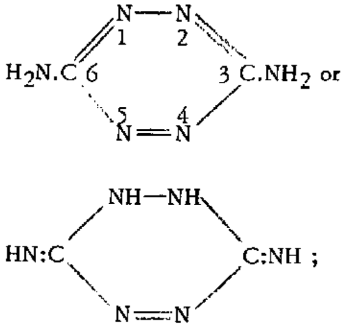
2,6-Dinitro-1,4-(diamino-N,N,N′,N′-tetramethyl)-benzene, long, slender deep purple ndls (from alc), mp 176°; was formed when 2,6-dinitrodimethyl-p-ansidine was heated in a sealed tube with excess dimethylamine in alc (Ref 3)

The expl props of the dinitro derivs were not reported

Refs: 1) Beil **13**, 60 2) Beil **13**, (16) 3) H.H. Hodgson & J.H. Crook, JCS **1936**, 1570 & CA **31**, 380(1937)

NOTE: Higher nitrated derivs of Diaminotetramethylbenzene were not found in Beil or in CA thru 1961

3,6-Diamino-sym-tetrazine (called 3,6-Diamino-1,2,4,5-tetrazin or 3,6-Diimino-1,2,3,6-tetrahydro-1,2,4,5-tetrazin in Ger),



mw 112.10, N 74.98%; orn-red microcrysts (from dioxane or water), mp sublimes 200-40°, 360° (dec); sl sol in w, alc, acet or ethyl acetate; insol in ether; readily dissolves in concd mineral acid from which it may be re-pptd unchanged on dilution with water; was prepd by an unequivocal synthesis in which 1,2,4,5-tetrazine-3,6-dicarbonyl azide was degraded thru Curtius rearrangement; also obtd by action of HNO_2 on diaminoguanidine or from action of dil NaOH on s-methylthiosemicarbazide (Ref 3). Lin et al (Ref 3) claim props distinctly different from those reported by Ponzio & Gastaldi (cited in Ref 1). See also Scott & Reilly (Ref 2)

The electronic structure of this compd detd by the method of antisymmetrical molecular orbitals (ASMO) has been reported by Paoloni (Ref 4)

Some of the salts of Diaminotetrazine are unstable and/or expl on heating (Ref 1):

Carbonate, $C_2H_4N_6 + H_2CO_3$, orn-yel pltlts, mp dec at 100° into CO_2 & water

Hydrobromide, $C_2H_4N_6 + HBr$, yel lfts, mp explodes ca 120°

Hydrochloride, $C_2H_4N_6 + HCl$, orn-yel lfts (from aq acet), mp dec ca 200°

Nitrate, $C_2H_4N_6 + HNO_3 + \frac{1}{2}H_2O$. orn-yel lfts (from w); anhyd salt, yel-brn crysts dec 180-82°

Oxalate, $2C_2H_4N_6 + C_2H_2O_4$, orn-red prisms, mp dec ca 205°

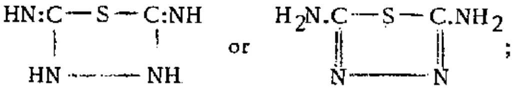
Picrate, $C_2H_4N_6 + C_6H_3N_3O_7$, yel amorph powd, mp expl

Refs: 1) Beil **26**, (130) 2) F.L. Scott & J. Reilly, Chem & Ind **1952**, 908 & CA **47**, 6886(1953) 3) C.H. Lin et al, JACS **76**, 427-30(1954) & CA **49**, 3207-08(1955) 4) L. Paoloni, Gazz **87**, 313-28(1957) & CA **51**, 14407(1957)

NOTE: See also 3,6-Diamino-dihydro-s-tetrazine

DIAMINOTHIADIAZOLE AND DERIVATIVES

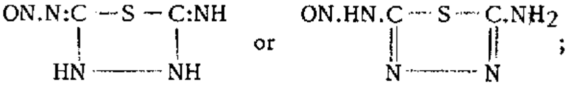
2,5-Diamino-1,3,4-thiadiazole (called 2,5-Diamino-1,3,4-thiodiazol or 2,5-Diimino-1,3,4-thiodiazolidin in Ger)



mw 116.08, N 48.27%; almost col prisms (from w), mp 210-12°; readily sol in w; was obtd by treating hydrazine-N,N'-bis-thiocarbonic acid amide with H_2O_2 in hot aq soln or with hydroxylamine. Other methods of prepn & props are given in Beil

Ref: Beil **27**, 667, (598) & [752]

5-Nitrosoamino-2-amino-1,3,4-thiadiazole,



mw 177.21, N 39.52%; brn crysts; mp explodes at 160-90°; diffc sol in alc; insol in w & eth; was prepd by treating the parent compd with isoamylnitrite in alc, or with $NaNO_2$ in acetic acid soln in the cold

Refs: 1) Beil **27**, (598) & [755] 2) R. Stollé & F. Fehrenbach, JPraktChem **122**, 300(1929)

NOTE: *Mononitro*, $C_2H_3N_5O_2S$, and *Dinitro*, $C_2H_2N_6O_4S$, derivs of Diaminothiadiazole were not found in Beil or in CA thru 1961

DIAMINOTOLUENE AND DERIVATIVES

Diaminotoluene or Toluenediamine (called Diamino-toluol; Methyl-phenylendiamin; or

Toluylendiamin in Ger), $(H_2N)_2C_6H_3.CH_3$; mw 122.17, N 22.93%. All possible isomers are known:

2,3-Diaminotoluene, crysts, mp 61-64°, bp 255° (Ref 1)

2,4-Diaminotoluene, ndls (from w) or rhmb prisms (from alc), mp 99°, bp 280-85° (Ref 2)

2,5-Diaminotoluene, pltlts (from benz), mp 64°, bp 273-74° (Ref 3)

2,6-Diaminotoluene, prisms (from w), mp 105°; prisms (from benz), mp 106° (Ref 4)

3,4-Diaminotoluene, lfts (from petr eth), mp 88.5-90°, bp 265° (Ref 5)

3,5-Diaminotoluene, liq, bp 283-85° (Ref 6)

$2,1^1$-Diaminotoluene, $H_2N.C_6H_4.CH_2.NH_2$, crysts, mp dec under distillation, given off NH_3 (Ref 7)

$3,1^1$-Diaminotoluene, liq, forms a cryst picrate (Ref 8)

$4,1^1$-Diaminotoluene, liq, bp 268-70°, d 1.08 at 20° (Ref 8)

All of the Diaminotoluenes form cryst salts and addn compds. Other props & methods of prepn are given in the Refs

Refs: 1) Beil **13**, 123, (39) & [60] 2) Beil **13**, 124 & (40) 3) Beil **13**, 144, (43) & [62] 4) Beil **13**, 148, (43) & [64] 5) Beil **13**, 148, (44) & [64] 6) Beil **13**, 164 7) Beil **13**, 165 8) Beil **13**, 174

Mononitrodiaminotoluene, $(H_2N)_2C_6H_2(NO_2).CH_3$ mw 167.17, N 25.14%. The following isomers are found in Beil:

5-Nitro-2,3-diaminotoluene, orn-red ndls (from dil alc), mp 185° & red ndls (from w), mp 175°; v sol in hot alc & AcOH; sol in hot w (Ref 1)

5-Nitro-2,4-diaminotoluene, yel ndls having a violet luster (from w), mp 154°; sl sol in hot w; sol in hot alc (Ref 2)

6-Nitro-2,4-diaminotoluene, red prisms (from w), mp 132° & orn-red crysts (from benz or petr eth), mp 130-31°; sol in dil HCl (Ref 3)

4-Nitro-2,5-diaminotoluene, red to dk-grn bronze luster ndls (from w), mp 173° (Ref 4)

5-Nitro-3,4-diaminotoluene, dk-red ndls (from w), mp 158°; sol in alc; sl sol in w (Ref 5)

Other props & methods of prepn of the mononitro derivs are given in the Refs

Refs: 1) Beil **13**, (39) & [60] 2) Beil **13**, 141, (42) & [62] 3) Beil **13**, 142 & [62] 4) Beil **13**, (43) 5) Beil **13**, [66]

Dinitro-diaminotoluene, $(H_2N)_2C_6H(NO_2)_2.CH_3$; mw 212.17, N 26.41%. The following isomers are reported in the literature:

3,5-Dinitro-2,4-diaminotoluene, golden-yel ndls, mp 240°; diffc sol in common org solvs; sol in NaOH; can be prepd by reaction of NH_3 on 2,4-dichloro-3,5-dinitrotoluene at 160° or on 2,4-dibromo-3,5-dinitrotoluene at 150°, and by heating on a steam bath alc NH_3 & 3,4,5-trinitro-2-methoxytoluene (Ref 1)

3,5-Dinitro-2,6-diaminotoluene, lt-brn crysts, mp 298°; v sl sol in alc; was prepd by heating 2,6-dibromo-3,5-dinitrotoluene with alc NH_3 at 150° (Ref 2)

2,4-Dinitro-3,5-diaminotoluene, yel crysts, mp 199° & orn-yel ndls (from alc), mp 210°; v sl sol in alc; was prepd by reaction of alc NH_3 on 3,5-dichloro-2,4-dinitrotoluene or 3,5-dibromo-2,4-dinitrotoluene at 125°, and on 3,5-dichloro-2,4-dinitrophenylacetic acid at 140° (Refs 3 & 5)

2,6-Dinitro-3,5-diaminotoluene, crysts, mp 196° (Ref 6); prepd by heating 6 ps of 3,5-dibromo-2,6-dinitrotoluene with alc NH_3 at 160° (Refs 4 & 5)

The expl props of the dinitro derivs were not detd

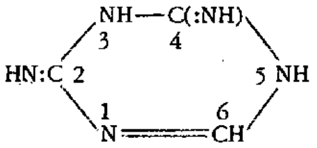
Refs: 1) Beil **13**, 142, (42) 2) Beil **13**, (43) 3) Beil **13**, 165 &[66] 4) Beil **13**, [66] 5) W. Borsche & W. Trautner, Ann **447**, 12, 13(1926) & CA **20**, 1222(1926) 6) S.S. Joshi & S.P. Gupta, JIndianChemSoc **36**, 329(1959) & CA **54**, 9921(1960)

Trinitrodiaminotoluene, $(H_2N)_2C_6(NO_2)_3.CH_3$; mw 257.17, N 27.24%. Only one isomer is known:

2,4,6-Trinitro-3,5-diaminotoluene, yel prisms (from alc), mp 222° & 225° (Ref 3); was prepd by heating 8 ps of 3,5-dibromo-2,4,6-trinitrotoluene with alc NH_3 under pressure on a steam bath (Refs 1 & 2). No expl props of this compd were detd

Refs: 1) Beil **13**, 165 2) A.W. Palmer, Ber **21**, 3501(1888) 3) S.S. Joshi & S.P. Gupta, JIndianChemSoc **36**, 329(1959) & CA **54**, 9921(1960)

2,4-Diamino-s-triazine or Formoguanamine (called 2,4-Diamino-1,3,5-triazin; 2,4-Diiminotetrahydro-1.3.5-triazin; or Formoguanamin in Ger),



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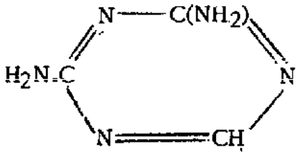


;

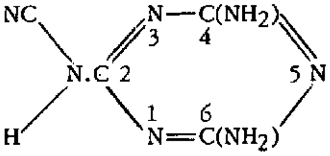
mw 111.11, N 63.04%; ndls (from w), mp 329°; mp 318°(Ref 4); sol in 20ps hot w, dil HCl or warm dil H_2SO_4; sl sol in alc; was prepd by heating guanidine formate to 200°; or by treating biguanide hydrochloride with abs alc KOH & chlf under cooling; as well as by heating anhyd biguanide sulfate with Na formiate to 150-200°; (Ref 1). Prepn of the pure compd in 80% yield is reported by Geigy (Ref 2). See also Yamashita (Ref 4) and Refs 7 & 9

The dissociation constant of 2,4-Diamino-s-triazine is 7.6 x 10^{-9} (Ref 3); its UV absorption spectra by Hirt & Salley (Ref 5). See also Paoloni (Ref 8)

Formoguanamine forms cryst salts & addn compds, some of which are probably expl. This compd has some diuretic action (Ref 10), and is useful as an intermediate in the prepn of dyes, synthetic resins & pharmaceuticals (Ref 6). Its expl props were not detd

Refs: 1) Beil **26**, 225 & (65) 2) J.R. Geigy, SwissP 255408(1949) & CA **44**, 174(1950) 3) J.R. Dudley, JACS **73**, 3007(1951) & CA **46**, 1005(1952) 4) M. Yamashita, JChemSocJapan, IndChemSect **54**, 786-88 (1951) & CA **48**, 3986(1954) 5) R.C. Hirt & D.J. Salley, JChemPhys **21**, 1181(1953) & CA **47**, 10353(1953) 6) D.W. Kaiser & J.J. Roemer, USP 2630433(1953) & CA **48**, 747-48(1954); USP 2658894(1953) & CA **48**, 12814(1954) 7) W.O. Fugate & L.C. Lane, Jr, USP 2653938(1953) & CA **48**, 12815(1954) 8) L. Paoloni, Gazz **84**, 729, 735(1954) & CA **50**, 1039(1956) 9) K. Odo et al, BullChemSocJapan **28**, 614-15(1955) & CA **50**, 11353 (1956) 10) G. Mauro, GazzMedItal **115**, 100-05(1956) & CA **50**, 17193-94(1956)

4,6;Diamino-s-triazine-2-carbamonitrile; 4,6-Diamino-2-cyanamino-s-triazine; (4,6-Diamino-s-triazine-2-yl)-cyanamide; or Cyanomelamine,



; mw 151.14, N 64.88%; col ndls, mp infusible; was prepd by addg $NaN(CN)_2$ & $H_2N.C(:NH)NH.CN$ to KOH in methyl cellosolve, heating at 130° for 2hrs, stirring, addg w, filtering, and acidifying with AcOH to ppt the product (Refs 2 & 3). See also Refs 4,5,6 & 7

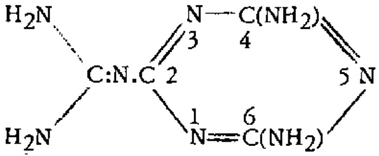
This compd & some of its derivs are useful as chem intermediates and as flameproofing agents

Refs: 1) Beil-not found 2) D.W. Kaiser & B.C. Redmon, USP 2510981(1950) & CA **44**, 9990(1950) 3) Ibid, USP 2537834(1951) & CA **45**, 4275(1951) 4) M. Kurabayashi JapP 4332(1952) & CA **48**, 4598(1954) 5) M. Kurabayashi & K. Yanagiya, ReptsGovtChemIndResInst, Tokyo **48**, 139-57(1953)(English summary); JChemSocJapan, IndChemSect **56**, 426-28 (1953) & CA **48**, 11429(1954)(Reaction products of dicyandiamide in the presence of NH_3) 6) Ibid, JChemSocJapan, IndChemSect **58**, 750-63(1955) & CA **50**, 12076(1956)(Ibid) 7) Ibid, ReptsGovtChemInd, ResInst, Tokyo **51**, 39-46(1956) & CA **50**, 14781(1956)(Ibid)

4,6-Diamino-s-triazine-2-ol. See Ammeline in Vol 1, p A273-R; and its **Dinitramino deriv,** Dinitroammeline, p A274-L

(4,6-Diamino-s-triazine-2-yl)-cyanamide. See 4,6-Diamino-s-triazine-2-carbamonitrile

(4,6-Diamino-s-triazine-2-yl)-guanidine; or Guanylmelamine



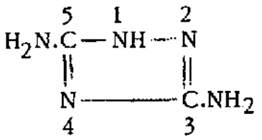
; mw 168.17, N 66.64%; crysts, mp 225°; was obtd as one of the products from reaction of cyanomelamine with KOH & NH_3. Its substituted derivs such as *Picrate*, mp 258-60° (Ref 2) & 290.5° (Ref 3) and *Dipicrate*, mp 257.7° (Ref 3) were also prepd as flameproofing agents. By depositing 10-40% of *Guanylmelamine Pyrophosphate* (obtd by reaction of guanylmelamine-HCl with di-Na di-H pyrophosphate) on a fabric, this compd was found to have fire retardant props for cellulosic materials (Ref 4)

The UV absorption spectra are reported by Hirt et al (Ref 5)

Refs: 1) Beil-not found 2) D.W. Kaiser & B.C. Redmon, USP 2537834(1951) & CA **45**, 4275(1951); BritP 653520(1951) & CA **45**, 10258(1951) 3) M.Kurabayashi & K. Yamagiya, ReptsGovChemIndResInst, Tokyo **48**, 139-57(1953)(English summary); JChemSoc-Japan, IndChemSect **56**, 426-28(1953) & CA **48**, 11429(1954) 4) A.M. Loukomsky, USP 2779691(1957) & CA **51**, 6181(1957) 5) R.C. Hirt et al, SpectrochimActa **1959**, 962-68 (English) & CA **54**, 8286(1960)

DIAMINOTRIAZOLE AND DERIVATIVES

3,5-Diamino-α-s-triazole, Aminoaminotriazole or Guanazole (called 3,5-Diimino-1,2,4-triazolidin; Urazol-diimid; 3,5-Diamino-1,2,4-triazol; or Guanazol in Ger),



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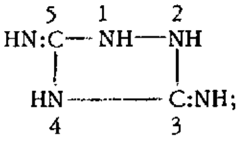
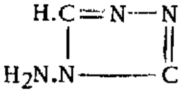


mw 99.10, N 70.68%; monoclinic prisms (from w), mp 204-06°; readily sol in w, giving an alk soln; mod sol in alc; insol in eth, chlf & benz; was first prepd in 1894 by heating at 100° for several hrs an alc soln of equimolar quantities of dicyandiamide & hydrazinohydrochloride; also prepd in good yield by heating a mixt of dicyandiamide & hydrazinehydrate on a w bath (Refs). Some derivs of Diaminotriazole are expl

Refs: 1) Beil **26**, 193 & (57) 2) G. Pellizzari, Gazz **24**(I), 491(1894) & JCS **66**(I), 518(1894) 3) K.A. Hofmann & O.Ehrhart, Ber **45**, 2733(1912) 4) R. Stollé & K. Krauch, JPraktChem **88**, 310-11(1913) 5) R. Stollé & W. Dietrich, JPraktChem **139**, 193-210 (1934) & CA **28**, 2714(1934)

3,4-Diamino-4H-1,2,4-triazole or 3,4-Diamino-γ-s-triazole,

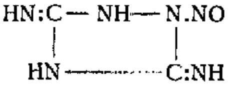


; large col plates (from alc), mp 216-18°; was prepd by refluxing 3,4-diamino-5-mercapto-4,1,2-triazole in alc with Raney Ni for 1 hr (Ref 2)

The *Nitrate*, $C_2H_6N_6O_3$, col ndls (from alc), mp 198-99°, was prepd by Lieber et al (Ref 3)

Refs: 1) Beil-not found 2) E. Hoggarth, JCS **1952**, 4816 & CA **48**, 5182(1954) 3) E. Lieber et al, JOC **18**, 227(1952) & CA **48**,1344(1954)

Mononitroso-3,5-diamino-α-s-triazole,



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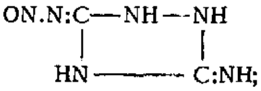
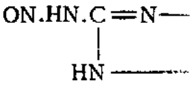


mw 128.10, N 65.61%; yel ppt; dec in hot w; insol in common org solvs; was prepd by treating guanazole with $NaNO_2$ in cold AcOH soln

Refs: 1) Beil **26**, (57) 2) R. Stollé & K. Krauch, JPraktChem **88**, 311(1913)

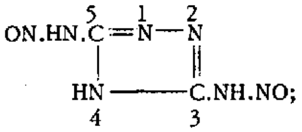
5-Nitrosamino-3-amino-γ-s-triazole,



; canary-yel crysts, mp dec ca 172°; warming with AcOH causing splitting off of HNO_2; with concd HCl it rearranges to 3-amino-1,2,4-5-diazonium chloride; was prepd from guanazole & NH_4NO_2 in alc (Ref 2)

Refs: 1) Beil-not found 2) R. Stollé & W. Dietrich, JPraktChem **139**, 193(1934) & CA **28**, 2714(1934)

3,5-Dinitrosamino-γ-s-triazole [called 3,5-Bis(nitrosoamino)-tetrazole in CA Coll Formula Index **14-40**(1920-46), p 181],



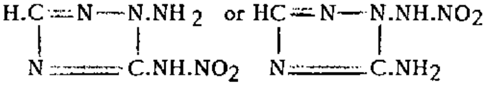
mw 157.10, N 62.42%; orn-red amor solid, mp dec 187°; was prepd from guanazole in 7N alc-HCl & NH_4NO_2

Reduction of the dinitroso compd with $SnCl_2$ & concd HCl gives *3-Amino-5-hydrazino-1,2,4-triazole di-HCl*, mp 217° (dec), which in an ice-cold aq soln & $NaNO_2$ gives **5-Azido-3-nitrosamino-1,2,4-triazole,** an ocher amor solid, mp detonates at 134° (Ref 2)

Compare with AMINOTRIAZOLE AND DERIVATIVES, Vol 1, of this Encycl, p A267-Rff

Refs: 1) Beil-not found 2) R. Stollé & W. Dietrich, JPraktChem **139**, 193(1934) & CA **28**, 2714(1934)

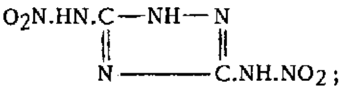
Mononitraminoaminotriazole, *3-Nitramino-2-amino-s-isotriazole,*



mw 144.10, N 58.33%; obtd from the nitrate deriv by treatment with H_2SO_4; its impact sensitivity expressed as FI is 47% of PA and its power & brisance are less than that of PA (Ref 2)

Refs: 1) Beil-not found 2) A.H. Blatt & F.C. Whitmore, "A Literature Survey of Explosives", OSRD **1085**(1942), p 62 3) Blatt, OSRD **2014**(1944)(Under Triazoles)

3,5-Dinitramino-α-triazole Salts,



this compd was isolated in the form of salts, of which the following were prepd by Henry et al (Ref 3):

Monoaminoguanidinium, $C_3H_9N_{11}O_4$, rosettes of ndls (from w), mp 180° dec

Monoammonium, $C_2H_6N_8O_4$, felted rosettes of fine wh ndls (from w), mp 182-84° decompg

Monoguanidinium, $C_3H_8N_{10}O_4$, crysts (from w), mp 176-79° & 186-87° dec

Monopotassium, $C_2H_2N_7O_4K$, crysts (from w), mp 199-200° dec

Prepn of the above salts is given by Henry et al (Ref 3)

Stollé & Dietrich (Ref 2) report a *Dinitrate* compd, pale-yel crysts, mp exploding when heated rapidly at 145°; was prepd by treating a soln of guanazole in 2NHCl with 65% HNO_3

Refs: 1) Beil-not found 2) R. Stollé & W. Dietrich, JPraktChem **139**, 193-210(1934) & CA **28**, 2714(1934) 3) R.A. Henry et al, JACS **75**, 961-62(1953) & CA **48**, 2050(1954)

Diammine-cadmium-nitrate. See under Ammines in Vol 1, p A277

Diammine-copper-nitrate. See Vol 1, p A280

Diammine-manganese-fulminate. See Vol 1, p A281

Diammine-zinc-fulminate. See Vol, p A281

Diammine-zinc-nitrate. See Vol 1, p A281

Diamond Ordnance Fuze Laboratories (DOFL). A US Ordnance Corps installation located in Washington, DC. These laboratories are engaged in research & development, procurement and associated activities for proximity, electronic & electric fuzes and related items. This facility is now called Harry Diamond Laboratories

Ref: OrdTechTerm(1962), p 99

Diamylamine Perchlorate or Dipentylamine Perchlorate, $[CH_3.(CH_2)_4]_2NH.HClO_4$, crysts, mp explodes at 323°; was prepd by double decompn of diamylamine HCl & Ag perchlorate (Ref 2)

Props & methods of prepn of diamylamine and of other salts are given in Refs 1, 3 & 4

Refs: 1) Beil **4**, (378), [642] & {330} 2) R.L. Datta & N.R. Chatterjee, JCS **115**. 1009(1919) 3) C.K. Hunt, IEC **35**, 1050-51 (1943) 4) Kirk & Othmer 1(1947), 849-51

Diamylether. See Amylether in Vol 1, p A396-L

α-Diamylose, $(C_6H_{10}O_5)_2$; mw 324.28; O 49.34%; was prepd by Leibowitz & Silmann (Ref 5); $Q_{combstn}$ 4285cal/g (Ref 4); X-ray spectrum (Ref 6); L & S also prepd some nitrated derivs which are expl:

α-Diamylose tetranitrate, $[C_6H_8O_3(ONO_2)_2]_2$; amor solid, not isolated from reaction mixt; was present in alc extract of crude hexanitrate

α-Diamylose hexanitrate, $[C_6H_7O_2(ONO_2)_3]_2$; crysts (from acet), mp deflgr at 206-07°; was prepd by nitration of α-diamylose or as the final product of the nitration of tetramylose

Compare with Amylose in Vol 1, p A398-R

Refs: 1) Beil-not found 2) P. Karrer & C. Nageli, Helv **4**, 169-73(1921) & CA **15**, 2072(1921)(The constitution of diamylose) 3) P. Karrer & P. Smirnoff, Helv **5**, 187-201 (1922) & CA **16**, 1747(1922)(The constitution of diamylose) 4) P. Karrer & W. Floroni, Ber **55B**, 2854(1922) & CA **17**, 987(1923) 5) J. Leibowitz & S.H. Silmann, Ber **58B**, 1889(1925) & CA **20**, 380(1926) 6) J.R. Katz & J.C. Derksen, ZPhysChem **158A**, 337(1932) & CA **26**, 3425(1932) 7) Davis(1943), 244

Diamylphthalate, $C_{18}H_{26}O_4$, mw 306.39, O 20.89%. The following isomers are of importance in proplnts:

Di-n-amylphthalate, Amylphthalate, Di-n-pentylphthalate or Diamylester of Phthalic Acid (called Phthalsäure-diamylester in Ger), $C_6H_4(OOC.CH_2.CH_2.CH_2.CH_2.CH_3)_2$; col, nearly odorless oily liq, fr p < -55°, bp 243-55° at 50mm & 342° at 760mm, flash p(CC) 245° (Ref 14) & 357°F (Ref 11), d 1.022, n_D 1.488 at 25°; obtd by esterification of phthalic anhydride with amylalcohol in the presence of 1% concd H_2SO_4 as a catalyst (Ref 14) and by other methods (Ref 10). Some other props are given in Refs 6 & 7

Di-n-amylphthalate is used as a plasticizer for NC lacquers (Ref 2), for cellulose acetate butyrate & ethylcellulose plastics (Ref 6a), and for organic coatings (Ref 5). McBride (Ref 3) patented a process for coating proplnt grains contg NC with an aq emulsion of diamylphthalate as a deterrent. Tibbits & Seavey (Ref 4) precipitated diamylphthalate from methaloic soln and coated proplnt grains contg NC & NG by agitating them in an excess of water. Di-n-amylphthalate (Amilol) was used in Germany & Sweden as a component of proplnts, such as Bofors NK (Nobel Krut). Admiral Alvaro-Alberto (Ref 13) gives the compn of NK-7 as follows: NC(sol) 70-64, NG 24-29, DPhA 1 & Amilol + vaseline 5-7%. Some investigators claim that Amilol functions as an anti-erosive agent in proplnts

A new method for detg phthalate esters in proplnts is reported by Grodzinski (Ref 9)

Di-iso-amylphthalate (called Diisoamylphthalat or Phthalsäure-diisoamylester in Ger), $C_6H_4[OOC.CH_2.CH_2.CH(CH_3)_2]_2$, col liq, bp 330-38° (dec) at 744mm, d 1.0220 at 15.6°/15.6°, n_D 1.4871 at 20°; smells sl of isoamylalcohol; can be prepd by ester interchange or "transesterification" of diethyl phthalate & $C_5H_{11}ONa$ (Refs 1 & 12)

This compd is used as a softening agent for NC & acetylcellulose lacquers, and to increase flexibility of nonboilable catgut sutures (Ref 8)

Refs: 1) Beil **9**, [587] 2) P.S. Symons, BritPlastics **2**, No 22: Synthetic and Applied Finishes 1, 169-71(1931) & CA **25**, 3182 (1931) 3) G.A. McBride, USP 1955927 (1934) & CA **28**, 4234(1934) 4) G.C. Tibbits & F.R. Seavey, USP 1967913(1934) & CA **28**, 5986(1934) 5) Fed Spec TT-D-291, Diamylphthalate (for use in organic coatings), Dec 1947 & CA **44**, 1265(1950) 6) E.S. Perry et al, JACS **71**, 3727, 3730(1949) &

CA **44**, 2306(1950)(Vapor press-temp relationship and latent heat of vaporization) 6a) Kirk & Othmer **3**(1949), 407-08 7) J. Haslam & W. Soppet, JApplChem (London) **1**, 112-19(1951) & CA **45**, 6869 (1951) 8) C.A. Powers & G.B. Ayres, USP 2694487(1954) & CA **49**, 3481(1955) 9) J. Grodzinski, AnalChem **27**, 1765-67 (1955) & CA **50**, 2171(1956) 10) K. Büchner, USP 2780643(1957) & CA **51**, 132924(1957) 11) Sax(1957), 544 12) A.N. Bourns et al, PrChemSoc **1957**, 120-21 & CA **51**, 15456(1957) 13) Admiral Alvaro-Alberto, Rio de Janeiro; private communication(14 Oct 1958) 14) CondChemDict(1961), 352

NOTE: No azido- or nitro derivs of Diamylphthalate were found in Beil

Dianilino-anthraquinone. See Bis(anilino)-anthraquinone in Vol 2, p B130-R

Dianilino-azobenzene. See Bis(anilino)-azobenzene in Vol 2, p B131-L

α,β-Dianilino-ethane or N,N'-Diphenylethylenediamine. See 1,2-Bis(N-anilino)-ethane in Vol 2, p B131-L

N,N'-Di(4-anilinophenyl)-urea. See N,N'-Bis(4-anilinophenyl)-urea in Vol 2, p B132-L

α-γ-Dianilino-propane or N,N'-Diphenyl-trimethylenediamine. See 1,3-Bis(anilino)-propane in Vol 2, p B132-R

DIANISOLE AND DERIVATIVES

Dianisole or Dimethoxybiphenyl (called Diemethoxy-diphenyl or Dianisol in Ger), $CH_3.O.C_6H_4.C_6H_4.O.CH_3$; mw 214.35, O 14.94%. Exists as

o,o-Dianisole, prisms (from alc), mp 154-55°, bp 299.5 to 301°; sol in hot alc & benz; sl sol in eth & petr eth (Ref 1)

m,m-Dianisole, ndls (from dil alc), mp 35.5° to 36°, bp 328° or 211-20° at 15mm press; readily sol in alc, eth, benz, chlf, glac acet ac & thiocarbonic acid, $CS(OH)_2$; insol in w (Ref 2)

p,p-Dianisol, crysts (from alc) or plts (from benz), mp 172-73°; readily sol in benz, chlf, hot alc; sl sol in eth; insol in petr eth & w (Ref 3)

Other props & methods of prepn are given in Refs

Refs: 1) Beil **6**, 989, (484) & [960] 2) Beil **6**, 991 & [961] 3) Beil **6**, 991, (486) & [962]

Dinitrodianisole, $[-C_6H_3(NO_2).O.CH_3]_2$; mw 304.25, N 9.21%. Several isomers are known:

3,5'-Dinitro-o,o-dianisole or 3,5'-Dinitro-2,2'-dimethoxybiphenyl, crysts, mp 133-35° (Ref 3)

4,4'-Dinitro-o,o-dianisole or 4,4'-Dinitro-2,2'-dimethoxybiphenyl, crysts, mp 248-49° (Ref 9)

5,5'-Dinitro-o,o-dianisole or 5,5'-Dinitro-2,2'-dimethoxybiphenyl, yel ndls or ndls (from acet + petr eth), mp 263-64°; readily sol in hot acet, chlf & alc; sol in benz; sl sol in glac acet ac & hot alc; insol in petr eth & water (Ref 1)

6,6'-Dinitro-o,o-dianisol or 6,6'-Dinitro-2,2'-dimethoxybiphenyl, yel crysts, mp 226-28° (Ref 6) & 231-32° (Ref 8)

2,2'-Dinitro-p,p-dianisole or 2,2'-Dinitro-4,4'-dimethoxybiphenyl, crysts, mp 136-37° (Refs 4 & 7)

3,3'-Dinitro-p,p-dianisole or 3,3'-Dinitro-4,4'-dimethoxybiphenyl, crysts, mp 221° (Ref 2) & 214° (Ref 5)

2,6-Dinitro-3,5-dimethoxybiphenyl, $C_6H_5.C_6H(NO_2)_2(OCH_3)_2$; pale-yel prisms (from ethyl acetate), mp 260-61° (Ref 10)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **6**, 990 & (485) 2) J. van Alphen, Rec **49**, 769(1930); Rec **50**, 657 (1931) & CA **25**, 4873(1931) 3) F. Calvet & E. Seijo, AnalesSocEspañFisQuim **31**, 882 (1933) & CA **28**, 1340(1934) 4) K. Hata et al, BullChemSocJapan **10**, 425(1935) &

CA **30**, 1056(1936) 5) H.H.Hodgson & P.F. Holt, JChemSoc **1937**, 37-38 & CA **31**, 2195(1937) 6) R. Adams & G.C. Finger, JACS **61**, 2828(1939) & CA **34**, 406(1940) 7) W.C. Lothrop, JACS **64**, 1700(1942) & CA **36**, 5805(1942) 8) N. Kornblum & D.L. Kendall, JACS **74**, 5782(1952) & CA **49**, 967(1955) 9) L.M. Litvinenko et al, ZhObshchKhim **27**, 315(1957) & CA **52**, 9043(1958) 10) J. Forrest, JChemSoc **1960**, 578 & CA **54**, 12056(1960)

Tetranitrodianisole, $[-C_6H_2(NO_2)_2.O.CH_3]_2$; mw 394.25, N 14.21%. The following isomers are reported in the literature:

3,5,3',5'-Tetranitro-2,2'-dimethoxybiphenyl, exists in two forms: lower-melting crysts (from acet or alc), mp 179° & higher-melting crysts, mp 191°, which are mutually interconvertible; was prepd by reaction of 2,2'-dimethoxybiphenyl & ethyl nitrate in concd H_2SO_4 below 0°; or by reaction of 3,5,3',5'-tetranitro-2,2'-dihydroxybiphenyl & diazomethane in eth & acet (Ref 1, 4, 5, 6, 8, 10 & 11). Other methods of prepn & props are given in the above Refs

4,4',6,6'-Tetranitro-2,2'-dimethoxybiphenyl, exists in two forms: lower-melting crysts, mp 168° convertible to higher-melting crysts (from alc), mp 189°; was prepd by nitrating guaiacol [o-methoxyphenol, $C_6H_4(OH)OCH_3$] to the dinitro deriv, which was converted to the 2,3,5-$Cl(O_2N)_2C_6H_2OCH_3$, and by boiling in Nitrobenz with Cu bronze for 1hr (Ref 9, p 453)

4,4'6,6'-Tetranitro-3,3'-dimethoxybiphenyl, crysts, mp 244°; was prepd from 3,3'-dichlorobiphenyl which was first nitrated to 4,4',6,6'-tetranitro-3,3'-dichlorobiphenyl, and by action of Na metholate in boiling MeOH on the tetrachloro deriv (Ref 9, p 361)

3,5,3',5'-Tetranitro-4,4'-dimethoxybiphenyl, almost col ndls (from concd HNO_3), mp 242-55°; was prepd by nitrating 4,4'-dimethoxybiphenyl with HNO_3 (Refs 2 & 7)

X,X,X,X-Tetranitro-4,4'-dimethoxybiphenyl, straw-yel ndls, mp 244.6°; was obtd by nitrating 4,4'-dimethoxybiphenyl with fuming HNO_3 (Ref 3)

No expl props of the tetranitro derivs were reported

Refs: 1) Beil **6**, (485) & [960] 2) Beil **6**, [963] 3) Beil **6**, 993 4) H. Raudnitz, Ber **60B**, 742(1927) & CA **21**, 1982(1927) 5, W. Borsche & E. Feske, Ber **60B**, 1220 (1927) & CA **21**, 2681(1927) 6) H. Raudnitz, Ber **61B**, 246(1928) & CA **22**, 1349 (1928) 7) J. van Alphen, Rec **49**, 774(1930) & CA **24**, 4777(1930) 8) J. van Alphen, Ber **64B**, 1819(1931) & CA **25**, 5157(1931) 9) J. van Alphen, Rec **51**, 361, 453(1932) & CA **26**, 2447, 3789(1932) 10) F.H. Case & R.U.Schock, Jr, JACS **65**, 2087(1943) & CA **38**, 89(1944) 11) D.B. Murphy et al, JACS **75**, 4289(1953) & CA **48**, 12075(1954)

NOTE: Hexanitro and higher nitrated derivs of Dianisole were not found in Beil or in CA thru 1961

Dianisoyl Peroxide or Anisoyl Peroxide, (called Dianisoylperoxyd in Ger), $CH_3O.C_6H_4.CO.OO.OC.C_6H_4.OCH_3$;

mw 182.17, O 52.70%; the p-deriv is col,odorless & tasteless, crysts (from eth acet), mp 128°; deflgr on heating on a Pt foil or in a capillary tube; insensitive to impact or pressure; deflgr in contact with concd H_2SO_4; sol in eth acet; diffc sol in petr eth; can be prepd by adding dropwise a soln of 3% H_2O_2 in pyridine to a soln of anisoyl chloride in acetone, and allowing the mixt to stand for 12hrs (Refs 1 & 2). Hey & Walker (Ref 3) reported a prepn by reaction of aryl nuclei with acyl peroxides. See also Refs 4, 5, 7, 9, 10 & 13 for prepn & decompn in soln. Its IR absorption spectrum is given by Davidson (Ref 6)

Dianisoyl peroxide is used as a polymerization catalyst (Refs 8, 11 & 12)

Refs: 1) Beil **10**, 963 2) L. Vanino & E. Uhlfelder, Ber **37**, 3624(1904) 3) D.H. Hey & E.W. Walker, JCS **1948**, 2213-20 & CA **43**, 3394(1949) 4) C.G. Swain et al, JACS **72**, 5426-34(1950) & CA **45**, 6603, (1951)(Spontaneous thermal decompn & effect of structure) 5) A.J. Bloomquist & A.J. Buselli, JACS **73**, 3883-88(1951) & CA **46**, 2522-23(1952)(Prepn & decompn in acetophenone) 6) W.H.T. Davidson, JCS **1951**, 2456-61 & CA **46**, 8965(1952)(IR absorption spectrum) 7) W. Cooper, JCS **1952**, 2408-15 & CA **47**, 3087(1953)(Radical-induced decompn) 8) M Takebayashi et al, BullChemSoc(Japan) **26**, 137-40, 475-79(1953) & CA **49**, 895, 6826(1955)(Use as catalyst in styrene polymerization) 9) R.I. Milyutinskaya & K.S. Bagdasar'yan, ZhFizKhim **28**, 797-800(1954) & CA **50**, 242 (1956)(Decompn in benz &nitrobenzene) 10) M. Imoto & S. Choe, JPolymerSci **15**, 475-91(1955) & CA **49**, 10663-64(1955) (Decompn in presence of dimethylaniline) 11) M. Imoto & K. Takemoto, JPolymerSci **18**, 377(1955) & CA **50**, 16174(1956)(Polymerization of acrylonitrile) 12) N.G. Saha et al, JCS **1956**, 427-36 & CA **50**, 6833(1956) (Prepn & study as initiator of polymerization of methylmethacrylate) 13) J.C. Bevington & T.D. Lewis, TrFaradSoc **54**, 1340-44 (1958) & CA **53**, 12016(1959)(Study of photodissociation)

Diaphragm Gauges. See under Gages

Diaryl Phthalates in Propellants. Substances such as diphenylphthalate (qv) were proposed for coating the grains of NC or NG proplnts to serve as deterrents, solvents, plasticizers, and/or stabilizers
Ref: C.E. Silk, USP 2335779(1943) & CA **38**, 3130(1944)

Diaspon Explosive. It was patented in England & France in 1881 by J. Anders and contained NG 47-63, Na nitrate 22-23, wood cellulose 8-18, sulfur 3.9-9 & NC 0.5-3%
Ref: Daniel(1902), 204

Diaspon Gelatin. It was patented in England & France in 1881 by J. Anders and contained NG 92-95, wood cellulose (nitrated to 11-12%N) 4.5-7 & alcohol 0.5-2%. These ingredients were heated together with 10-15% of solvent (3/1-ether/alcohol) to 40-45° and then the solvent evapd
Ref: Daniel(1902), 204

Diatomaceous Earth or Diatomite. See "Celite" in Vol 2, p C93-L and under Kieselguhr

Diaza. A prefix denoting replacement of two ring carbons by nitrogens, and applied to names of heterocyclic compds derived from names of carbocyclic compds

See also Aza, Vol 1, p A516-R

Diaza Alkanes, Polynitro. They are prepd by acidifying the appropriate amine with HNO_3 to form compds useful as expls. Thus the compd bis(1,1,1-trinitroethyl-2-N-nitroethylene-imine)-dinitromethane, $[(CH_2)_2\overset{+}{N}H-CH_2.C(NO_2)_3]C(NO_2)_2$, was added to a 5% soln of 100% HNO_3 & AcOH. The soln was poured on ice, and the wh ppt was washed with w, and recrystd from hot 100% HNO_3 The product, $[(CH_2)_2\overset{+}{N}(NO_2)CH_2.C(NO_2)_3]_2C(NO_2)_2$, mp 170-75°, $Q_{combstn}$ 1978cal/g, had an impact value of 10-15cm for a 2kg wt; Pb block value 177 (**TNT** = 100); and BM value 155 (TNT = 100). The compd bis(2-dinitropropylyl-3-N-nitroethylene-imine)-dinitromethane, $[(CH_2)_2\overset{+}{N}(NO_2).CH_2.C(NO_2)_2.CH_3]_2C(NO_2)_2$, was similarly prepd from the appropriate amine
Ref: M.B. Frankel, USP 2978510(1961) & CA **55**, 15934(1961)

1,4-Diazacyclohexane. See Piperazine

Diazacyclohexanone. See Tetrahydropyrimidone

1,3-Diazacyclohexene. See Tetrahydropyrimidine

1,3-Diazacyclopentane. Same as Cyclo-2,4,5-trimethylene-1,3-diamine (qv) or Imidazolidine, Vol **3**, p C610-R

Diazacyclopentanone-1. See Imidazolidone or Ethyleneurea

1,3-Diazacyclopentene. See Imidazoline

4,7-Diazadecane; 2,2,4,7,9,9-Hexanitro-, $[H_3C.C(NO_2)_2.CH_2.N(NO_2).CH_2\text{-}]_2$; mw 428.26, N 29.44%. An expl compd first prepd by Evans Res Associates and described in their classified rept ERD-LA-71, "Three Year Summary Progress Rept" (June 1954). The Los Alamos Scientific Laboratory has since prepd the compd by a more direct method and detd some of its props [See M.B. Frankel & K. Klager, USP 309873(1963) & CA **60**, 1588(1964)]

3,5-Diazaheptane; 1,1,1,3,5,7,7,7-Octanitro; $CH_2[.N(NO_2).CH_2.C(NO_2)_3]_2$; mw 546.23, N 41.03%; crysts (from cyclohexane), mp 84-85°, $Q_{combstn}$ 1423cal/g, Trauzl Pb block value 196% (TNT = 100), Ballistic Mortar value 156% (TNT = 100); was prepd by condensing 2-trinitro-1-ethanol, $C(NO_2)_3.CH_2OH$, with $CH_2(NH_2)_2$ to form the hexanitro compd, $CH_2[NH.CH_2C(NO_2)_3]$, which was acidified with HNO_3 to yield the useful expl octanitro compd
Ref: M.B. Frankel & K. Klager, USP 2978506(1961) & CA **55**, 15933(1961)

2,5-Diazahexane. Same as N,N'-Dimethylethylenediamine

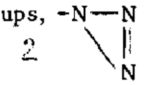
1,3-Diazapentane. See Cyclo-2,4,5-trimethylene-1,3-diamine in Vol **3**, p C610-R

1,5-Diazapentane. Same as Diaminopropane

Diazide of Diglycolamidic Acid. See Diglycolamidic Diazide under Diglycolamidic Acid

Diazide of Oxalic Acid. See Oxalyl Diazide under Oxalic Acid

Diazide of Phthalic Acid. See Phthalyl Diazide under Phthalic Acid

Diazido. A prefix indicating the presence of two azido groups,  also designated as $(\text{-}N_3)$ in org compds. See also Azides, Organic in Vol 1, p A626-L

α-δ-**Diazidoadipic Acid or Ditriazoadipic Acid** (called by Bertho & Maier *α-α'*-Diazidoadipinsäure), $HO.OC.CH(N_3).CH_2.CH_2.CH(N_3).CO.OH$; mw 228.17, N 36.84%; col crysts (from w) mp 147° (decompn); was prepd from ethyl-*α,δ*-dibromoadipate and Na azide in ethanol with subsequent alk hydrolysis. Its expl props were not examined.
(Compare with Adipyldiazide, described under Adipic Acid, p A104-R in Vol 1 of this Encycl
Refs: 1) Beil-not found 2) A. Bertho & J. Maier, Ann **498**, 56(1932) & CA **26**, 5910(1932)

3,3'-Diazidoazoxybenzene. See in Vol 1, p A666-R

Diazido-benzene. See in Vol 2, p B42-R

Diazidobenzoic Acid. See in Vol 2, p B69-R

Di(*α*-azidobenzylidene)-azine. (called in Ger *Dibenzhydrazidazid).* See Bis(*α*-azidobenzylidene)-azine in Vol 2, p B133-L

Di(*α*-azidobenzylidene)-hydrazine. (called in Ger *Dibenzyhydrazidhydrazid).* See Bis (*α*-azidobenzylidene) hydrazine in Vol 2, p B133-R

Diazidobiphenyl. See in Vol 2, p B123-L

Diazidobiphenylcarboxylic Acid. See in Vol 2, p B125-R

Diazido Compounds. They are described, with a few exceptions, under corresponding parent compounds, such as: Acetic Acid (Vol 1, p A27-L); Acetic Anhydride (Vol 1, p A31-L); Aminomesitylene (Vol 1, p A225-R); Anthraquinone (Vol 1, p A459); Benzene (Vol 2, pp B42-3); Benzoic Acid (Vol 2, p B69-R); Biphenyl (Vol 2, p B123-L); etc

2,5-Diazido-3,6-dichloro-1,4-benzoquinone. See in Vol 2, p B80-L

1,3-Diazido-4,6-dinitro-benzene. See in Vol 2, p B43-L

Diazidoethylacetate. See under Ethyl Acetate

Diazido-oxalylhydrazine. See under Oxalylhydrazine

Diazidophenol. See under Phenol

Diazidopropane. See under Propane

Diazidopropene. See under Propene

Diazidopropylene. See under Propylene

Diazidotetrammine Complexes. See under Ammines in Vol 1, ppA278-A279

Di(azidothioformyl)-disulfide. See Bis (azidothioformyl)-disulfide in Vol 2, p B133-R

2,6-Diazido-4-trimethylammonium-1,4-benzoquinone. See in Vol 2, p B84-R

1,3-Diazido-2,4,6-trinitro-benzene. See in Vol 2, p B43-L

Diazo. A prefix indicating the presence of the bivalent -N:N- radical in an org compd. Its isomer, >N:N is called *diazonium.* See also Azo-compounds in Vol 1, p A644-R

Diazoacetamide. (called Diazoessigsaure-amid or Diazoacetamid in Ger), N:N:CH.CO.NH_2; mw 85.07, N 49.40%; golden-yel prisms (from alc), mp 114°(dec); sol in w, alc & concd aq NH_3; prepn & other props are given in Beil
Ref: Beil **3**, (215-16)

Diazoacetic Acid. (called Diazoessigsaure in Ger), $N_2CH.COOH$; mw 86.05, N 32.56%; The free acid is not stable, but it forms some metallic salts which are expl: *Potassium salt,* $KC_2HN_2O_2$, yel ndls or lemon-yel lftlts, explodes violently with a loud noise on heating (Refs 1 & 2) and *Sodium salt,* $NaC_2HN_2O_2$, yel crysts (from w), grn-yel ndls (from w + alc-eth), detonates on heating (Ref 1)
Refs: 1) Beil **3**, (209) & [1138]; **25**, 109
2) E. Müller, Ber **41**, 3136-37(1908) & CA **3**, 68(1908)

Diazoacetic Acid, Ethyl Ester; Ethyldiazoacetate or Diazoacetic Ester (called Diazoessigsäure-äthylester or Diazoessigester in Ger), $N_2CH.CO_2.C_2H_5$; mw 114.10, N 24.55%; lemon-yel, unpleasant smelling, expl oil, fr p - 22°, bp 140° at 720mm, 69° at 29mm & 29-31° at 5mm Hg (distillation even under reduced pressure is dangerous, Ref 2); d 1.088 at 21°, n_D 1.4616; v sol in alc, benz, ligroin & eth; sl sol in w; was prepd by action of aq $NaNO_2$, in dil H_2SO_4, on glycine ethyl ester hydrochloride, $H_2N.CH_2.COO.C_2H_5.HCl$ (Ref 1)

Its *Mercury salt,* $Hg(C_4H_5N_2O_2)_2$, yel rhmb crysts (from eth), mp 104°(dec); sl sol in alc & eth; prepd by treating diazoacetic ester with yel HgO, while cooling with ice; deflagrates or explodes mildly on strong impact (Ref 1, p 215)

Refs: 1) Beil **3**, (211, 215), [390] & {1140}; **25**, 110 2) E.B. Womack & A.B. Nelson in OrgSynth, Coll Vol **3**(1955), 392-93 3) Sax(1957), 545

Diazoacetic Acid, Methyl Ester or Methyldiazoacetate (called Diazoessigsäure-methylester in Ger), $N_2CH.CO_2.CH_3$; mw 100.08, N 27.99%; lt-yel expl liq, bp 31° at 12mm Hg; distillation, even under reduced pressure, is extremely dangerous, since heat causes the compd to detonate violently; d 1.158 at 25°, n_{α} 1.4515 & n_{β} 1.4676 at 20.6°; was prepd by diazotization of aminoacetic acid methyl ester with HNO_2 (Refs 1, 2 & 3). Its *Mercury salt*, $Hg(C_3H_3N_2O_2)_2$, sulfur-yel crysts (from eth), mp 123° (dec), sol in hot eth; was obtd by reaction of the methyl ester with yel HgO, under cooling. This salt is probably expl (Compare with Hg salt of ethyl ester)
Refs: 1) Beil **3**, (209), [389] & {1138}; **25**, 109 2) N.E. Searle, USP 2490714(1949) & CA **44**, 3519(1950) 3) N.E. Searle in OrgSynth **36**(1956), 25-28

Diazoacetone or Diazo-2-propanone (called Diazoaceton, 1-Diazo-propanon(2), or Acetyldiazomethan in Ger), $N_2CH.CO.CH_3$; mw 84.08, N 33.32%; lt-yel oil, fr p does not solidy at -18°, forms lt-yel crysts with liq CO_2, bp 46-47° at 15mm of Hg; explodes on heating, d 1.0661 at 21.7°; miscible in equal proportions of a mixt of alc, eth & w; was prepd from acetyl chloride & diazomethane in ether or from diacetyldiamethane & dil NaOH at 0° (Ref)
Ref: Beil **1**, (396), [823] & {3093}

Diazoacetonitrile (called Diazoacetonitril or Diazoessigsäurenitril in Ger), $N_2CH.CN$; mw 67.05, N 62.67%; orn-yel, sl elec conductive liq, fr p does not solidify at - 18°, bp 46.5° at 14-15mm Hg; sol in w; darkens on exposure to light; burns with a luminous flame; explodes on contact with CuO and forms an expl Hg compd; was obtd in low yield by reaction of HNO_2 on aminoacetonitrile hydrochloride (Ref 1)

Phillips & Champion (Ref 2) reported an expln occurred during the prepn of diazoacetonitrile and recommended that the compd be used only in dil soln, because of its highly expl nature when concd
Refs: 1) Beil **3**, (216) 2) D.D. Phillips & W.C. Champion, JACS **78**, 5452(1956) & CA **51**, 718(1957)

DIAZOAMINOBENZENE AND DERIVATIVES

Diazoaminobenzene, Diazobenzeneanilide, or Benzeneazoanilide [called Diazoamidobenzene by Sax (Ref 5); called Diazoaminobenzol or 1,3-Diphenyl-triazen-(1) in Ger], $C_6H_5.N:N.NH.C_6H_5$; mw 197.23, N 21.31%; yel crysts (from alc) or pltlts (from benz), mp 98-100°, bp explodes when heated to 150° (Ref 6); sol in eth & alc; mod sol in hot alc; v sl sol in cold alc; insol in w; has been prepd by the action of $NaNO_2$ on aniline sulfate or aniline hydrochloride, by the action of $NaNO_2$ & Na acetate on aniline hydrochloride in the presence of iron, by action of amyl nitrite on aniline, and by other methods (Refs 1 & 3)

According to work done in Japan (Ref 4), this compd is reported to be the most effective cellulating agent and has found use in the prepn of cellulose rubber
Refs: 1) Beil **16**, 687, (404) & [351] 2) Thorpe **3**(1939), 595-96 3) W.W. Hartman & J.B. Dickey, OrgSyntheses **14**(1943), 24-26 & Coll Vol **2**(1943), 163 4) J. Tanaka, ReptsOsakaPrefect.IndResInst **2**, 27-33(1950); **3**, No 3, 5-12(1951) & CA **46**, 11743(1954) 5) Sax(1957), 545 6) CondChemDict(1961), 354

Mononitrodiazoaminobenzene, $O_2N.C_6H_4.N:N.NH.C_6H_5$; mw 242.23, N 23.13%. The *2-Nitro*, orn-yel ndls (from alc), mp 104.5-105°; *3-Nitro*, yel prisms (from eth), mp 129-31°; and *4-Nitro* derivs, yel ndls (from benz), mp ca 148° (with decompn) are described in Beil
Ref: Beil **16**, 696, 697 699, (406) & [354]

Dinitrodiazoaminobenzene, $O_2N.C_6H_4.N:N.NH.C_6H_4.NO_2$; mw 287.23,

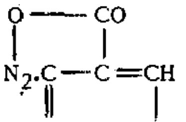
N 24.38%. The following derivs are the more important dinitro compds:
2,2'-Dinitro-diazoaminobenzene, yel ndls or scales (from alc), mp 196-96.5°; diffic sol in boiling alc (Refs 1, 3 & 4)
3,3'-Dinitro-diazoaminobenzene, straw-yel ndls (from alc), mp 195-96° (violent decompn), sl sol in acetic acid; v sl sol in alc & eth; insol in benz, eth acet, chlf & hot aq alkali (Refs 1, 3 & 4)
4,4'-Dinitro-diazoaminobenzene, yel ndls (from alc), orn-yel lfts (from benz), mp 220-36° (with decompn); sl sol in chlf & benz; diffc sol in boiling alc & acet; forms salts (such as of Na, Cu, Ag, Cd & Co), which are expl (Refs 2, 3 & 4)

Other props and methods of prepn of the dinitro derivs are given in the Refs
Refs: 1) Beil **16**, 697 & [354] 2) Beil **16**, 700, (406) & [354] 3) B. Houston & T.B. Johnson, JACS **47**, 3011-18(1925) & CA **20**, 372(1926) 4) F. Dwyer & J.C. Earl, Chem & Ind **1940**, 136 & CA **34**, 3245(1940)

NOTE: Higher nitrated derivs of Diazoaminobenzene were not found in Beil or in CA thru 1961

Diazoaminobenzene, Amine of. See Aminodiazoaminobenzene in Vol 1, p A195-L

6-Diazo-3-aminobenzoic Acid (called 6-Diazo-3-aminobenzoesäure in Ger),



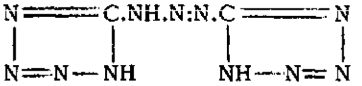
mw 163.13, N 25.76%; brass-yel ndls or lfts + $3.H_2O$; loses w of crystn by heating on a water bath, mp explodes on heating; mod sol in hot w; sl sol in hot alc; insol in eth; was prepd by treating 2,5-diaminobenzoic acid with nitrous acid. This compd does not exhibit the characteristics of an acid. It posseses basic properties and is a true diazo compd. Other props are given in the Refs
Refs: 1) Beil **16**, 611-12 2) P. Griess, Ber **17**(1884), 603-08

4-Diazoaminobiphenyl; 1,3-Di(p-biphenyl)-triazene or 4,4'-Diphenyl-diazoaminobenzene. See I, III-Bis(p-biphenyl)-triazene in Vol 2, p B136-L

Diazoamino Compounds. They are described, with few exceptions under individual diazoamino compds. However, several general Refs on this subject are given below
Refs: 1) A. Mangini & I. Dejudicibus, GazzChimItal **63**, 601-12(1933) & CA **28**, 1672-74 (1934) 2) A. Mangini, GazzChimItal **65**, 298-308(1935) & CA **29**, 6580-83(1935) 3) Thorpe **3**(1939), 595-97 4) P. Petitcolas et al, USP 2675374(1954) & CA **49**, 1337-38 (1955)

Diazoaminocresol and its nitrated derivs are mentioned under Dinitroaminocresols, Vol 1, p A194-L, and are reported R. Nietzki & F. Ruppert, Ber **23**, 3479-80(1890) & JCS **60** I(1891), 309

5,5'-Diazoaminobistetrazole or 5,5'-Diazoamino-di-1-H-Tetrazole [called 5.5'-Diazoaminotetrazol or 1,3-Di-[tetrazolyl-(5)-triazen in Ger],



; mw 181.13, N 85.07%; almost col, lustrous, thin doubly refractive plates + 1 mole H_2O; was obtd by treating aminoguanidine nitrate with $NaNO_2$ in the presence of dil acetic acid, or by diazotizing aminotetrazole (Refs 1 & 2)

Its *Copper salt*, $Cu_3(C_2N_{11})_2 + 2NH_3$, dk-grn plts, explodes by friction, impact or heating; this salt with $KClO_3$ is a more powerful primer mixt than MF-$KClO_3$ (Ref 2); *Silver salt*, $Ag_2C_2HN_{11} + H_2O$, powd, explodes on heating or strong friction; and *Barium salt*, $Ba_3(C_2N_{11})_2 + 8H_2O$, yel pltlts (from w), explodes on mild heating
Refs: 1) Beil **26**, (191) 2) K.A. Hofmann & H. Hock, Ber **43**, 1866-71(1910) & CA **4**, 2807(1910) 3) J. Reilly et al, Nature **159**, 643-44(1947) & CA **41**, 5510(1947) 4) J. Reilly et al, SciProcRoyDublinSoc **24**, 349-53(1948) & CA **43**, 1769(1949)

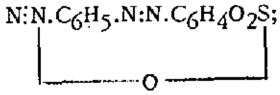
Diazoaminomethane. Same as Dimethyltriazene

Diazoaminonaphthalene [called 1.1'-(or α,α'-) Diazoaminonaphthalin or 1.3-Di-α-naphthyl-triazen-(1) in Ger], $C_{10}H_7.N:N.NH.C_{10}H_7$; mw 297.34, N 14.13%; yel-brn plts (from alc), mp - melts to a resin at water-bath temp, explodes on heating to a higher temp (ca 100°); was obtd by action of a dil alc soln of $NaNO_2$ on α-naphthyl-amine hydrochloride
Refs: 1) Beil **16**, 716 2) Sax(1957), 716

Diazoaminonaphthalene Bromide Hydrobromide, See Aminodiazonaphthalene Bromide Hydrobromide in Vol 1, p A195-L

Diazoanhydride. Same as 1,2,3-Oxdiazole

4'-Diazoazobenzene-4-sulfonic Acid [called 4'-Diazoazobenzol-sulfonsäure-(4) in Ger],



$N{:}N.C_6H_5.N{:}N.C_6H_4O_2S$; mw 288.24, N 19.44%;

pale-yel ndls, mp - explodes on heating; sol in concd KOH; insol in org solvs; was prepd by Griess (Ref 2) on passing nitrous gas thru a slurry of 4'-aminoazobenzene-4-sulfonic acid, and allowing the mixt to stand for a long time. Seidler (Ref 2) patented the use of this compd in some expl mixts
Refs: 1) Beil **16**, 617 2) Seidler, GerP 46205(1863) 3) P. Griess, Ber **15**, 2186(1882)

Diazobenzene and Diazonium Benzene Derivatives. See Vol 2, pp B54-R & B58-L.

Diazobenzene Hydrate. See Benzenediazonium Hydroxide in Vol 2, p B55-L

Diazobenzeneimide or Diazobenzeneimine. Same as Azidobenzene described in Vol 2, p B42-L

Diazobenzene Nitrate. See Benzenediazonium Nitrate in Vol 2, p B56-L

Diazobenzene Oxalate. See Benzenediazonium Oxalate Vol 2, p B56-R

Diazobenzene Perchlorate. See Benzenediazonium Perchlorate in Vol 2, p B56-R

Diazobenzenephenylhydrazide. Same as I, III-Diphenyl-tetrazene

Diazobenzene Picrate. See Benzenediazonium Picrate in Vol 2, p B57-L

Diazobenzene Sulfate. See Benzenediazonium Sulfate in Vol 2, p B57-L

Diazobenzene Sulfocyanate or Diazobenzene Thiocyanate. See Benzenediazonium Sulfocyanate in Vol 2, p B57-L

Diazobenzene Sulfonic Acid. See Benzenediazonium Sulfonic Acid in Vol 2, p B57-L

Diazobenzene Tetrachloroiodide. See Benzenediazonium Tetrachloroiodide in Vol 2, p B57-R

Diazobenzene Tribromide. See Benzenediazonium Tribromide in Vol 2, p B58-L

Diazobenzene Trinitromethane. See Benzenediazonium Salt of Trinitromethane in Vol 2, p B58-L

Diazobiphenyl Perchlorate. See Biphenyldiazonium Perchlorate in Vol 1, p A191-L, under Aminobiphenyls

Diazocamphor. See Vol 2, p C23-R

Diazocarbazole. See Vol 2, p C47-L

Diazochemistry. Title of a book by Heinrich Zollinger, "Azo and Diazo Chemistry-Aliphatic and Aromatic Compounds", Interscience, NY (1961) (Translated by Harry E. Nurston).

See also Diazocompounds

Diazochloronitrophenol. See Mononitrodiazochlorobenzoquinone under Chlorobenzoquinone and Derivative, Vol3, p C253-L

Diazocompounds. A group of very reactive org compds, many of which are explosive, containing the bivalent radical -N:N- or N⁚N=. They are formed when nitrous acid acts at low temp on the salts of primary amines. The process of prepg diazocompds is called *diazotization* and was discovered about 1860 by P. Griess (Ref 4)

Von Herz (Ref 1) prepd several diazo compds (such as *Diazodinitrophenol, Diazodinitrophloroglucinol and Diazodinitroresorcinol*) which he claimed to be suitable for use in detonators & percussion caps.

Most diazo- and diazonium compounds are described in this Encyclopedia, either as individual diazocompds or under the corresponding parent compd, such as **Benzene Diazo- and Diazonium Derivatives** (Vol **2**, pp B54-R to B58-L)

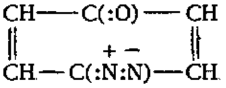
See also Diazochemistry, Diazohydrocarbons and Diazonium Salts

Refs: 1) E. von Herz, BritP 207563 (1922) & CA **18**, 1573-74 (1924) 2) N.V. Sidgwick et al, JCS **1933**, 406-12 & CA **27**, 3459 (1933) (Dipole moments and structures) 2a) Sidgwick, OrgChem of N (1937), 347-62 & 400-25 3) Thorpe **3** (1939), 580-600 4) Hackh's (1944), 266-67 5) P. Nawiasky et al, C & EN **23**, No 14, 1247 (1945) (Explosive reaction of diazonium compds with sulfides of sodium) 6) L. Hellerman & R.L. Garner, JACS **68**, 819-23 (1946) & CA **40**, 4049 (1946) (Aliphatic diazocompds) 7) K.H. Saunders, "The Aromatic Diazo Compounds and Their Technical Applications", Longmans, Green & Co, London (1948), 402 pp (Reviewed in Chemistry in Canada **1**, 14 (1949) 8) W.W. Becker, AnalChem **22**, 185-88 (1950) & CA **44**, 3407 (1950) (Detn of N-contg functional groups in org compds) 9) O. Süs et al, Ann **598**, 123-38 (1956) & CA **51**, 321-23 (1957) (Light-induced degradation products of diazo derivs) 10) I.A. D'yakonov, "Alifaticheskie Diazosoedineniya" (Aliphatic Diazo Compounds), Izdatel', Leningrad, Moscow (1958), 138 pp

Diazocyclobutane. See under Cyclodiazo Compounds in Vol 3, p C593-L

Diazocycloethane. See under Cyclodiazo Compounds in Vol 3, p C593-L

4-Diazo-2,5-cyclohexadien-1-one [called p-Benzquinonediazide by Anderson et al (Refs 2 & 3) and also Diazophenol (Ref 1)],



$$\begin{array}{lcl} \mathrm{CH} & \text{——}\ \mathrm{C(:O)}\ \text{——} & \mathrm{CH} \\ \| & \overset{+\ -}{} & \| \\ \mathrm{CH} & \text{—}\ \mathrm{C(:N:N)}\ \text{—} & \mathrm{CH} \end{array}$$

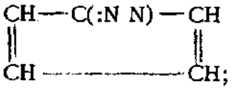
; mw 108.10, N 25.92%; colorless ndls + $4H_2O$, mp 38-39°, explodes violently at 75°; sl sol in w & alc; diffc sol in eth & benz; was obtd by digesting an abs alcoholic soln of p-hydroxybenzenediazonium chloride with Ag oxide and cooling the filtrate to -30°. This compd & its derivs are light-sensitive material. Its salt, $C_6H_4N_2O + CdI_2$, explodes at 168° (Ref 1)

The *Mononitro derivs* are described under Benzenediazoöxide, Vol **2**, p B58

Refs: 1) Beil **16**, 526, (363) & [288] 2) L.C. Anderson & M.J. Roedel, JACS **67**, 955-58 (1945) & CA **39**, 3270 (1945) 3) J.D.C. Anderson et al, JCS **1949**, 2082-88 & CA **44**, 6725 (1950) 4) O. Süs et al, Ann **598**, 123 (1956) & CA **51**, 321 (1957)

Diazocyclomethane. See under Cyclodiazo Compounds in Vol 3, p C593-L

Diazocyclopentadiene,

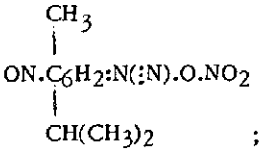


mw 92.10, N 30.42%; red liq, bp 52-53° at 50mm, d 1.059, n_D 1.6150; yel crysts (from alc, then pentane at -70°), mp -23 to 22°; was prepd by reaction of a suspension of cyclopentadienyllithium with a cooled, stirred soln of p-toluenesulfonyl azide in dry ether (Ref 2). Its structure was established by analysis, mw detn, and IR & UV absorption spectra

Ramirez & Levy (Ref 3) advise extreme caution during all operations with Diazocyclopentadiene, since during one prepn, they reported, a violent expln took place after distillation

Refs: 1) Beil-not found 2) W. von E. Doering & C.H. DePuy, JACS **75**, 5955-57(1953) & CA **49**, 884(1955) 3) F. Ramirez & S. Levy, JOC **23**, 2037(1958) & CA **53**, 10088(1959)

Diazocymol, $C_{10}H_{14}N_2O$. Its expl deriv is *5-Nitroso-p-cymol-2-diazoniumnitrate* or *6-Nitroso-p-cymol-3-diazoniumnitrate,*



$$\underset{CH(CH_3)_2}{\overset{CH_3}{ON.C_6H_2}}:N(:N).O.NO_2$$

; mw 252.23, N 22.22%; canary-yel lt flakes, mp 56-60° (dec) and explodes on heating at higher temp and on impact; diffc sol in org solvs; was prepd by action of nitrogen dioxide on p-dinitrosocymol, or on quinonedioxime

Refs: 1) Beil **16**, 509-10 2) R. Oliveri-Tortorici, Gazz **30** I, 534, 537(1900) & JCS **78** I, 553(1900)

Diazo-o-dihydroxybenzene. See Diazopyrocatechol

Diazo-m-dihydroxybenzene. See Diazoresorcinol

Diazodinitrophenol (DADNPh). See Dinitrobenzenediazoöxide in Vol 2, p B59-L

Diazodinitrophenol, Analytical Procedures. Qualitative Tests

Accdg to StdMethodsChemAnalysis, **2B** (1963), p 1346, DADNPh can be identified by the following procedure:

Dissolve ca 0.05 g of unknown sample (which should be a low density substance, greenish-yel to brn in color), in acetone and then add a large volume of ice water. The formation of a bright-yel ppt might indicate the presence of DADNPh. In order to be sure, prepare a satd soln of the sample in 200 ml of w and add to this 5 ml of 20% NaOH soln. After mixing, observe if there is an evolution of colorless gas and the soln assumes a reddish-brn coloration. These phenomena take place if the sample is DADNPh

US military specification requirements and tests for Diazodinitrophenol intended for use in loading fuze detonators and the manufacture of priming compositions are listed and described in Joint Army-Navy Specification JAN-D-552, issued in 1950. The requirements are also listed in TM 9-1300-214/TO 11A-1-34(1967), p 7-9

Requirements:

1) *Color* - Greenish-yel to brn
2) *Form* - Tubular crysts having a maximum length of 0.2 mm
3) *Granulation* - 100% shall pass thru a No 100 US Std Sieve, which conforms to Federal Spec RR-S-366
4) *Bulk density* (dry) - Minimum 0.4 g/cc
5) *Acidity (as HCl)* - Maximum 0.01%
6) *Sand test* - 0.40 g shall crush not less than 33 g of sand

Sampling

DADNPh is delivered to the plant in waterproof bags packed in drums. Its moisture content shall be not less than 40%. For sampling, remove (by means of a horn or plastic spoon) from each bag in the drum sufficient material to form a total of 100 g, which will serve as a "primary" sample. If the sample appears to be not sufficiently moist, add some water and spread sample on a smooth surface in an even layer. Divide the material into squares ca ½x½ inches, remove from each square ca 1/4th portion, and place them into a wide mouth bottle, provided with a rubber stopper. This will be the so-called "retainer" sample and it should be ca 20 g (dry wt). Repeat the above procedures with each primary sample. Collect the remaining portions of each primary sample into one container, blend them, spread on a flat surface and divide as above into squares. Remove from each square an equal portion to make (after blending) a "composite" (of several primary samples), weighing ca 50 g (dry wt). Keep the bottle with this sample, as well as with the 20 g sample, tightly closed. Label each bottle accordingly

Preparation of a dry sample

Since the sample as received will contain ca 40% water, and since some testing procedures require a dry material, transfer 5 g of "composite" sample on a piece of filter paper placed in a Nutsch funnel, connected to a suction flask. Spread the sample evenly (using a stream of water from a suction bottle) and then apply the suction to air dry the material. Transfer the sample to a paper tray, spreading it in a thin layer, and then dry it in a hot oven at 60° for a minimum of 16 hours. Cool the sample in a desiccator, or in a rubber-stoppered bottle until used

Tests:

1) *Color.* Determine it by visual inspection of wet sample

2) *Form.* Spread a thin layer of wet sample over a 0.5 square inch area of the central surface of a 1-in by 3-in glass microscope slide, in such a manner that the individual crysts are discernible. Measure the crystal length by means of a micrometer eye-piece using a magnification of ca 40

3) *Granulation.* Place ca 10 g of wet sample on a No 100 US Standard Sieve and wash thru with a jet of water

4) *Bulk density.* Fill with dry DADNPh a special, accurately tared (W_1), hollow metallic cylinder of 1-cc capacity. To achieve this, hold the cylinder in a vertical position with open end up and pour the material from a folded piece of glazed paper held at the mouth of the cylinder. Remove the surplus of material from the top and after leveling it, tap the cylinder gently with the finger to cause settling. Weigh accurately, (W_2) and calculate the bulk density from the formula: Bulk d = $W_2 - W_1$

5) *Sand Test.* Conduct this test in the same manner as described in Vol 1 of Encycl, pp XXI & XXII, but instead of using five No 6 caps contg 0.400 g of explosive, use only two

6) *Acidity.* Transfer (using a hard rubber spatula) a weighed (on a rough balance) 4 to 5 g wet composite sample to a 250 ml beaker, add 25 ml of distd w, swirl until the mixture becomes homogeneous and then allow the solid to settle. Decant the supernatant liquid thru an accurately tared sintered glass crucible (designated as A) into a suction flask and treat the deposit in the 250 ml beaker with a second 25 ml portion of distd w. Decant the liquid thru the crucible into the same flask and transfer the combined filtrate to a 100 ml beaker. Add, with constant stirring, 10 ml of 0.1N silver nitrate and if a turbidity results, filter thru a small accurately tared sintered glass crucible of fine porosity (designated as B). Wash with alcohol, then with ether, and dry at 100° for 1 hour; cool and weigh. Transfer the DADNPh from the 250 ml beaker to crucible (A) using a stream of w from a wash bottle, rinse the ppt in the crucible with 3 successive 25 ml portions of water, followed by alcohol and ether. Suck until free from ether, dry at 50° for 1 hr, cool in a desiccator and weigh. Calculate the wt of AgCl to percentage of acidity as HCl as follows:

$$\%\ \text{Acidity (as HCl)} = \frac{25.5B}{A},$$

where: B = weight of AgCl in crucible (B); and
A = wt of dry sample in crucible (A)

As an example of a pyrotechnic mixture contg DADNPh (Diazodinitrophenol) may be cited

Igniter Composition M1A1, which consists of DADNPh, NS (Nitrostarch), powdered wood charcoal and potassium chlorate (percentages are not given). Its US military requirements were covered by Specification MIL-S-10641A (Ord)(1957) and its analytical procedures are described as

Method No 322 in "Laboratory Manual" of A.R. Lusardi, Vol 2, Section 3, PicArsn, Dover, NJ (1962)

Procedures:

a) *Total Volatiles (TV).* Transfer ca 1 g of accurately weighed sample into a tared dish, ca 2 inches in diameter, and place the ensemble in a drying oven at 80° to 85° C for 2 hrs. Cool in a desiccator and weigh

$$\text{Percentage of TV} = (100A)/W$$

where: A = loss in wt of sample in the dish and
W = wt of sample

b) *Diazodinitrophenol.* Transfer ca 1 g of accurately weighed original sample to a 250 ml beaker and add 150 ml of ethylene chloride Heat the beaker & contents on a steam bath for 2 hrs, with occasional stirring. Filter the hot contents of the beaker thru a tared medium-size sintered glass crucible, using suction. Wash the beaker and residue in the crucible with boiling ethylene chloride until the filtrate coming thru the crucible is no longer colored yellow. Dry the crucible in an oven at 80°±5°C for 2 hrs, cool in a desiccator and weigh

$$\text{Percentage of DADNPh} = \frac{100(A\text{-}B\text{-}C)}{WD}$$

where: A = wt of crucible and sample;
B = wt of crucible with residue;
C = wt of TV's in sample as detd in opn (a);
W = wt of sample on a volatile-free basis; and
D = % solubility of DADNPh in ethylene chloride expressed as a decimal fraction obtd as follows:

Solubility of DADNPh in Ethylene Chloride. Place 0.1900±0.0010 g of dry DADNPh (from the lot used in the manuf of Compn M1A1 being analyzed) in a 250 ml beaker and add 150 ml of ethylene chloride. Heat the beaker & contents on a steam bath for 2 hrs with occasional stirring. Filter the hot contents of the beaker thru the same type as above sintered glass crucible and wash the beaker and residue in the crucible with boiling ethylene chloride until the disappearance of yel col of filtrate. Dry the crucible with residue at 100±5° C for 1 hr, cool in a desiccator and weigh. The loss in weight in the crucible corresponds to value D given in the above equation for calculating the percentage of DADNPh. As this value is usually 0.9726, the detn of solubility can be avoided unless there is a doubt

c) *K Chlorate.* Extract the residue in the crucible of opn (b) with ten 5-ml portions of hot w, stirring the residue after the addn of each portion before applying suction. Dry the crucible with residue at 80±5°C for 2 hrs, cool in a desiccator and weigh

$$\text{Percentage of K Chlorate} = \frac{100(A\text{-}B)}{W}$$

where: A = wt of filtering crucible with residue before extraction with w;
B = wt of the same ensemble after extraction with w; and
W = wt of sample on volatile-free basis

d) *Nitrostarch.* Extract the residue of opn (c) with ten 5-ml portions of hot acetone, stirring the residue in the crucible after the addn of each portion before applying suction. Dry the ensemble at 100±5° for 2 hrs, cool in a desiccator and weigh

$$\text{Percentage of NS} = \frac{100(B\text{-}C)}{W}$$

where: B = wt of crucible & residue before extraction with acetone;
C = wt of the same ensemble after extraction with acetone; and
W = wt of sample on a volatile-free basis

e) *Powdered Wood Charcoal.* It is calcd from the following equation:

$$\text{Percentage of Wood Charcoal} = \frac{100(C\text{-}E)}{W}$$

where: C = wt of crucible with residue after extraction with acetone;
E = wt of empty crucible; and
W = wt of sample on a volatile-free basis

Note: In Lusardi's Manual, the K chlorate is detd in a different manner than in our opn (c). It is calcd by subtracting from 100 combined percentages of DADNPh, NS and charcoal, after detg the charcoal content

2,2'-Diazo-3,3'-dioxy-4,6,4',6'-tetranitro-m,m'-biphenol or Bis(2-diazo-3-oxy-4,6-dinitro)-m,m -biphenol. See Vol 2, p B122-L

DIAZODIPHENYLAMINE AND DERIVATIVES

4-Diazodiphenylamine (called 4-Diazodiphenylamin or p-Chinon-anil-diazid in Ger), $C_6H_5.N:C_6H_4:N_2$;mw 195.22, N 21.53%; brn-yel ppt, mp - expl on heating; insol in w; was prepd by treating an aqueous suspension of diphenylamine-diazoniumsulfate with ice-cold NH_3, or by diazotizing 4-aminodiphenylamine with $NaNO_2$ & excess HCl and allowing the cold diazoniumchloride soln to stand with dil NH_3 added

Refs: 1) Beil **16**, 603 2) A. Hantzsch, Ber **35**, 895(1902) 3) G.T. Morgan & F.M.G. Micklethwait, JCS **93**, 613(1908) 4) G.T. Morgan & H.M. Read, JCS **121**, 2710(1922)

Mononitrodiazodiphenylamine,
$O_2N.C_6H_4.N:C_6H_4:N_2$; mw 240.22, N 23.33%. Two derivs are known:

4-Diazo-2'-nitrodiphenylamine, scarlet-red amor ppt, mp - darkens at 30° and explodes at 85-90° or by friction; was obtd when the 2'-nitro-4-aminodiphenylamine hydrochloride soln was diazotized, and well-cooled NH_3 was added to a Na acetate soln of the diazotized salt (Refs 1 & 2, p 12)

4-Diazo-4 -nitrodiphenylamine, red crysts having a Cu luster, mp - explodes when heated to 60-65° or by friction; was prepd by diazotizing 4'-nitro-4-aminodiphenylamine and treating the diazonium salt with NH_3 (Refs 1 & 2, p 11)

Refs: 1) Beil **16**, 603 2) G.T. Morgan & F.M.G. Micklethwait, JCS **93**, 611, 612(1908)

4-Diazo-2 ,4 -dinitrodiphenylamine,
$(O_2N)_2C_6H_3.N:C_6H_4:N_2$; mw 285.22, N 24.56%; red crysts, decomp on exposure to light, mp - expl 110-15°; was prepd by treating an aq soln of 1-(2,4-dinitroanilino)-benzenediazoniumchloride with $KHCO_3$

Refs: 1) Beil **16**, 603 2) G.T. Morgan & F.M.G. Micklethwait, JCS, **93**, 610-11(1908)

4-Diazo-2',4',6'-trinitrodiphenylamine,
$(O_2N)_3C_6H_2.N:C_6H_4:N_2$; mw 330.22, N 25.45%; brn-red crysts (from benz + petr eth), mp - explodes at 120-30°; was obtd when a freshly prepd soln of diazotized 4-amino-2',4',6'-trinitrodiphenylamine was filtered into an aq Na acetate soln

Refs: 1) Beil **16**, 603 2) G.T. Morgan & F.M.G. Micklethwait, JCS **93**, 609(1908)

NOTE: Higher nitrated derivs of Diazodiphenylamine were not found in Beil or in CA thru 1961

Diazoethane (called Diazoäthan in Ger), $CH_3.CH:N:N$; mw 56.07, N 49.97%; gas, bp -19 to -17^b at 89.5mm press; can be prepd by reaction of N-nitroso-N-ethylurea with concd aq KOH in ether and by other methods (Ref 1). Its UV absorption spectrum is reported by Brinton & Volman (Ref 2). Other props are given in Beil 1(Ref 1)

Refs: 1) Beil **1**, (327), [675] & {2658}; and **23**, 28 2) R.K. Brinton & D.H. Volman, JChemPhys **19**, 1394(1951) & CA **46**, 2398 (1952)

Diazoethoxane or Ethylhyponitrite (called Diazoäthoxan or Diäthylhyponitrit in Ger), $C_2H_5.O.N:N.O.C_2H_5$; mw 118.14, N 23.71%; oil lighter than w, bp - on heating begins to decomp and explodes at 80-82°; cannot be distilled even at low press; readily sol in alc, acet & benz; insol in water; d 0.953 at 16°; d 1.0326 at 20°; n_D 1.4047 at 20°; dipole moment (in benz) 1.5 D; was prepd from Ag hyponitrite ($Ag_2N_2O_2$) and ethyl iodide & dry ether in a freezing mixt

Refs: 1) Beil **1**, 328 & {1321} 2) J.R. Partington & C.C. Shah, JCS **1932**, 2595 & CA **27**, 471(1933) 3) E.C.E. Hunter & J.R. Partington, JCS **1933**, 311 & CA **27**, 2671(1933)

1-Diazoethylbenzene of Methylphenyl-diazomethane [called (α-Diazo-äthyl)-benzol or Methyl-phenyl-diazomethan in Ger], $C_6H_5.C(N_2).CH_3$;mw 132.16, N 21.20%;

lt red crysts (from petr eth) at -80°, mp -10° to a dk-red liq; dec briskly at RT within a few minutes, and can explode when in large quantities; sol in org solvs; was obtd by oxidation of acetophenonehydrazone with yel mercuric oxide in cold petr eth (Refs 1, 2 & 3). Some later refs in the literature report reactions of Diazoethylbenzene

Refs: 1) Beil **7**, (151) 2) H. Staudinger & A. Gaule, Ber **49**, 1907(1916) & JCS **110** I, 848(1916) 3) W.R. Branford & J.S.Stevens, JCS **1952**, 4677 & CA **47**, 10508(1953)

Diazoguanidine Cyanide. See Cyanoazoguanidine in Vol 3, p C587-R

Diazoguanidine Picrate. $CH_4N_5 + C_6H_3N_3O_7$. This salt is mentioned as one of the ingredients of a detonator chge. It may be mixed with Tetryl. The detonator chge is made by mixing a primary initiation chge (LA or LST in Al shell or MF in a Cu or Cu-alloy shell) with the Diazoguanidine Picrate with or w/o Tetryl added. No description of the salt was found in the literature and the patent was not available to us

Refs: 1) Beil-not found 2) ICI of Australia & New Zealand, AustralianP 102202 (Oct 1937) & CA **32**, 2753(1938)

Diazoguanylazide or Nitroso-aminoguanylazide (called Diazoguanylazid or Nitrosoaminoguanylazid in Ger), $N_3.C(:NH).NH.N:N.OH$ or $N_3.C(:NH).NH.NH.NO$; known only in the form of its salts such as:

Sodium salt, $NaCHN_7O$, obtd by treating guanyldiazoguanyltetrazene with dil NaOH. The add of Cu acetate gives the *Copper salt*, $CuCHN_7O$, light greenish-blue plts, very expl salt

Refs: 1) Beil **3**, (60) & {239} 2) K.A. Hofmann et al, Ber **43**, 1093(1910) & JCS **98** I, 446(1910)

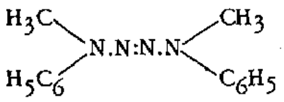
3,5-Diazoheptane; 3.5-Dinitro Derivative. See under Diethyl-methylenediamine in this Vol

Diazohydrocarbons. 1-Substituted-1-nitroso-3-nitroguanidines when treated with aq KOH gave diazohydrocarbons. McKay et al (Ref) used this method to prepare Diazomethane, Diazoethane, Diazo-n-propane and other Diazocompds. See Ref 2 for their decompn studies

See also Diazochemistry and Diazocompounds

Refs: 1) A.F. McKay et al, CanJRes **28B**, 683-88(1950) 2) G.D. Buckley & N.H. Ray, JCS **1952**, 3701-04 & CA **47**, 8006(1953) (Decompn of aliphatic Diazocompds by trimethyl borate)

Diazohydrazides. This name was given by Wohl & Schiff (Ref 2) to compds of the general formula $R'.N:N.NR''.NH_2$, where R' is an H or alkyl & R'' is an aryl group. These compds may be considered as derivatives of hypothetical "tetrazene", $HN:N.NH.NH_2$, and they resemble the compd



$$\begin{matrix}H_3C & & CH_3\\ & N.N:N.N & \\ H_5C_6 & & C_6H_5\end{matrix}$$

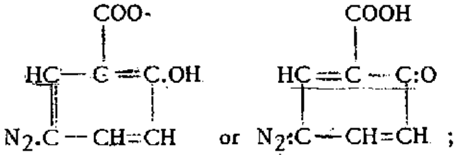
prepd earlier by Fischer (Ref 1) and called "Dimethyldiphenyltetrazone". Wohl & Schiff (Ref 2) prepd a number of similar compds and suggested that the name "Diazohydrazides" was simpler. It allowed isomerides to be readily distinguished and was in better agreement with their chem nature

Diazohydrazides are cryst substances which explode when heated but not when subjected to impact or friction. They are sol in alc, eth, acet or benz; sl sol in petr eth; insol in w or dil acids. On oxidation they yield Bisdiazotetrazones, R N:N.NR$''$.N:N.NR .N:NR , which are generally light in color, but darken in the light, and explode when subjected to heat, impact or friction (Ref 2)

See also Refs 3 & 4 for addnl info on Diazohydrazides

Refs: 1) E. Fischer, Ann **190**, 172(1878) 2) A. Wohl & H.Schiff, Ber **33**, 2741-58(1900) & JCS **78** I, 706-08(1900) 3) O. Dimroth & G. deMontmollin, Ber **43**, 2904-15(1910) & CA **5**, 493-95(1911)(Diazohydrazides) 4) K.A. Hofmann & H. Hock, Ber **44**, 2946-56(1911) & CA **6**, 755(1912)(Diazohydrazide from Diazotetrazole)

5-Diazo-2-hydroxybenzoic Acid or 5-Diazosalicylic Acid [called 5-Diazo-salicylsäure or Benzochinon-(1.4)-diazid-(4)-carbonsäure-(2) in Ger]

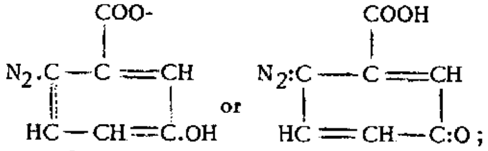


; mw 164.12, N 17.07%; yel ppt (from w) or blue-gray ndls; mp - explodes 155-162°; mod sol in hot w & aq Na_2CO_3; sl sol in cold w & org solvs; was prepd by diazotizing the hydrochloride of 5-amino-salicylic acid with NaNO in alcoholic HCl soln

See Diazosalicylic Acid

Refs: 1) Beil **16**, 553 & [299] 2) E. Puxeddu, Gazz **59**, 13(1929) & CA **23**, 3913(1929) 3) Sax (1963), 673

6-Diazo-3-hydroxybenzoic Acid [called 6-Diazo-3-oxy-benzoesäure or Benzochinon-(1.4)-diazid-(1)-carbonsäure-(2) in Ger],



mw 164.12, N 17.07%, ndls, mp - explodes at 169°; diffc sol in cold w; was prepd by diazotizing 6-amino-3-hydroxybenzoic acid with $NaNO_2$ in ice-cold dil HCl

Refs: 1) Beil **16**, [299] 2) H. Limpricht, Ann **263**, 234(1890) 3) E. Puxeddu, Gazz **59**, 14(1929) & CA **23**, 3913-14(1929)

Diazoimide. A current CA name for Hydrazoic Acid or Hydrogen Azide described in Vol 1, p A537-L

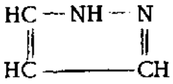
Diazoimide of Benzenesulfonyltolylene. See Benzenesulfonyltolylenediazoimide in Vol 2, p B62-R

Diazoimide of Benzenesulfonylxylylene. See Benzenesulfonylxylylenediazoimide in Vol 2, p B63-L

Diazol. One of the names for Diazodinitrophenol, listed under Diazophenol and Derivatives and described in Vol 2, p B59-L under Benzenediazoöxide and Derivatives

1,2-DIAZOLE AND DERIVATIVES

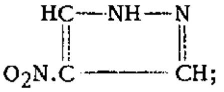
1,2-Diazole or Pyrazole (called Pyrazol in Ger),



; mw 68.08, N 41.15%; ndls or prisms (from petr eth), mp 69-70°, bp 184-85° at 719mm of Hg; d 1.0012 at 99.8°; sol in w, alc, eth & benz; can be prepd by reaction of α-bromoacrolein or acroleindibromide with cold hydrazine hydrate soln, or by other methods; forms numerous salts and addn compds. Other props are given in Beil

Ref: Beil **23**, 39, (15) & [33]

4-Nitro-1,2-Diazole or 4-Nitro-pyrazole (called 4-Pyrazol in Ger),



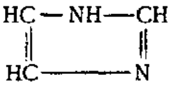
mw 113.08, N 37.16%; ndls (from benz), mp 162°, explodes when heated on a Pt foil, bp 323°; sol in alc & eth; sl sol in cold w & benz; was prepd by nitration of 1,2-diazole with mixed HNO_3 - H_2SO_4 acids and by other methods (Ref). Other props are given in Beil

Refs: 1) Beil **23**, 44 2) F. Reicheneder & K. Dury, BelgP 660636(1965) & CA **63**, 18096(1965)

NOTE: Higher nitrated derivs of 1,2-Diazole were not found in Beil or in CA thru 1961

1,3-DIAZOLE AND DERIVATIVES

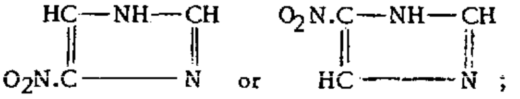
1,3-Diazole,Glyoxaline or Imidazole (called Imidazol or Glyoxalin in Ger),



; mw 68.08, N 41.15%; wh prisms (from benz), mp 88-90°, bp 255-56°, bp 138.2° at 12mm of Hg; d 1.0303 at 100.9°; sol in pyridine & chlf; mod sol in w & alc; sl sol in eth; can be prepd from glyoxal & NH_3 with or without the addn of formaldehyde; from imidazolthione-2 & HNO_3; by decarboxylation of imidazole-4,5-dicarboxylic acid; and by other methods (Refs 1, 2 & 3)

This compd forms numerous salts & addn compds. The *Silver Nitrate salt,* $2C_3H_4N_2 + AgNO_3$, crysts (from alc), mp 129-31°; explodes on heating to a higher temp (Ref 1)
Refs: 1) Beil **23**, 45, (17) & [34] 2) K. Hofmann, "Imidazole and Its Derivatives", Interscience, NY (1953), 447pp 3) OrgSynthColl-Vol **3**(1955), 471-74[original by H.R. Snyder et al, OrgSynth **22**(1942), 65-68]

4 (or 5)-Nitro-1-3-Diazole or 4 (or 5)-Nitroimidazole [called 4(or 5)-Nitro-imidazol in Ger],



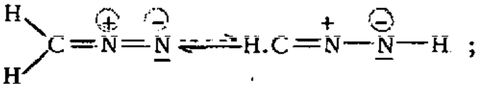
mw 113.08, N 37.16%; ndls (from alc or glacial acet acid), mp 312-13°, sol in hot HCl & glacial acet acid; sl sol in hot w & alc; insol in eth & chlf; can be prepd by heating imidazole with nitrosyl hydrogen sulfate on a water bath, or by heating 5-nitro-imidazole carbonic acid to 150° (Ref). Other props are given in Beil
Ref: Beil **23**, 50, (18) & [42]

NOTE: Higher nitrated derivs of 1,3-Diazole were not found in Beil or in CA thru 1961

Diazomethane (called Diazomethan in Ger), $CH_2:N\vdots N$; mw 42.04, N 66.64%; poisonous yel gas condensing to a yel liq, bp -24 to -23° and solidifying in pale-yel crysts, fr p -145°; explodes violently when heated to a higher temp; sol in eth; more important methods of prepn are from nitrosomethylurea, nitrosomethylurethane, a mixt of chloroform & hydrazine hydrate, or 1-methyl-1-nitroso-3-nitroguanidine by reaction with KOH; from nitrosyl chloride & methylamine; by treatment of the Na salt of formaldehyde oxime with chloramine; and by other methods

Its *Sodium salt,* $NaCHN_2$, colorless flaky ppt, explodes violently when dry even in an atmosphere of N when lightly touched, and spontaneously in light (Ref 1, p {2674})
Refs: 1) Beil **1**, (318), [650] & {2597, 2674}; **23**, 25 2) E.W.R. Steaci, JPhysChem **35**, 1493-95(1931) & CA **25**,3224(1931) (Thermal decompn & expln at low temps) 2a) F.W. Kirkbride & R.G.W. Norrish, JCS **1933**, 119-26 (Absorption spectrum and photochemical decompn of Diazomethane) 2b) Thorpe **3**(1939), 599-600 3) M.D. Owen, CurrentSci **12**, 228(1943) & CA **38**, 2006(1944) (Preparation) 4) CollOrgSynth **2**(1943), 165-67 5) CollOrgSynth **3**(1955), 244-48 6) Sax (1957), 547 7) H.K. Reimlinger, ChemBer 2547-50(1961) & CA **56**, 1337(1962)(N,N - Dinitroso-N,N'-dimethyloxamide as a starting material for Diazomethane) 8) CollOrg-Synth **4**(1963), 250-53 9) Sax(1963), 672

iso-Diazomethane (called Isodiazomethan in Ger)



mw 42.04, N 66.64%; pale-yel oil of low viscosity, fr p - begins to decomp at 15° and then explodes at 35-40°; can be synthesized from an ether suspension of *Diazomethyl lithium,* $(CHN_2)Li$, by acid hydrolysis. Diazomethyl lithium explodes on contact with air; is obtd by reaction of N_2O & CH_3Li or from diazomethane & CH_3Li (Refs 1, 3 & 4). Iso-diazomethane can also be prepd by the action of acetic acid in ether on the Na salt of diazomethane (Ref 2)
Refs: 1) Beil **1**, {2604} 2) E. Müller & W. Kreutzmann, Ann **512**, 264-75(1934) & CA **29**, 769(1935) 3) E. Müller & D. Ludsteck, ChemBer **87**, 1887-95(1954) & CA **50**, 232 (1956) 4) E. Müller & W. Rundel, ChemBer **90**, 1302-06, 2673-78(1957) & CA **53**, 2074 (1959); CA **54**, 10839(1960)

Diazomethanedisulfonic Acid or Diazomethionic Acid (called Diazomethandisulfonsäure or Diazomethionsäure in Ger), $N\vdots N:C(SO_3H)_2$; obtd in the form of its *Potassium salt,* $K_2CO_6N_2S + H_2O$, yel crysts & orn-yel ndls or prisms, mp - explodes ca 210°; mod sol in w; was prepd by treating K aminomethanedisulfonate with HNO_2 and by other methods
Refs: 1) Beil **3**, (58), [99] & {235} 2) H. von Pechmann & P. Manck, Ber **28**, 2377(1894) 3) H J. Backer, Rec **48**, 619(1929) & CA **23**, 4445(1929)

4-Diazo-1-[6-methyl-2-benzthiazolyl]- benzene or **6-Methyl-2-[4-diazophenyl]-benzthiazole,** $C_{14}H_{10}SN_3.OH$; known in the form of its salts, some of which are explosive:

Chlorate: $C_{14}H_{10}SN_3.ClO_3$, yell amorphous powder, mp - explodes violently at 110°; diffc sol in w

Nitrate: $C_{14}H_{10}SN_3.NO_3$, yell-brown ppt which explodes in dry state at 145°

Chromate: $C_{14}H_{10}SN_3.HCrO_4$, yell ppt which detonates at 120°

Carbonate: $C_{14}H_{10}SN_3.HCO_3$, red-brown amorphous powder, mp 128° with weak explosion

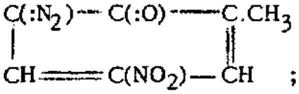
Nitroprusside: $(C_{14}H_{10}SN_3)_2[Fe(CN)_5NO]$, lt yell amorphous powder, which explodes at 140°

Refs: 1) Beil **27**, [507-8] 2) G.T. Morgan & D. Webster, JCS **119**, 1076 (1921)

Diazomethyl Lithium, See under isoDiazomethane

Diazomethylbenzene. See Diazotoluene

6-Diazo-2-methyl-4-nitro-2,4-cyclohexadiene-1-one (CA nomenclature) (called 5-Nitro-2-oxy-3-diazatoluene by Hodgson & Ward),

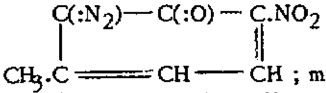


$$\begin{array}{l} C(:N_2)-C(:O)-C.CH_3 \\ | \qquad\qquad\qquad\qquad \| \\ CH{=}C(NO_2)-CH \end{array}$$

; mw 179.13, N 23.46%; lt-yel crysts (from hot w), mp 123° (decomp); was obtd as an intermediate when 5-nitro-3-amino-o-cresol was diazotized

Refs: 1) Beil-not found 2) H.H. Hodgson & E.R. Ward, JCS **1945**, 665 & CA **40**, 1149 (1946)

6-Diazo-5-methylyl-2-nitro-2,4-cyclohexadiene-1-one (CA nomenclature) (called 3-Methyl-6-nitro-1-chinon-2-diazid in Ger),



$$\begin{array}{l} \qquad C(:N_2)-C(:O)-C.NO_2 \\ \qquad | \qquad\qquad\qquad\qquad \| \\ CH_3.C{=}CH-CH \end{array}$$

; mw 179.13, N 23.46%; rust-brn, flat ndls, mp 128-29°, explodes violently on rapid heating; was obtd by adding KI dropwise in water to $2,3,4\text{-}H_2N.(NO_2)_2C_6H_2.CH_3$ in concd H_2SO_4, after treatment with HSO_3NO_2, and allowing to stand for 36 hrs

Refs: 1) Beil-not found 2) W. Theilacker & F. Baxmann, Ann **581**, 130 (1953) & CA **48**, 12116 (1954)

DIAZONAPHTHALENE AND DERIVATIVES

Diazonaphthalene or Naphthalenediazonium Hydroxide, $C_{10}H_7N_2OH$, mw 172.18, N 16.27%. Exists in three modifications, all in the form of salts, some of which are expl (Ref 1)

1(or alpha)-Diazonaphthalene [called 1(or a)-Diazonaphthalin or Naphthalin-diazonium-hydroxyd-(1) in Ger], $C_{10}H_7.N(:N).OH$; forms the following unstable salts:

Borofluoride, $C_{10}H_7N_2.BF_4$, mp - dec at 113°

Chloride, $C_{10}H_7N_2.Cl$, orn-yel crysts (from alc), mp 96° (dec) and explodes on further heating

Dichloroiodide, $C_{10}H_7N_2.Cl_2I$, lt-yel fine cryst powd, mp 98° (dec)

Mercuric chloride, $C_{10}H_7N_2.Cl + HgCl_2$ lt-yel crysts, mp 120-21° (dec)

a-Naphthalenethiosulfonate, $C_{10}H_7.N_2.S.SO_2.C_{10}H_7$, brn crysts, very unstable

Nitrate, $C_{10}H_7N_2O.NO_2$, col crysts, mp explodes on heating, and detonates violently by impact or friction

Perchlorate, $C_{10}H_7N_2.ClO_4$, double-refractive crysts, very expl when dry

Sulfate, $C_{10}H_7.O.SO_3H$, yel crysts (from alc + eth), mp 115° (dec) and exploding mildly

2(or beta)-Diazonaphthalene [called 2(or β)-Diazonaphthalin or Naphthalin-diazonium-hydroxyd-(2) in Ger], $C_{10}H_7.N(:N).OH$; forms, with bases, some expl salts:

Benzenethiosulfonate, $C_{10}H_7.N_2.S.SO_2.C_6H_5$, lemon-yel ndls, dec on standing in dry state at RT

Borofluoride, $C_{10}H_7.N_2.BF_4$, ndls or plts (from MeOH or acet), mp - turns red at 105° & dec at 108°

Chloride, $C_{10}H_7.N_2.Cl$, yel ndls, mp - explodes on heating and detonates on impact

Dichloroiodide, $C_{10}H_7.N_2.Cl_2I$, lt-yel crysts, mp 90° (dec)

Mercuric chloride, $C_{10}H_7.N_2.Cl + HgCl_2$, yel crysts, mp 120-25° (dec)

a-Naphthalenethiosulfonate, $C_{10}H_7N_2.S.SO_2.C_{10}H_7$, lt-yel crysts, very unstable

Nitrate, $C_{10}H_7.N_2.O.NO_2$, col ndls (from glac acetic acid or MeOH), explodes on heating or impact

Perchlorate, $C_{10}H_7N_2.ClO_4$, crysts, very expl when dry

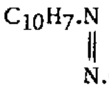
Sulfate, $C_{10}H_7.N_2.O.SO_3H$, wh plts (from MeOH + eth), mp, explodes on heating

p-Toluenethiosulfonate, $C_{10}H_7.N_2.S.SO_2.C_6H_4.CH_3$, yel ndls, very unstable

Unnamed Salt, $C_{10}H_7.N_2.Br + 2CuBr$, fiery-red ppt, dec on standing at RT

Unnamed salt, $2C_{10}H_7.N_2.SCN + Co(SCN)_2$, pale-green ppt, mp - dec at 70° and explodes on contact with H_2SO_4 (See Ref 2)

iso-Diazonaphthalene [called α(& β)-Naphthalin-isodiazohydroxyd or α(& β)-Naphthalin-anti-diazohydroxyd in Ger],



$C_{10}H_7.N{=}N.OH$; forms with bases, some expl salts:

The *β-Silver salt*, $AgC_{10}H_7N_2O$, wh crysts, explodes on heating rapidly (See Ref 3)

Refs: 1) Beil **16**, 510, (361) & [282] 2) Beil **16**, 511, (361) & [283] 3) Beil **16**, 511-12

Nitro Derivatives of Diazonaphthalene

Mononitrodiazonaphthalene, $O_2N.C_{10}H_6.N(:N).OH$ mw 217.18, N 19.35%. Several isomers are known:

4-Nitro-1-diazonaphthalene or 4-Nitronaphthalene-1-diazoniumhydroxide, forms a *Nitrate*, lt-yel ndls, explodes mildly on heating (Ref 1)

5-Nitro-1-diazonaphthalene, the normal *sodium salt*, ppt (from alc + eth), begins to decomp below 100°; the isodiazo *sodium salt*, dk-yel ndls (from alc + eth), dec at 165° (Ref 2)

1-Nitro-2-diazonaphthalene or 1-Nitronaphthalene-2-diazoniumhydroxide, its *Nitrate salt*, $O_2N.C_{10}H_6.N_2.ONO_2$, lt-yel ndls, explodes on heating; the *Sulfate salt*, $O_2N.C_{10}H_6.N_2.O.SO_3H$, orn-yel ndls explodes mildly on heating (Ref 3)

6-Nitro-2-diazonaphthalene or 6-Nitronaphthalene-2-diazoniumhydroxide, forms a Sulfate, ppt, which decomp in acid soln at RT (Ref 2)

Refs: 1) Beil **16**, 511 & [282] 2) Beil **16**, [283] 3) Beil **16**, 513, (361) & [283]

NOTE: Higher nitrated derivs of Diazonaphthalene were not found in Beil or in CA thru 1961

DIAZONAPHTHOL AND DERIVATIVES

Diazonaphthol, $N_2.C_{10}H_5.OH$; mw 170.16, N 16.46%. Two isomers are known:

2-Diazo-1-naphthol [called 2-Diazo-naphthol-(1) or Naphthochinon-(1,2)-diazid-(2) in Ger], golden-yel ndls or plts, sensitive to light, mp 76-77° and explodes at 112°; sol in alc; was prepd by action of $NaNO_2$ on 2-amino-1-naphthol in HCl in the presence of Cu sulfate, and by other methods (Ref 1). Its *4-Bromo deriv* yel ndls (from w), decomp violently at 148-51°

1-Diazo-2-naphthol [called 1-Diazo-naphthol-(2) or Naphthochinon-(1,2)-diazid-(1) in Ger], wine-yel or yel-brn prisms from petr eth or dil alc), mp 94-95°, highly expl on further heating and light sensitive; can be prepd by diazotizing 1-amino-2-naphthol in HCl & a Cu salt, and by other methods (Ref 2)

Refs: 1) Beil **16**, 533 & [289] 2) Beil **16**, 534 & [290] 3) J.D.C. Anderson et al, JCS **1949**, 2082 & CA **44**, 6725 (1950) (Dipole moment & spectroscopic data for diazonaphthols)

Nitroderivatives of Diazonaphthol

Mononitrodiazonaphthol, $O_2N(N_2).C_{10}H_4.OH$; mw 215.16, N 19.53%. Several isomers are known:

3-Nitro-2-diazo-1-naphthol, crysts (from benz), mp 187° (dec); was prepd by diazotization of 1-chloro-3-Nitro-2-naphthylamine and pouring the diazonium soln in aq Na acetate (Ref 9)

6-Nitro-2-diazo-1-naphthol [called 6-Nitro-2-

diazo-naphthol-(1) or 6-Nitro-naphthochinon-(1.2)-diazid-(2) in Ger], golden-yel plts or ndls (from acet), mp - explodes at 142-45°; sol in chlf, acet & acet acid; sl sol in alc, benz & petr eth; insol in w; was prepd by diazotizing 1,6-dinitro-2-naphthylamine (Refs 1 & 3, p 2211). See also Ref 7

8-Nitro-2-diazo-1-naphthol [called 8-Nitro-2-diazo-naphthol-(1) or 8-Nitro-naphthochinon-(1,2)-diazid-(2) in Ger], lt-orn prisms (from acet), mp - explodes violently at 155-60°; was prepd by diazotizing 1,8-dinitro-2-naphthylamine (Refs 1 & 3)

2-Nitro-4-diazo-1-naphthol (called 2-Nitro-4-diazonaphthalene-1-oxide by Hodgson & Smith), pale-yel ndls (from alc), mp 163° (dec); prepd by diazotizing 2-nitro-4-amino-1-naphthol (Ref 5)

4-Nitro-1-diazo-2-naphthol [called 4-Nitro-1-diazo-naphthol-(2) or 4-Nitro-naphthochinon (1.2)-diazid-(1) in Ger], yel ndls (from petr eth), mp 130-34° (dec); readily sol in acet, glacial acet acid & benz; mod sol in alc; sl sol in w; was prepd by diazotizing 2,4-dinitro-1-naphthylamine (Refs 1, 4 & 6)

7-Nitro-1-diazo-2-naphthol, crysts (from dioxane), mp 160-62° (dec); was obtd when the 2,7-dinitro-1-naphthylamine diazotized salt was poured into water contg Na acetate (Ref 8)

Refs: 1) Beil **16**, 533 2) Beil **16**, (365) 3) F. Gaess & A. Ammelburg, Ber **27**, 2211, 2244 (1894) & JCS **66** I, 605 (1894) 4) G.T. Morgan & E.D. Evans, JCS **115**, 1130 (1919) & CA **14**, 733 (1920) 5) H.H. Hodgson & E.W. Smith, JCS **1935**, 673 & CA **29**, 5099 (1935) 6) H.H. Hodgson & S. Birtwell, JCS **1943**, 322 & CA **37**, 6259 (1943) 7) H.H. Hodgson & H.S. Turner, JCS **1944**, 9 & CA **38**, 2031 (1944) 8) A. Hardy & E.R. Ward, JCS **1957**, 2637 & CA **51**, 15477 (1957) 9) E.R. Ward et al, JCS **1957**, 4820 & CA **52**, 10027 (1958)

Dinitrodiazonaphthol, $C_{10}H_4N_4O_5$, mw 260.16, N 21.54%. Several isomers are known:

3,5-Dinitro-1-diazo-2-naphthol, crysts (from aq alc), mp 128-30° (dec); obtd by pouring the diazotized soln of 1,3,5-trinitro-2-naphthylamine into water (Ref 5)

3,8-Dinitro-1-diazo-2-naphthol, crysts (from acet), mp 220° (dec); prepd from diazotized 1,3,8-trinitro-2-naphthylamine by pouring the diazonium soln into water (Ref 5)

4,5-Dinitro-1-diazo-2-naphthol, yel ndls (from glacial acetic acid), mp - dec slowly when heated gradually; explodes at 160° when heated rapidly; was prepd by dissolving 2,4,5-trinitro-p-toluenesulfon-1-naphthalide in H_2SO_4 below 20° and mixing with $NaNO_2$ in H_2SO_4 & glacial acetic acid and pouring the diazotized soln into water (Ref 2)

4,7-Dinitro-1-diazo-2-naphthol, crysts (from dioxane), mp 158° (decomp); was obtd when diazotized 2,4,7-trinitro-1-naphthylamine was poured into water (Ref 4)

4,8-Dinitro-1-diazo-2-naphthol, golden-yel ndls (from aq dioxane), mp 152-55° (dec); prepd by pouring the diazotized soln of 2,4,8-trinitro-1-naphthalene on crushed ice (Ref 3)

Refs: 1) Beil-not found 2) H.H. Hodgson & H.S. Turner, JCS **1944**, 9 & CA **38**, 2031 (1944) 3) E.R. Ward & L.A. Day, JCS **1951**, 785 & CA **45**, 9014 (1951) 4) A. Hardy & E.R. Ward, JCS **1957**, 2639 & CA **51**, 15477 (1957) 5) E.R. Ward et al, JCS **1957**, 4822 & CA **52**, 10028 (1958)

NOTE: Higher nitrated derivs of Diazonaphthols were not found in Beil or in CA thru 1961

Diazonaphtholcarboxylic Acid, $N_2(HO).C_{10}H_4.COOH$; mw 214.17, N 13.08%. Two isomers are known:

1-Diazo-2-naphthol-4-carboxylic Acid [called 1-Diazo-naphthol-(2)-carbonsäure-(4) or Naphthochinon-(1.2)-diazid-(1)-carbonsäure-(4) in Ger], yel plts (from MeOH), mp - deflagrates at 151°; obtd together with 1,2-naphthoquinone-4-carboxylic acid by diazotizing 4-amino-3-hydroxy-1-naphthoic acid with $NaNO_2$ & $CuSO_4$ in water (Refs 1 & 2)

1-Diazo-2-naphthol-3-carboxylic acid [called 1-Diazo-naphthol-(2)-carbonsäure-(3) or Naphthochinon-(1.2)-diazid-(1)-carbonsäure-(4) in Ger], dk-yel rodlets or ndls having a blue-black luster, mp 182-83° (dec); prepd by diazotizing 4-amino-3-hydroxy-2-naphthoic acid with $NaNO_2$ & $CuSO_4$ in w or with $NaNO_2$ & HCl (Refs 1 & 2)

Refs: 1) Beil **16** [299] 3) R. Lesser & G. Gad, Ber **58B**, 2555, 2556(1925) & CA **20**, 1233(1926)

1-Diazo-3-nitrobenzene Perchlorate or 1-Diazo-m-nitroanilino-perchlorate (called *Blitzpulver* in Ger)(Flash Powder). See m-Nitrobenzene-diazonium Perchlorate in Vol 2, p B56-R

5-Diazo-4-iso-nitrosomethyl-uracil. See under Diazouracil and Derivatives

5-Diazo-4-iso-nitrosomethyl-uracil, Nitro Derivative. See under Diazouracil and Derivatives

Diazonium Chloride of Aminodicarboxybiphenyl. See Aminodicarboxybiphenyldiazonium Chloride in Vol 1, p A195-L

Diazonium Compounds. See Diazocompounds

Diazonium Compounds & Sodium Sulfide. Diazonium compds, such as diazotized *4-chloro-o-toluidine* treated with Na_2S, NaHS or Na polysulfide solns, form dangerous expls (Ref 1). Hodgson (Ref 2) reported that the addn of *o-nitrophenyldiazonium chloride* to Na_2S_2 soln at 0° gives a red ppt which explodes when touched with a glass rod. Similarly, when *diazotized m-chloroaniline* is added dropwise to Na_2S_2 soln, every drop reacts violently

Refs: 1) P. Nawiasky, C & EN **23**, 1247 (1945) & CA **39**, 4228(1945) 2) H.H. Hodgson, Chem & Ind **1945**, 362-63 & CA **40**, 5251(1946)

Diazonium Derivatives of Anilinobenzene. See Anilinobenzenediazonium Hydroxide and Derivatives in Vol 1, p A421

Diazonium Derivatives of Azobenzene. See Azobenzenediazonium Derivatives in Vol 1, p A650

Diazonium Derivatives of Benzaminobenzene. See Benzanilide-diazonium Derivatives in Vol 2, p B41-L

Diazonium Derivatives of Benzanilide. See Vol 2, p B41-L

Diazonium Derivatives of Benzoquinone. See Vol 2, p B81-L

Diazonium Fluoroborates. Dr R.P. Johnson & J.P. Oswald of Abbot Laboratories, North Chicago, Ill urge caution regarding the stability of aromatic diazonium fluoroborate salts. These salts are intermediates in the Schiemann-Balz reaction for replacing the aromatic primary amino group by fluorine

According to a literature prepn (Ref 1), Johnson & Oswald prepd *3-Pyridyldiazonium fluoroborate,* $C_5H_4N.N_2BF_4$, and spread the salt on Al foil to dry. A small sample was removed from the damp solid to determine its decomp point. The test sample exploded at 47°, the remaining material detonated (Ref 2)

Doak & Freedman of Raleigh, NC (Ref 3) point out that literature refs indicate this compd to be notoriously unstable. These authors report having prepd a considerable number of diazonium fluoroborates, and conclude that it is important to check the decompn point on a small sample of any new diazonium fluoroborate before allowing the bulk of the material to dry. They suggest that if a decompn point is above 100°, the salt can be safely dried and stored, however, if the decompn point is between RT & 100°, the salt may be dangerous. Any diazonium fluoroborates which show signs of decompn at or below RT should never be allowed to dry completely

Refs: 1) T. Talik & Z. Talik, RoczChem **38**(5), 777(1964) & CA **61**, 10653(1964)
2) Anon, C & EN **45**, No 44, p 44(16 Oct 1967)
3) G.O. Doak & L.D. Freedman, C & EN **45**, No 53, p 8(Letter to Editor)(18 Dec 1967)

Diazonium Hydroxide of Aminobenzene. See Aminobenzenediazonium Hydroxide in Vol 1, p A187-L

Diazonium Salts. These are substances having the general formula $\left[\begin{array}{l}R.N\\ \ \ \vdots\\ \ \ N\end{array}\right].A$ where R is a radical such as C_6H_5-, C_6H_5O-, and A is an acid radical such as Cl-, ClO_4-, NO_3-. The most important diazonium salts are described separately

Hantzsch (Ref 1) examined some diazonium halides & thiocyanates, which were colored expl compds. He suggested that their color & expl props are interrelated and are due to the presence of the "azo-isomer", in which halogen or thiocyanate is linked directly to N as in $\begin{array}{l}Ar.N.(Br,Cl)\\ \ \ \ \ \ \ \ \vdots\\ \ \ \ \ \ \ \ N\end{array} \rightleftharpoons \begin{array}{l}Ar.N\\ \ \ \ \ \ \ \colon\\ \ \ \ \ \ \ N.(Br,Cl)\end{array}$

Thus, the colored expl diazohalides are regarded as consisting chiefly of a true azo-compd, whereas the colorless diazohalides, which are much less expl, are regarded as diazonium salts. See also Ref 3

Herz (Ref 2) claimed that perchlorates of many negatively substituted diazonium compds are powerful & brisant expls suitable as primer compds in detonators. He suggested also that the perchloric acid diazo compds of mono- & polynitrated aromatic hydrocarbons can be used in primers & detonators

Recently in the US numerous aromatic diazonium chromates, nitrates, perchlorates, persulfates & picrates were examined for their suitability in primers. Some of the conclusions are: 1) *Chromates & Persulfates* are of the same order of sensitivity as MF 2) *Nitrates* are too reactive, too sensitive and have too little power 3) *Perchlorates & Picrates* are too sensitive & dangerous to handle

See also Diazo compounds and Diazotization

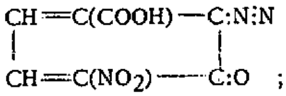
Refs: 1) A. Hantzsch, Ber **33**, 2179-88 (1900) & JCS **78** I, 568(1900) 2) E. Herz, GerP 258679(1911) & CA **7**, 2687(1913); BritP 27198(1912) & CA **8**, 1672(1914) 3) R.P. Johnson & J.P. Oswald, C & EN **45**, No 44, p 44(16 Oct 1967)(Deton of 3-Pyridyldiazonium fluoroborate reported at Abbott Laboratores, North Chicago, Ill)

Diazoöxide of Benzene. See Benzenediazoöxide or Benzoxadiazole in Vol 2, p B58

Diazo Oxides. Diazo oxides are obtd from substituted aminophenols & aminonaphthols. They are also obtd from aminophenolcarboxylic acids with the amino group either ortho or para to the hydroxyl group. In comparison with diazonium salts (qv), diazo oxides are more stable, more deeply colored, less soluble and less reactive. They do not readily couple to give azo colors except in strongly alkaline solns. Although diazo oxides are generally stable, some (such as 4,6-Dinitrobenzene-2-diazo-1-oxide, Vol 2, p B59-L) are used as detonating expls

Refs: 1) G.T. Morgan & J.W. Porter, JCS **107** I, 645-59 (Constitution of internal diazo oxides) 2) G.T. Morgan & H.P. Tomlins, JCS **111**, 497-506(1917)(Constitution of internal diazo oxides) 3) G.T. Morgan & E.D. Evens, JCS **115**, 1126-40(1919)(Constitution of internal diazo oxides) 4) Thorpe **3**(1939), 580, 594 & 597 5) Thorpe **8**(1947), 274 6) Groggins(1952), 144-45 (Prepn & reactions of diazo oxides, not found in later editions)

2-Diazo-3-oxidobenzoic Acid, 4-Nitro Derivative. (called 6-Diazo-4-nitro-5-oxo-1,3-Cyclohexadiene-1-carboxylic Acid in CA)



$$\begin{array}{l}CH{=}C(COOH){-}C{:}N{:}N\\ |\qquad\qquad\qquad\ \ \ |\\ CH{=}C(NO_2){-\!-}C{:}O\end{array}$$

; mw 209.12, N 20.10%; orn-colored powd + H_2O, stable in darkness, turns brn-red in light, mp - loses H_2O at 80° & deflagrates at 176°; decomp in boiling w; sol in alc & eth; sl sol in cold w; insol in benz; was prepd by adding HNO_3 to 4-nitroanthranilic acid in concd H_2SO_4 below -5°, pouring the ppt on ice and extracting with eth (Ref 2)

Refs: 1) Beil-not found 2) G. Berti et al, Gazz **85**, 1634, 1641-42(1955) & CA **50**, 10041(1956)

DIAZOPHENOL AND DERIVATIVES

Diazophenol, $O.C_6H_4.N{:}N$; mw 120.11, N 23.33%.

2-Diazophenol. See Benzenediazoöxide, Vol 2, p B58-L

4-Diazophenol. See Benzenediazoöxide, Vol 2, p B58-L and 4-Diazo-2,5-Cyclohexadien-1-one in this Vol

Mononitroderivs

4-Nitro-2-diazophenol. See 4-Nitrobenzene-2-diazo-1-oxide under Mononitrobenzenediazooxide, Vol 2, p B58-R

5-Nitro-2-diazophenol. See 5-Nitrobenzene-2-diazo-1-oxide under Mononitrobenzenediazoöxide, Vol 2, p B58-R

2-Nitro-4-diazophenol. See 2-Nitrobenzene-4-diazo-1-oxide under Mononitrobenzenediazoöxide, Vol 2, p B58-R

3-Nitro-4-diazophenol. See 3-Nitrobenzene-4-diazo-1-oxide under Mononitrobenzenediazoöxide, Vol 2, p B58-R

Dinitroderivs.

4,6-Dinitro-2-diazophenol. See Dinitrobenzenediazooxide or Diazodinitrophenol, Vol 2, p B 59-L. This compd is also known as Diazol

2,6-Dinitro-4-diazophenol. See 2,6-Dinitrobenzene-4-diazo-1-oxide, Vol 2, p B59-R

2,3,6-Trinitro-4-diazophenol. See 2,3,6-Trinitrobenzene-4-diazo-1-oxide, Vol 2, p B60-L

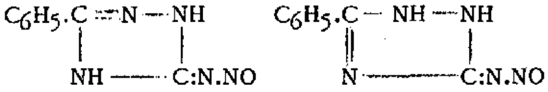
2-Diazophenol-6-diazonium chloride, 4-Nitro Derivative [called 4-Nitro-2-diazo-phenol-diazoniumchlorid-(6) or 5-Nitro-o-chinon-diazid-(1)-diazoniumchlorid-(3) in Ger], $O_2N.C_6H_2ON_2.N({:}N)Cl$, mw 225.58, N 31.05%; obtd from 4-nitro-2,6-diaminophenol by using an excess of HNO_2 & HCl. Its *Chloroplatinate salt,* $2C_6H_2N_5O_3Cl + PtCl_4$, yel plts, is expl

Refs: 1) Beil **16**, 532 2) P. Griess, Ber **19**, 318(1886) & JCS **50** I, 460(1886)

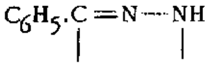
Diazophenol & Hydrazine Reaction Products. Hydrazine reacts with phenol diazonium salts to give products presumed to be the hydrazine salts of a *Tetrazenephenol.* Hydrazine dissolved in water is added to an alc-water suspension of Diazodinitrophenol and stirred for 30 min. The product, brn-red ndls, is the hydrazine salt of 2,4-dinitro-6-(tetrazeno) phenol. The latter salt by reaction with aq KNO_3 forms the *Potassium salt* of 2,4-dinitro-6-(tetrazeno) phenol, dk-red crysts. Both salts decomp with expl violence on heating to 200°. They are claimed to be useful in primer compns (Ref)

Ref: J.F. Kenney, USP 2728760(Dec 1955) & CA **50**, 7462-63(1956)

5(or 3)-Diazo-3(or 5)-phenyl-1,2,4-triazole [called 5(or 3)-Nitrosimino-3(or 5)-phenyl-1,2,4-triazolin or 5(or 3)-Diazo-3(or 5)-phenyl-1,2,4-triazol in Ger],



or



N═C N(N).OH; mw 189.18, N 37.02%; yel crysts, mp - explodes violently when heated in the dry state, or by friction or contact with HI; was prepd by diazotizing 5-amino-3-phenyl-1,2,4-triazole with $NaNO_2$ & HCl in the cold (Refs 1 & 2)

Refs: 1) Beil **26**, (46) 2) W. Manchot, Ber **43**, 1313(1910) & CA **4**, 2479(1910)

Diazopropane, $C_3H_6N_2$; mw 70.09, N 39.97%. Two isomers are known:

1-Diazopropane (called 1-Diazo-propan in Ger), $CH_3.CH_2.CH{:}N{:}N$, liq, bp -8.0 to -7.5° at 41.5mm of press; its ether soln is deep yel-brn in color (Ref 1)

2-Diazopropane (called 2-Diazo-propan or Dimethyldiazomethan in Ger), $(CH_3)_2C{:}N{:}N$, mobile red liq of unpleasant, overpowering odor, bp -31.2° at 14mm press; dec at RT with evolution of heat & loss of N; may deflgr suddenly; reacts violently with acids (Refs 2 & 3)

Other props & methods of prepn are given in Beil
Refs: 1) Beil **1** (334) & {2691} 2) Beil **1** (344) & {2745} 3) H. Staudinger & A. Gaule, Ber **49**, 1905(1916) & CA **11**, 596(1917)

Diazopropanone or Acetyldiazomethane. See Diazoacetone

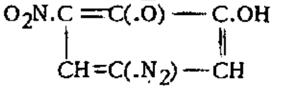
Diazopropene, Diazopropylene or Vinyldiazomethane. (called Diazopropen, Vinyl-diazomethan or Diazopropylen in Ger), CH_2:CH.CH:N:N; mw 68.08, N 41.15%; not isolated in the free state, gives a wine-red eth soln; isomerizes to pyrazole; can be prepd by adding KOH in methanol to a soln of allylnitrosourethane in eth (Refs 1, 2 & 4) and by the method of Adamson & Kenner (Refs 1 & 3)
Refs: 1) Beil **1**, (378) & {2960} 2) S. Nirdlinger & S.F. Acree, AmChemJ **43**, 358(1910) & CA **4**, 1617(1910) 3) D.W. Adamson & J. Kenner, JCS **1935**, 286 & Nature **135**, 833(1935); CA **29**, 2910, 5413(1935) 4) C.D. Hurd & S.C. Lui, JACS **57**, 2656 (1935) & CA **30**, 1736(1936)

5-Diazo-3-isopropyl-asym(1,2,4)-triazole Chloroaurate, $C_5H_7N_5 + AuCl_3$, yel flocculent ppt, stable at RT but exploding violently on heating. It was obtd by treating 3-amino-5-isopropyl-asym-triazole, $C_5H_{10}N_4 + HNO_3$, with a concd soln of chloroauric acid.
Refs: 1) Beil **26**, [81] 2) J. Reilly & P.J. Drumm, JCS **1926**, 1731-2, 1735 & CA **20**, 3293-94(1926)

DIAZOPYROCATECHOL AND DERIVATIVES

Diazopyrocatechol or Diazo-o-dihydroxybenzene, $N_2.C_6H_3(OH)_2$, may be considered the parent compd of the following derivs, although it was not found in the literature and was not used to prep them:

6-Nitro-4-diazopyrocatechol [called 6-Nitro-4-diazobrenzcatechin or 6-Nitro-2-oxy-benzochinon-(1.4)-diazid-(4) in Ger],



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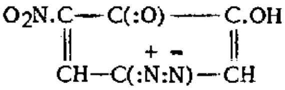
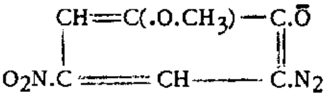
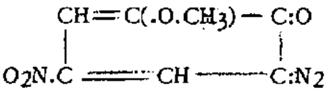


; mw 181.11, N 23.20% golden-orn ndls + ½w, mp - explodes when heated to 159-60°, but not by impact; was prepd by diazotizing the hydrochloride of 6-nitro-4-aminopyrocatechol (Refs 1, 2 & 3).

5-Nitro-3-diazopyrocatechol-1-methyl ether [called 5-Nitro-3-diazo-brenzcatechin-1-methyläther or 5-Nitro-3-methoxy-o-chinon-diazid-(1) in Ger; and called 6-Methoxy-4-nitro-o-guinonediazide in CA Collective Formula Index 14-40]

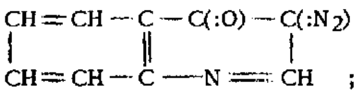


or



; mw 195.13, N 21.54%; orn-colored ndls + ½ H_2O (from dil alc), mp - explodes violently at 169-72°; readily sol in glacial acet ac, chlf & ethyl acet; sol in warm alkalies, giving a blue-red soln; sl sol in acet & benz; was prepd by diazotizing either 5-nitro-3-aminobenzopyrocatechol-1-methyl ether or 2-bromo-5-nitro-3-aminoanisole (Refs 1, 2 & 3)
Refs: 1) Beil **16**, 535 & [291] 2) R. Meldola et al, JCS **69** II, 1332, 1334(1896) 3) G. Heller et al, Ber **56**, 1872, 1875(1923); JCS **124** I, 1092, 1095(1923) & CA **18**, 385(1924)

3-Diazo-4(3H)-quinolone or Quinolone-3,4-dione-3-diazide,

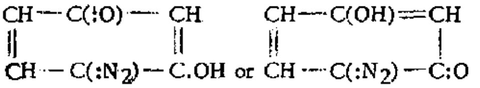


; mw 171.15, N 24.55%; yel lfts (from benz), mp 129-30° (dec); was produced by diazotizing 3-amino-4-hydroxyquinoline-HCl followed by alkalizing with Na_2CO_3 soln (Ref 2). This compd and many of its unstable derivs, prepd by Süs & Glos (Ref 3), were patented as sensitizing agents on lithographic printing plates

Refs: 1) Beil-not found 2) O. Süs et al, Ann **583**, 150(1953) & CA **49**, 1728(1955) 3) O. Süs & M. Glos, USP 2859112(1958) & CA **53**, 9866(1959)

DIAZORESORCINOL AND DERIVATIVES

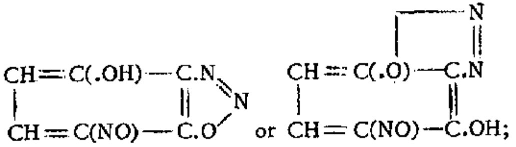
Diazoresorcinol or Diazo-m-dihydroxybenzene,



(called 4-Diazo-3-hydroxy-2,5-cyclohexadiene-1-one in CA 5th Decennial, Formula Index, p 148F); mw 136.11, N 20.58%; no props are reported. Its prepn is given in Refs 2 & 3

Refs: 1) Beil-not found 2) N.N. Vorozhtzov & A.M. Gorkov, ZhObshchKhim **2**, 421(1932) & CA **27**, 969(1933) 3) J. Poskocil & Z.J. Allan, ChemListy **47**, 1801(1953) & CA **48**, 4221(1954)

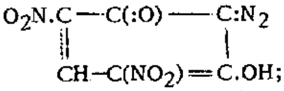
4-Nitroso-2-diazoresorcinol (called 4-Nitroso-2-diazoresorcin in Ger),



mw 165.11, N 24.45%; gray-grn ndls (from acet), mp - begins to decomp at 176° & explodes at 195°; dissolves in alk to form red solns, from which it is pptd unchanged on acidification; was prepd by diazotizing 2-aminoresorcinol-HCl (Refs 1 & 2)

Refs: 1) Beil **16**, 541 2) H. Kauffmann & E. de Pay, Ber **39**, 324(1906) & JCS **90** I, 168(1906)

4,6-Dinitro-2-diazoresorcinol [called 4.6-Dinitro-2-diazoresorcin or 4.6-Dinitro-3-oxy-o-chinon-diazid-(2) in Ger],



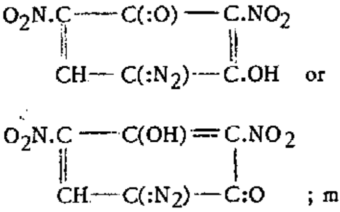
mw 226.11, N 24.78%; yel crysts; obtd from its K salt by treating with concd H_2SO_4

Potassium salt, $KC_6HN_4O_6$, yel ndls (from very concd aq soln)

Potassium salt monohydrate, $KC_6HN_4O_6 + H_2O$, brn prisms with a blue glittering surface

No expl props are reported (Refs 1, 4 & 5)

2,6-Dinitro-4-diazoresorcinol [called 2.6-Dinitro-4-diazoresorcin; 3.5-Dinitro-2-oxy-p-chinon-diazid-(1); or 3.5-Dinitro-4-oxy-o-chinon-diazid-(1) in Ger],



; mw 226.11, N 24.78%; golden-yel plts, mp - very expl; was prepd by dissolving 2,3,6-trinitro-4-diazophenol in boiling Na acetate and redissolving the Na salt in a large quantity of hot w acidified with HCl (Refs 2 & 3)

Refs: 1) Beil **16**, 535 2) Beil **16**, 536 3) R. Meldola & J.G. Hay, JCS **95** II, 1384 (1909) 4) B. Glowiak, BullAcadPolonSci, SerSciChém, Géol et Géograph **8**, 1-4(1960) (in English) & CA **54**, 12029(1960)(IR spectrum) 5) B. Glowiak, RoczChem **34**, 1349-63 (1960) & CA **55**, 19838(1961)(IR spectroscopy data)

6-Nitro-4-diazoresorcinol-3-methyl ether [called 6-Nitro-4-diazo-resorcin-3-methyläther or 5-Nitro-2-methoxy-p-chinon-diazid-(1) in Ger], $C_7H_5N_3O_4$; mw 195.13, N 21.54%; yel ndls or flakes, mp - explodes at 178°; was prepd by diazotizing 4,5-dinitro-2-aminoanisole

Ref: Beil **16**, 536 & (365)

4-Nitro-5-bromo-2-diazoresorcinol-1-methyl ether, $C_7H_4N_3O_4Br$; mw 274.04, N 15.33%; orn-yel crysts, mp 189°, explodes when heated above its mp; was obtd from 4-nitro-3,5-dibromo-anisole-diazonium-2-hydroxide by treating with H_2SO_4 or acetic acid soln

Ref: Beil **16**, (365)

2,5-Dinitro-4-diazoresorcinol-1-methyl ether, $C_7H_4N_4O_6$; mw 240.13, N 23.33%; orn prisms (from acet ac), mp 197-98°, deflgr on rapid heating; was prepd by diazotizing 2,3,5-trinitro-4-aminoanisole

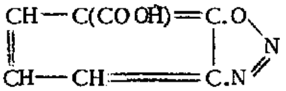
Ref: Beil **16**, (365)

2,6-Dinitro-4-diazoresorcinol-1-methyl ether, $C_7H_4N_4O_6$, mw 240.13, N 23.33%; yel prisms

(from acet ac), mp 191-94° (dec); was prepd by diazotizing 2,3,6-trinitro-4-aminoanisole
Ref: Beil **16**, (366)

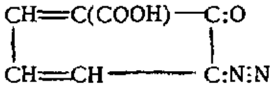
DIAZOSALICYLIC ACID AND DERIVATIVES

3-Diazosalicylic Acid [called 3-Diazo-salicylsäure or o-Chinon-diazid-(1)-carbonsäure-(3) in Ger],



$$\begin{array}{lll} CH-C(COOH)=C.O & & \\ \| \qquad\qquad\quad | & N & \\ CH-CH=C.N & & \end{array}$$

or



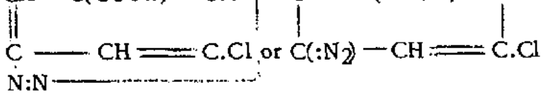
$$\begin{array}{l} CH=C(COOH)-C{:}O \\ | \qquad\qquad\qquad | \\ CH=CH-C{:}N{:}N \end{array}$$

; mw 164.12, N 17.07%; yel ndls (from acet), mp - dec at 155°; was prepd by diazotizing 3-aminosalicylic acid in glacial acetic acid
Refs: 1) Beil **16**, 553 2) O. Zahn, JPrChem **61**, [2], 533 (1900) & JCS **78** I, 549 (1900)

3-Diazo-5-nitro-salicylic Acid [called 5-Nitro-3-diazo-salicylsaure or 5-Nitro-benzoquinon-(1.2)-diazid-(1)-carbonsäure-(3) in Ger], $O_2N.C_6H_2.N_2O(COOH)$; mw 209.12, N 20.10%; yel ndls (from alc), mp - becomes brn at 100° & dec at 145-50°, explodes violently on rapid heating; was prepd by diazotizing 5-nitro-3-aminosalicylic acid
Refs: 1) Beil **16**, (368) 2) R. Meldola et al, JCS **111**, 541 (1917)

5-Diazosalicylic Acid. See 5-Diazo-2-hydroxybenzoic Acid

3-Chloro-5-diazosalicylic Acid [called 3-Chlor-5-diazosalicylsaure or 6-Chlor-benzoquinon-(1.4)-diazid-(4)-carbonsaure-(2) in Ger],



$$\begin{array}{l} CH-C(COOH)=C.O \\ \| \\ C-CH=C.Cl \\ N{:}N \end{array} \quad \text{or} \quad \begin{array}{l} CH=C(COOH)-C{:}O \\ | \qquad\qquad\qquad | \\ C(:N_2)-CH=C.Cl \end{array}$$

mw 166.57, N 16.82%; ochre-yel scales (from acet ac), mp - detonates at 193°; almost insol in w & alc; was prepd by diazotizing 3-chloro-5-aminosalicylic acid
Refs: 1) Beil **16**, (369) 2) R. Meldola et al, JCS **111**, 543 (1917)

Diazostilbene, $C_6H_5.CH{:}CH.C_6H_4N_2$; known in the form of the salts of stilbene diazonium hydroxide, $C_6H_5.CH{:}CH.C_6H_4.N({:}N).OH$. The following salts of Nitrostilbene diazonium hydroxide, $C_6H_5.CH{:}CH.C_6H_3(NO_2).N({:}N).OH$, are either expl or unstable on heating:

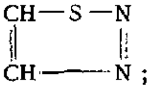
4-Nitrostilbene-2-diazoniumhydroxide (Refs 1 & 2):
Chloride, $O_2N.C_{14}H_{10}.N_2.Cl$, dk-yel ndls, mp - dec at 107°
Sulfate, $O_2N.C_{14}H_{10}.N_2.O.SO_3H$, lt-yel ppt, mp dec 135-57°

2-Nitrostilbene-4-diazoniumhydroxide (Refs 1 & 3):
Chloride, $O_2N.C_{14}H_{10}.N_2.Cl$, dk-violet plts, mp - expl 105-110°
Nitrate, $O_2N.C_{14}H_{10}.N_2.O.NO_2$, lt-brn ndls, mp - explodes violently on heating
Sulfate, $O_2N.C_{14}H_{10}.N_2.O.SO_3H$, lt-brn ndls, mp - darkens at 108° and decomp explosively at 165°

The salts are formed by diazotizing in alcoholic-acid soln the appropriate nitroaminostilbene with amyl nitrite

Refs: 1) Beil **16**, 514 2) F. Sachs & S. Hilpert, Ber **39**, 905 (1906) & JCS **90** I, 242 (1906) 3) P. Pfeiffer & J. Monath, Ber **39**, 1305 (1906) & JCS **90** I, 413 (1906)

Diazosulfide or 1,2,3-Thiadiazole (called 1.2.3-Thiodiazol or "Diazosulfid" in Ger),



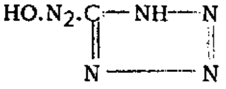
$$\begin{array}{l} CH-S-N \\ \| \qquad\quad \| \\ CH-N \end{array}$$

; mw 86.13, N 32.53%; clear liq, bp 83-85° at 60mm press, bp 151° at 742mm, volatile with steam, d 1.3202 at 0°; mod sol in alc & eth; sol in 3 parts w at 10°; dec on heating in NaOH; was prepd by heating 1,2,3-thiadiazole-4-carboxylic acid to 230-60°

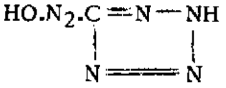
It forms salts, such as:
Chloroaurate, $C_2H_2N_2S + AuCl_3$, pale-yel ndls (from alc), mp - darkens at 152° and decomp at 165° w/o melting
Hydrochloride, $C_3H_2N_2S + HCl$, hygr ndls (from eth + HCl), or prisms (by sublimation), mp 70°
Hydrochloride Chloroaurate, $C_2H_2N_2S + HCl + AuCl_3$, yel ndls, mp - dec 150-60°
Refs: 1) Beil **27**, 562 2) O. Wolff et al, Ann **333**, 11, 18 (1904) & JCS **86** I, 829-30 (1904)

Diazotate. A metal salt and tautomer of Diazonium Hydroxide; it is acid in character and contains the ArN:NO- radical [Hackh's (1944), 267]

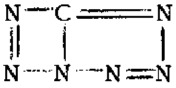
5-Diazotetrazole or Tetrazolyl-5-diazonium Hydroxide [called 5-Diazo-tetrazol or Tetrazol-diazoniumhydroxyd-(5) in Ger],



Formula I



Formula II



Formula III

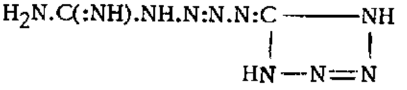
mw 114.07, N 73.68%; crys, extremely sensitive compd in the free state (Formula III); exploding in concd soln even at 0°; forms salts which are more stable but still explosive; it is formed when the Na salt of 5-aminotetrazole is diazotized. Its *Sodium tetrazolyl-isodiazotate salt*, Na_2CON_6, ndls (from concd aq soln + alc), deflgr on heating (Refs 1 & 2)

Rathsburg (Ref 4) patented the use of the expl salts of tetrazole & triazole or their derivs (including 5-Diazotetrazole) in detonators & percussion caps

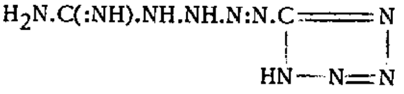
When the above Na salt of Diazotetrazole in a moderately concd soln is boiled for a long time and then cooled, or when CO_2 is passed into the boiling soln, there is formed a *Sodium* deriv of 5-hydroxyazotetrazole: $Na_2C_2N_{10}O + 5H_2O$, yel plts, explodes with great violence. The corresponding *Barium salt*, $BaC_2N_{10}O + 4H_2O$, yel ndls (from w), is also highly expl (Refs 1 & 3)

Refs: 1) Beil **26**, 596 2) J.Thiele, Ann **270**, 59(1892) & JCS **62** II, 1299(1892) 3) J. Thiele & J.T. Marais, Ann **273**, 150(1893) & JCS **64** I, 441(1893) 4) H.Rathsburg, BritP 185555(1921) & CA **17**, 1147(1923) 5) J. Reilly et al, SciProcRoyDublinSoc **24**, 349(1948) & CA **43**, 1770(1949(Prepn)

Diazotetrazole-aminoguanidine or [Guanyl (tetrazolyl-5)]-1, IV-tetrazene {called 1-Guanyl-4-[tetrazolinyliden-(5)]-tetrazen or 1-Guanyl-4[tetrazolyl-(5)]-tetrazen in Ger},



$H_2N.C(:NH).NH.N:N.N:C$—NH, HN—N=N or



$H_2N.C(:NH).NH.NH.N:N.C$=N, HN—N=N ;

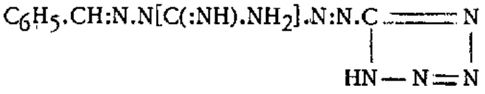
mw 170.15, N 82.33%; yel ndls + H_2O, mp 142° (dec), deflgr in contact with a flame; mod sol in dil HNO_3; diffc sol in w, giving an acid soln; was prepd from 5-diazotetrazole & aminoguanidine. Its use in primers was patented by Rathsburg (Ref 4). It forms some expl salts, such as:

Periodide, $C_2H_6N_{10}$ + HI + I, blk to brn pleochroic, cubic crysts; explodes gently when heated, but violently in contact with HNO_3 or $AgNO_3$

Silver salt, yel crysts, explosive

Refs: 1) Beil **26**, (123) 2) K.A. Hofmann et al, Ber **43**, 1091(1910); JCS **98** I, 446 (1910) 3) K.A. Hofmann & H. Hock, Ber **44**, 2947(1911) & JCS **100** I, 1047-48(1911) 4) H.Rathsburg, BritP 201009(1922) & CA **18**, 472(1924)(One example of a primer compn contains: MF 30, $KClO_3$ 25, Sb_2S_3 30 & Diazotetrazole-aminoguanidine 15%)

Diazotetrazole-benzylideneaminoguanidine {called 4-Benzal-3-guanyl-1-[tetrazolyl-(5)-tetrazen-(1); [Tetrazol-5-diazo]-[β-benzal-α-guanyl-hydrazid]; or N-[Tetrazol-5-diazo] -N-benzalamino-guanidin in Ger},



$C_6H_5.CH:N.N[C(:NH).NH_2].N:N.C$=N, HN—N=N

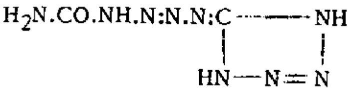
or its tautomer; mw 257.24, N 54.45%; orn-red ndls + H_2O (from alc), mp - dec at 132°; is decompd by acids; was prepd by reaction of 5-diazotetrazole & benzalaminoguanidine in acetic acid soln; forms with concd NaOH a *Sodium salt*, orn-red ndls, which in aq soln is stable on heating to 90° (Refs 1 & 2)

Rathsburg (Ref 3) patented the use of this compd in primer compns & detonators

Refs: 1) Beil **26**, (191) 2) K.A. Hofmann & H. Hock, Ber **44**, 2951(1911) & JCS **100** I, 1048(1911) 3) H. Rathsburg, BritP 201009(1922) & CA **18**, 472(1924); FrP 573107(1924)

Diazotetrazole-phenylhydrazine. See N^1-Amino-N^2-phenyl-N^3-(tetrazolyl-5)-triazene in Vol 1, p A247-R

Diazotetrazole-semicarbazide or (Tetrazolyl-5)-carboxamide-I, IV-tetrazone [called 4-[Tetrazolinyliden-(5)]-tetrazen-carbonsäure-(1)-amid or 4[Tetrazolyl-(5)]-tetrazen-carbonsäure-(1)-amid in Ger],



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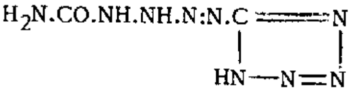


;

mw 171.13, N 73.67%; crysts + H_2O, mp 122°, deflgr when heated at higher temps; diffc sol in w, giving an acid soln; was prepd from 5-Diazotetrazole & semicarbazide or acetone-semicarbazone (Refs 1 & 2)

Refs: 1) Beil **26**, (123) 2) K.A. Hofmann & H. Hock, Ber **44**, 2950-51(1911) & JCS **100** I, 1048(1911)

Diazotization. This is a reaction by which a diazo compd is obtd from a primary amine. The reaction is as follows:

$R\text{-}NH_2 + 2HX + NaNO_2 \rightarrow R\text{-}N_2^+X^- + NaX + 2H_2O$

where X = Cl^-, Br^-, NO_3^-, HSO_4^- & BF_4^-

By means of different reactions, the diazonium group can be replaced by certain elements or groups:

a) the hydroxyl group replaces the diazonium group on heating with dil H_2SO_4

$R.N_2X + H_2O \rightarrow R.OH + HX + N_2$

b) the diazonium group is replaced with halogen when the double salt formed with cuprous halides is heated

$R.\ N_2X.Cu_2Cl_2 = R.X + Cu_2Cl_2 + N_2$

c) a nitrile is formed when a soln of the diazonium salt is heated with KCN, $CuSO_4$ or Cu powder

$R.N_2X + KCN = R.CN + KX + N_2$

d) the diazonium group is replaced with H by treating with aliphatic alcohols in alkaline soln

$R.N_2X + C_2H_5OH = R.H + CH_3CHO + HX + N_2$

e) ethers are sometimes formed when a diazonium salt is heated with alcohol in neutral soln

$R.N_2X + CH_3OH = R.O.CH_3 + HX + N_2$

f) by reduction of a diazonium salt with K bisulfite, hydrazines are obtd

$R.N_2X + 2KHSO_3 + 2H_2O = R.NH.NH_2.HX + 2KHSO_4$

Diazotization is an important reaction because of the practical uses of diazo compds. One use of diazo compds is in the prepn of light-sensitive printing papers, since most diazo compds are stable in the dark but decomp in light. The largest & most varied use of diazotization and diazo compds is for coupling with amino or hydroxy aromatic compds to produce azo dyes. The coupling occurs accdg to the equation:

$R\text{-}N_2^+X^- + HR' \rightarrow R\text{-}N{=}N\text{-}R' + HX$

where R represents an aryl radical and R' represents an alkyl or aryl radical whose conjugate acid HR' is capable of coupling

See also Diazocompounds and Diazonium Salts

Refs: 1) W. Fuchs, Rec **42**, 511-12(1923) & CA **17**, 3489(1923)(The Witt method of diazotization) 2) Groggins (1952) 135-75 (Chapter 3, "Diazotization and Coupling" by H.E. Woodward; not found in later editions) 3) Kirk & Othmer, 2nd ed, **2**(1963), 868-70(Diazotization)

DIAZOTOLUENE AND DERIVATIVES

Diazotoluene, Diazomethylbenzene or Toluenediazonium Hydroxide (called Diazo-toluol; Diazo-methyl-benzol or Toluol-diazoniumhydroxyd in Ger),

$CH_3.C_6H_4.N(:N).OH$; mw 136.15, N 20.58%. The following isomers are known only in the form of their salts:

o-(or 2-) Diazotoluene Salts (Ref 1):

Chloride, $CH_3.C_6H_4.N_2.Cl$; wh crys, hygr, mp-deflgr on heating; sol in acet, chlf & AcOH; was prepd by diazotizing o-toluidinehydrochloride

Lead Tetrachloride, $2CH_3.C_6H_4.N_2Cl + PbCl_4$; yel crysts, mp - explodes at 78-80°; stable if kept dry and in absence of air; was prepd by treating the chloride salt with lead tetrachloride

Nitrate, $CH_3.C_6H_4.N_2.O.NO_2$; wh ndls (from alc + eth); mp - highly expl in dry state; v sol in alc & MeOH; sol in acet ac; insol in chlf & benz; was prepd by diazotizing o-toluidine with amyl nitrite in the presence of HNO_3

Perchlorate, $CH_3.C_6H_4.N_2.ClO_4$;ndls; extremely sensitive to shock, can be exploded by touching even when wet with ether; v sl sol in w; was prepd by diazotizing o-toluidine-hydrochloride with $NaNO_2$ in the presence of perchloric acid

Sulfate, $CH_3.C_6H_4.N_2.O.SO_3H$; wh ndls, hygr, mp - deflgr on heating & explodes mildly on impact; readily sol in acet ac; sol in MeOH; sl sol in alc; insol in eth, chlf, CS_2, petr eth, acet, benz & nitrobenz; was prepd by passing N oxides thru a soln of o-toluidine in alc to which H_2SO_4 was added dropwise

m-(or 3-) Diazonitrotoluene Salts (Ref 2):

Borofluoride, $CH_3.C_6H_4.N_2.BF_4$;crysts, dec at 108°

Chloride + Bismuth Trichloride, $CH_3.C_6H_4.N_2.Cl + BiCl_3$; yel plts, mp 120° (dec); diffc sol in dil HCl

Nitrate, $CH_3.C_6H_4.N_2.O.NO_2$; wh ndls, very expl

Sulfate, $CH_3.C_6H_4.N_2.O.SO_3H$; wh plts, becomes red on exposure to light, very unstable

p-(or 4-) Diazonitrotoluene Salts (Ref 3):

Lead Tetrachloride, $2CH_3.C_6H_4.N_2.Cl + PbCl_4$; yel crysts, explodes at 72-75°; insol in w & org solvs

Nitrate, $CH_3.C_6H_4.N_2.O.NO_2$; ndls, expl; sol in MeOH, alc, acet ac & acet; sl sol in chlf

Perchlorate, $CH_3.C_6H_4.N_2.ClO_4$; ndls (from warm dil alc) or lfts, explodes mildly on heating, less sensitive to impact than the ortho salt; sol in warm acet acid & warm dil alc & w; almost insol in abs alc; insol in eth, chlf, CS_2, acet, benz & nitrobenz. This compd was proposed by von Herz as a chge for use in detonating caps (Ref 3a)

Tetrachloroiodide, $CH_3.C_6H_4.N_2.ICl_4$; yel crysts (from iodotrichloride in concd HCl), mp 95° (dec)

See also the following Refs for addnl info on Diazotoluenes

Refs: 1) Beil **16**, 495, (358) & [278] 2) Beil **16**, 500 & [279] 3) Beil **16**, 501, (359) & [280] 3a) E. von Herz, USP 1054411(1913) & CA **7**, 1419(1913) 4) H.R. Lee, JIndEngChem **13**, 104950(1921) & CA **16**, 38(1922)(Decomp of p-diazotoluene chloride in acid soln and its use in coupling reactions) 5) A. Algerino, Gazz **62**, 1145-50(1932) & CA **27**, 2942(1933)(Diazonium dichloroiodides of Diazotoluene) 6) D. Bigiavi & C. Albanese, Gazz **65**, 773-88(1935) & CA **30**, 2934-35(1936) (Reduction of Diazotoluenes in relation to azoxy derivs) 7) I.V. Grachev & N.A. Kirzner, ZhObshchKhim **18**, 1525-36(1948) & CA **43**, 2491-92(1949)(Acid & base props of Diazotoluenes) 8) E.S. Lewis, ChemBer **91**, 2350-58(1958) & CA **53**, 6123(1959) (Spectroscopic studies)

Mononitrodiazotoluenes, $CH_3.C_6H_3(NO_2).N_2.OH$; mw 181.15, N 23.20%; known in the form of their salts:

5-Nitro-2-diazotoluene Salts (Ref 1):

Nitrate, $CH_3.C_6H_3(NO_2).N_2.O.NO_2$; straw-yel ndls, deflgr at 97°; was prepd by diazotizing 5-nitro-2-aminotoluene with amyl nitrite in abs alc in the cold

5-Nitro-2-isodiazotoluene snd Its Salts (Refs 1 & 3): yellowish crysts, deflgr at 40°

Potassium, $KC_7H_6N_3O_3 + H_2O$; yel ndls (from MeOH), mp-becomes lt yel at 150°, and dec at 202°; sol in alc & w with a red color; attempts to prep a large amount of the parent compd & its K salt resulted in a violent expln of the material in a desiccator

Sodium, $NaC_7H_6N_3O_3$; yel crysts (from MeOH), very expl

3-Nitro-4-isodiazotoluene (Ref 2): lt-yel ndls, mp - dec on standing & turning brn

Sodium salt, $NaC_7H_6N_3O_3$; lt-yel ndls (from alc + eth), deflgr on heating above its mp; v sl sol in alc & w

Refs: 1) Beil **16**, 499, (359) & [279] 2) Beil **16**, 506 & (360) 3) J. Meisenheimer & E. Hesse, Ber **58**, 1175-77(1919); JCS **116**, 391(1919) & CA **14**, 53(1920) 4) I.V. Grachev & N.A. Kirzner, ZhObshchKhim **18**, 1525-36 (1948) & CA **43**, 2491-92(1949)(Acid & base props)

Dinitrodiazotoluenes, $CH_3.C_6H_2(NO_2)_2.N_2.OH$; mw 226.15, N 24.78%.

3,5-Dinitro-2-diazotoluene, yel amor powd, no props are reported; was obtd by diazotizing 3,5-dinitro-2-aminotoluene with $NaNO_2$ & concd H_2SO_4, or by adding $K_2S_2O_5$ & fuming HNO_3 to the amine at -5 to 0° (Refs 1 & 3)

3,5-Dinitro-2-diazotribromotoluene, $CH_3.C_6H_2(NO_2)_2.N_2.Br_3$; yel-red crysts, mp-deflgr at 64° (Ref 1)

2,3-Dinitro-4-diazotoluene, no props of this compd are reported (Refs 2 & 4)

Refs: 1) Beil **16**, 499 & [279] 2) Beil **16**, [281] 3) G.T. Morgan & H.D.K. Dree, JCS **111**, 791 (1920) 4) L.A. Elson et al, JCS **1929**, 2741

NOTE: No higher nitrated derivs of Diazotoluene were found in Beil or in CA thru 1961

DIAZOTOLUENESULFONIC ACID AND DERIVATIVES

Diazotoluene sulfonic Acid [called Diazotoluol-sulfonsaure in Ger], $CH_3.C_6H_3(N_2O).SO_2$; mw 198.21, N 14.14%. The following isomers are known:

4-Diazotoluene-2-sulfonic Acid, yel or brn ndls, mp - darkens at 126° & dec at 135°; explodes when heated on a Pt block or by impact; insol in alc; was prepd by introducing nitrous gas into an alc suspension of 4-aminotoluene-2-sulfonic acid (Ref, p 566)

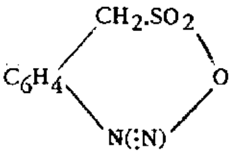
5-Diazotoluene-2-sulfonic Acid, listed in Ref, p 567, but no props are given

4-Diazotoluene-3-sulfonic Acid, plts, mp - not reported; sol in w at 60° w/o decompn; diffc sol in cold w; prepd by reaction of 4-aminotoluene-3-sulfonic acid in alc or w at 30-40° & HNO_2 (Ref, p 566)

6-Diazotoluene-3-sulfonic Acid, wh nds, mp - not reported; sol in hot w; sl sol in cold w; insol in alc; prepd from 6-aminotoluene-3-sulfonic acid & nitrous gas in a saturated aq soln (Ref, p 568)

2-Diazotoluene-4-sulfonic Acid, prisms, burns brightly when heated on a Pt foil & explodes by impact; insol in cold w; was prepd by introducting nitrous gas in an aq alc soln of 2-aminotoluene-4-sulfonic acid (Ref, p 569)

4-Diazotoluene-1'-sulfonic Acid,



, prisms, mp - deflgr when heated on a Pt foil; readily sol in w; insol in alcohol; prepd by reaction of nitrous gas & 4-aminotoluene-1'-sulfonic acid in water (Ref, 570)

Ref: Beil **16**, 566-70

Mononitrodiazotoluene-sulfonic Acid, $CH_3.-C_6H_2(NO_2).(N_2O).SO_2$; mw 243.21, N 17.28%:

6-Nitro-4-diazotoluene-2-sulfonic Acid, dk-red crysts, mp - deflgr when heated; mod sol in w; sl sol in alc; prepd by treating 4-amino-2-sulfonic acid with cold fuming HNO_3 (Ref 1, p 567). Batik (Ref 2) found that this compd was formed on acidification of the waste waters from the purification of TNT with Na_2SO_3 soln (Sellite Method). He reported that the substance explodes easily on heating as low as 75°. Investigations by Dr B.T. Fedoroff at Keystone Ord Works conducted during WW II showed that this compd is very sensitive to impact & friction

2-Nitro-4-diazotoluene-3-sulfonic Acid, ndls, mp - explodes violently on heating or by impact; was prepd from 4-aminotoluene-3-sulfonic acid & fuming HNO_3 (Ref 1, p 568)

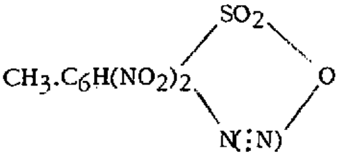
6-Nitro-4-diazotoluene-3-sulfonic Acid, brn prisms, mp - explodes by heating, but not by impact; insol in w & alc; was obtd by reaction of K salt of 6-nitro-4-aminotoluene-3-sulfonic acid with KNO_2 & dil H_2SO_4 (Ref 1, p 568)

3-(or 5) Nitro-2-diazotoluene-4-sulfonic Acid, crysts, mp - deflgr by heating on a Pt block & explodes on impact; almost insol in cold w; obtd when dehydrated 2-aminotoluene-4-sulfonic acid was treated with cold fuming HNO_3 (Ref 1, p 569)

Refs: 1) Beil **16**, 567-69 2) B. Batik, Chim & Ind, Special No, 960-63 (1933) & CA **28**, 645 (1934)

Dinitrodiazotoluene-sulfonic Acid, $CH_3.C_6H(NO_2)_2.N_2O.SO_2$; mw 288.21, N 19.45%

eso-Dinitro-4-Diazotoluene-2-sulfonic Acid,



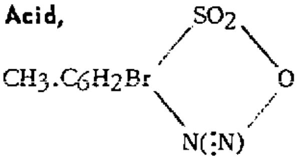
; yel ndls, decomp in light, explodes violently on heating; obtd with the 6-nitro deriv when 4-aminotoluene 2-sulfonic acid is treated with cold fuming HNO_3 (Ref, p 567)

3,5-Dinitro-2-diazotoluene-4-sulfonic Acid, col crysts, turning brn in the light, deflgr on heating; prepd by nitrating 2-aminotoluene-4-sulfonic acid with HNO_3 at -5° (Ref, p 569

Ref: Beil **16**, 567, 569

Diazotoluenesulfonic Acid Halogenated Derivatives

4-Bromo-3-(or 5, or 6)-diazotoluene-2-sulfonic Acid,



; crysts, explodes on impact; obtd by diazotizing an alc suspension of 4-bromo-3(or 5, or 6)-aminotoluene-2-sulfonic acid (Ref, p 567)

5-Bromo-4-diazotoluene-3-sulfonic Acid, $C_7H_5N_2O_3BrS$; brn-red powd, expl on heating or on impact; sol in cold w with slow decompn; dec rapidly in hot w; prepd by action of nitrous gas on a concd aq or alc soln of 5-bromo-4-aminotoluene-3-sulfonic acid (Ref, p 568)

3,5-Dibromo-2-diazotoluene-4-sulfonic Acid, $C_7H_4N_2O_3Br_2S$; ndls, deflgr violently on heating; almost insol in w & alc; prepd by passing nitrous gas in an aq suspension of 3,5-dibromo-2-aminotoluene-4-sulfonic acid (Ref, p 569)

Ref: Beil **16**, 567-69; no later refs found

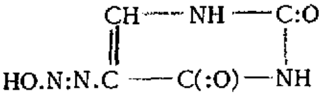
5-Diazo-1,2,4-triazole-3-carboxylic Acid. Same as 5-Nitrosamino-α-symtriazole-3-carboxylic Acid, Vol 1, p A270-L. This compd was patented as a constituent of priming mixts: Diazotriazolecarboxylic acid 1-10, L St 30-60% and remaining ingredients Sb_2S_3, PbO_2, $Ba(NO_3)_2$ & CaSi (Ref 1). See also Refs 2 & 3

Refs: 1) DWM, FrP 824130 (1 Feb 1938) & CA **32**, 5630(1938) 2) Blatt, OSRD **2014** (1944)(Listed under Triazoles) 3) H. Ficheroulle & A. Kovache, MP **41**, 8-9(1959) (Called Acide diazotriazol carbonique in French)

Diazotrinitrophenol. See Trinitrobenzenediazoöxide in Vol 2, p B60-L

Diazouracil and Derivatives

5-Diazouracil; 5-Diazo-2,4-pyrimidinediol or 5-Diazo-4-hydroxy-2(1H)-pyrimidinone (called 5-Diazo-uracil; 5-Diazo-2.4-dioxo-tetrahydropyrimidin or 5-Diazo-2.4-dioxy-pyrimidin in Ger),



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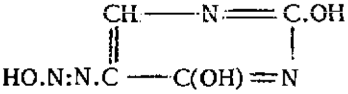
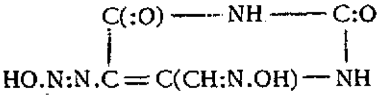


; mw 156.10, N 35.89%; exists in two forms: ruby-red plts (from w), or yel crysts (from w), mp - explodes when heated on a Pt foil. The red form is obtd from 5-aminouracil & $NaNO_2$ in dil HCl under cooling, or from 5-diazouracil ethyl ether by hydrolyzing with cold 1% KOH and carefully adding HCl to the K salt. The yel form is obtd by heating 5-diazouracil ethyl ether with water or by long standing of 5-diazouracilcarboxylic acid at RT

The *Potassium salt,* $KC_4H_3N_4O_3$, yel-red ndls, explodes when heated on a Pt foil

Refs: 1) Beil **25**, 565 2) R. Behrend & P. Ernert Ann **258**, 357(1890) & JCS **58** II, 1241(1890) 3) A. Angeli, Gaz **24** II, 368 (1894) & JCS **68** I, 328(1895)

5-Diazo-4-isonitrosomethyl-uracil [called 5-Diazo-4-isonitrosomethyl-uracil; 5-Diazo-2.6-dioxo-4-iminomethyl-tetrahydropyrimidin or 5-Diazo-2.6-dioxypyrimidin-aldoxim-(4) in Ger.],



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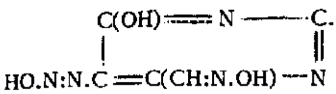


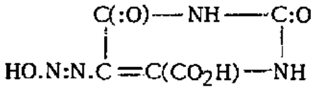
$$\begin{array}{l} C(OH)=N-C.OH \\ \;\;| \qquad\qquad\quad \| \\ HO.N:N.C=C(CH:N.OH)-N \end{array}$$

; mw 199.13, N 35.17%; ndls (from alc) or prisms + 1½ H_2O (from w), mp - explodes on heating; readily sol in alkalies & concd H_2SO_4; sl sol in alc; insol in dil acids; was prepd by treating 5-amino-4-methyluracil in acid soln with $NaNO_2$, or from 5-bromo-4-methyluracil by heating with NH_3 in a tube at 150° and reacting the products with $NaNO_2$ in dil acid soln

Its *Nitro deriv,* $C_5H_4N_6O_6$, ndls, mp - explodes at 100°; dec on heating in w; was obtd by dissolving 5-diazo-4-isonitrosomethyluracil in red fuming HNO_3

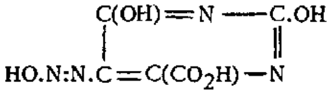
Refs: Beil **25**, 566 2) R. Behrend, Ann **245**, 213-223(1888) & JCS **54** II, 809-10(1888)

5-Diazouracil-4-carboxylic Acid [called 5-Diazo-uracil carbonsäure-(4); 5-Diazo-2.6-dioxo-tetrahydropyrimidin-carbonsäure-(4) or 5-Diazo-2.6-dioxy-pyrimidin-carbonsäure-(4) in Ger.],



$$\begin{array}{l} C(:O)-NH-C:O \\ \;\;| \qquad\qquad\quad | \\ HO.N:N.C=C(CO_2H)-NH \end{array}$$

or

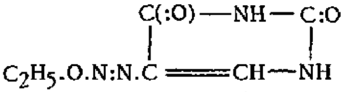


$$\begin{array}{l} C(OH)=N-C.OH \\ \;\;| \qquad\qquad\quad \| \\ HO.N:N.C=C(CO_2H)-N \end{array}$$

; mw 200.11, N 28.00%; unstable yel ndls, mp - turns brn at 70-80° & explodes at 130°; sl sol in w with an acid reaction; readily sol in alkalies; was prepd by reaction of 5-aminouracilcarboxylic acid with cold $NaNO_2$ & HCl. On standing for a long time at RT, the compd is converted into yel Diazouracil

Refs: 1) Beil **25**, 566 2) R. Behrend & P. Emert, Ann **258**, 349(1890) & JCS **58** II, 1240(1890)

5-Diazouracil Ethyl Ether (called 5-Diazo-uracil-äthyl-äther in Ger),



$$\begin{array}{l} \qquad\quad C(:O)-NH-C:O \\ \qquad\quad\;\;| \qquad\qquad\quad | \\ C_2H_5.O.N:N.C=CH-NH \end{array}$$

or its tautometric form; mw 184.16, N 30.43%; lt-yel lfts (from alc), mp - explodes by heating on a Pt foil; dec on heating with alc at 80-100°; almost insol in cold alc, eth & cold w; is formed when 5-diazouracilcarboxylic acid is boiled with alc

*Refs:*1) Beil **25**, 565 2) R. Behrend & P. Emert, Ann **258**, 354(1890) & JCS **58** II, 1240-41(1890)

Diazoxylene and Derivatives

Diazoxylene or Diazodimethylbenzene (called Diazo-xylol, Diazo-dimethyl-benzol or Xylol-diazonium-hydroxyd in Ger), $(CH_3)_2.C_6H_3.N_2.OH$; mw 150.18, N 18.65%. Known in the form of salts of its isomers:

4-Diazo-m-xylene Salts {Ref , pp 507, (360) & [281]}:

Borofluoride, $C_8H_9.N_2.BF_4$; dec at 108°

Dichloroiodide, $C_8H_9.N_2.Cl_2I$; lt-yel, fine cryst powd, mp 79° (dec)

2-Diazo-m-xylene Salts {Ref, p [281]}:

Chloride, $C_8H_9.N_2.Cl$; crysts (from alc + eth)

2-Diazo-p-xylene Salts (Ref, p 507)

5-Nitro-4-isodiazo-m-xylene Salts {Ref,p[281]}:

Potassium, $(CH_3)_2.C_6H_2(NO_2).N_2.K$; lt-yel plts, deflgr on heating rapidly; very sl sol in w

3,5-Dinitro-2-diazo-p-xylene Salts [Ref, p (361)]:

Tribromide, $(CH_3)_2.C_6H(NO_2)_2.N_2.Br_3$; red crysts, dec on storage, especially in sunlight

3,5-Dichloro-2-diazo-p-xylene Salts (Ref, p 508):

Tribromide, $C_8H_7Cl_2.N_2.Br_3$; lt-yel ndls (from MeOH), mp - dec at 155°

Chloroplatinate, $2C_8H_7Cl_2.N_2.Cl + PCl_4$; yel-brn plts, mp - dec at 159°

Ref: Beil **16**, 507, 508, (360, 361) & [281]

NOTE: Higher nitrated derivs were not found in Beil or in CA thru 1961

Diazoxylenesulfonic Acid and Derivatives

Diazoxylenesulfonic Acid (called Diazo-Xylol-sulfonsäure in Ger), $(CH_3)_2.C_6H_2.N_2.O.SO_2$; mw 212.16, N 13.21%. The following isomers are found in Beil:

6-Diazo-m-xylene-4-sulfonic Acid, prisms (from alc) or flesh-colored plts, deflgr on heating; mod sol in w; diffc sol in abs alc

4-Diazo-m-xylene-5-sulfonic Acid, prisms (from w), deflgr on heating; v sl sol in cold w, alc & eth

5-Diazo-p-xylene-2-sulfonic Acid, yel-wh plts, stable at RT, deflgr on heating; dec in w at 60-70°

Other props & methods of prepn are given in Beil

Nitro Derivatives of Diazoxylenesulfonic Acid

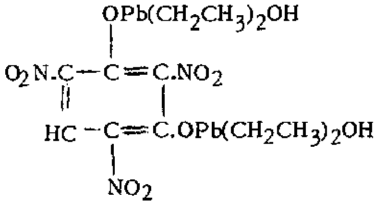
2-Nitro-6-diazo-m-xylene-4-sulfonic Acid, $(CH_3)_2.C_6H(NO_2).N_2.O.SO_2$; mw 257.16, N 16.34%; col ndls, turning brn in the light, deflgr on heating or on impact; sol in HNO_3; almost insol in w; was obtd by nitrating 6-amino-m-xylene-4-sulfonic acid with cold HNO_3

2-(or 5) Nitro-6-diazo-m-xylene-4-sulfonic Acid, col quadratic tablets, explodes when heated on a Pt block; diffc sol in cold w; obtd by nitrating 6-amino-m-xylene-4-sulfonic acid

6-Nitro-4-diazo-m-xylene-5-sulfonic Acid, reddish-colored ndls (from w at 60°), deflgr on heating or on impact; v sl sol in cold w; insol in alc & eth; was prepd by diazotizing 6-nitro-4-amino-m-xylene-5-sulfonic acid

Ref: Beil **16**, 570-71; no later refs found

Dibasic Diethyl Lead Styphnate or Bis-basic Diethyl Lead Styphnate,



; mw 807.76, Pb(tetravalent) 51.2%, yel crysts, mp - explodes at 229°. Can be prepd by treating diethyl lead dichloride in w at RT with equivalent amt of di-ammonium styphnate. Was patented by Burrows et al (Ref 2) as a component of ignition compns in electric detonators. Requires firing current of 0.36 amp in cemented charge

Refs: 1) Beil-not found 2) L.A. Burrows et al, USP 2105635(1935) pp 2-3 (Other Burrows compns are listed in Vol 2, pp B362-63)

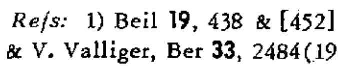
Dibenzalazobisaminoformamidine [called 1,1'-Azobis(benzylideneaminoformamidine) in CA 5th Decennial Index], $H_5C_6.CH:N.NH.C(:NH).N:$-$N.C(:NH).NH.N:CH.C_6H_5$; mw 320.35, N 39.48%; purplish-red flat ndls, mp 176-77° (dec); was obtd when a hot, saturated alc soln of dibenzal-1,6-*diaminobiguanidine* was cooled slowly while exposed to air (Ref 2)

Refs: 1) Beil-not found 2) R.A. Henry et al, JACS **75**, 959(1953) & CA **48**, 2051(1954)

Dibenzal-1,6-diaminobiguanidine [called 1,6-Bis(benzylidene-amino)-biguanidine in CA 5th Decennial Index], $H_5C_6.CH:N.NH.C(:NH).NH.$—$NH.C(:NH).NH.N:CH.C_6H_5$; mw 322.37, N 34.76%; wh, felted ndls (from benz + abs alc), mp 212° (decomp); was prepd from 1,6-dinitrobiguanidine as starting material. Its *Monopicrate salt*, $C_{22}H_{20}N_{10}O_8.3H_2O$, crysts (from 95% alc), mp - dec at 214-15°; and *Picrolonate salt*, $C_{26}H_{25}N_{11}O_8$, crysts, mp - dec at 248-49°; were both prepd by Henry et al (Ref 2)

Refs: 1) Beil-not found 2) R.A. Henry et al, JACS **75**, 959(1953) & CA **48**, 2051(1954)

Dibenzal Diperoxide or Dibenzylidene Diperoxide (called 3.6-Diphenyl-1.2.4.5-tetroxan, Dibenzylidendiperoxyd or Dibenzaldiperoxyd in Ger),



; mw 244.24, O 26.20%; prisms or ndls, mp 191° & 202° (dec); sol in eth; sl sol in warm chlf & acet ac; diffc sol in alc & petr eth; can be prepd by treating benzaldehyde with H_2O_2 & concd H_2SO_4 or ozonizing β,γ-dioxy-Δ-phenylvaleronitrile

Refs: 1) Beil **19**, 438 & [452] 2) A. Baeyer & V. Valliger, Ber **33**, 2484(1900) 3) F.I. Berezovskaya & O.N. Semikhatova, ZhFizKhim **7**, 939-58(1936) & CA **31**,6957(1937)(Decompn & catalysis of org peroxides) 4) Tobolsky & Mesrobian (1954), 172 5) N.A. Milas et al, JACS **77**, 2537(1955) & CA **50**, 5512(1956) 6) Hawkins (1961), 259 & 294

Dibenzalhydrazine. See Benzaldehydeazine, Vol 2, p B35-R

Di(benzeneazo)-azobenzene. See Bis(benzeneazo)-azobenzene in Vol 2 of Encycl, p B134-L

Di(benzeneazo)-azoxybenzene. See Bis(benzeneazo)-azoxybenzene in Vol 2, p B134-L

Di(benzeneazoxy)-azoxybenzene. See Bis (benzeneazoxy)-azoxybenzene in Vol 2, p B134-R

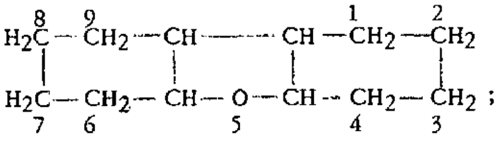
Di(benzenediazo)-glycocoll or Di(phenylazo)-glycine. See Bis(phenylazo)-glycine in Vol 2, p B153-L

Dibenzhydrazidazid. Ger name for compd described as Bis(α-azidobenzylidene)-azine in Vol 2, p B133-L

Dibenzoat des Peroxyd XX. The name given by Criegee to $9,9^1$-Bis(benzoylperoxyfluorenyl)-peroxide. See Vol 2 of Encycl, p B136-L

Dibenzofuran and Derivatives

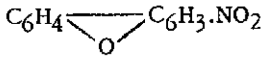
Dibenzofuran, Diphenylene Oxide or Biphenylene Oxide (CA 5th Decennial Subject Index, p 4106s) (called Diphenylenoxyd or Dibenzofuran in Ger),



mw 168.18, O 9.51%; wh lfts or scales (from alc), mp 82-87°, bp 288°; d 1.0866 at 99.3°; n_D 1.6079 at 99.3°; very sol in eth, benz & acet ac; sl sol in alc; insol in w. Other props & methods of prepn are given in Beil

Ref: Beil **17**, 70-71, (30) & [67-68]

Mononitrodibenzofuran,



$C_6H_4\underset{O}{<>}C_6H_3.NO_2$; mw 213.18, N 6.57%. Several isomers are described in the literature:

2-Nitrodibenzofuran, yel ndls (from acetic acid), mp 181-82° & 186-186.5°; diffc sol in hot alc (Ref 1)

3-Nitrobenzofuran, yel ndls (from alc or glac acet ac), mp 151-52° & 158.5-159.5°; mod sol in glac acet acid; sl sol in alc (Ref 1)

Yamasiro (Ref 2) also prepd the 1 & 4-mononitro derivs

Refs: 1) Beil **17**, 72 & [69] 2) S. Yamasiro, BullChemSocJapan **16**, 61(1941) & CA **35**, 5111 (1941)

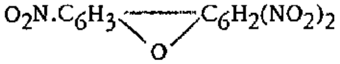
2,7-Dinitrobenzofuran (called 2,6-Dinitrodiphenylenoxyd in Ger),

$O_2N.C_6H_3\underset{O}{<>}C_6H_3.NO_2$; mw 258.18; N 10.85%; ndls (from glac acet ac), mp 245° & 255-56°; sol in acet; sl sol in alc, glac acet ac & xylene; almost insol in petr eth; insol in benz; obtd by contact of dibenzofuran & nitrogen dioxide at RT (Ref 1)

Yamasiro (Ref 2) also prepd the 1,8-; 2,8-; 3,8-; 4,8-; 4,6-; and 3,7-dinitro derivs

Refs: 1) Beil **17**, (30) & [69] S. Yamasiro, BullChemSocJapan **16**, 61(1941) & CA **35**, 5111(1941)

Trinitrodibenzofuran,



$O_2N.C_6H_3\underset{O}{<>}C_6H_2(NO_2)_2$; mw 303.18, N 13.86%. Several isomers are known:

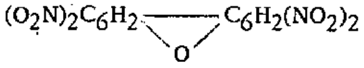
2,3,7-Trinitrodibenzofuran, (called 2.3.6-Trinitro-diphenylenoxyd in Ger), prisms (from benz), mp 223-228° and when pure 236-37°; sl sol in alc, benz & glac acet ac; almost insol in petr eth; obtd with other nitroderivs by nitrating 2-nitrodibenzofuran with fuming HNO_3 (Refs 2 & 4)

2,7,X-Trinitrobenzofuran, (called 2.6.X-Trinitro-diphenylenoxyd in Ger), yel ndls, mp 142-43°; prepd from 2,6-dinitrodibenzofuran by heating with fuming HNO_3 (Refs 1 & 3)

Other props are given in the Refs. Yamasiro (Ref 5) also prepd 2,4,8-; 2,4,7-; 2,4,6-; 2,3,8-; and 1,3,8-trinitro derivs

Refs: 1) Beil **17**, (30) 2) Beil **17**, [69] 3) A. Mailhe, CR **154**, 1516(1912) & CA **6**, 2240(1912) 4) H. Raudnitz, Ber **60B**, 740, 742(1927) & CA **21**, 1981(1927) 5) S. Yamasiro, BullChemSocJapan **16**, 61 (1941) & CA **35**, 5111(1941)

2,3,7,8-Tetranitrodibenzofuran (called 2,3.6,7-Tetranitro-diphenylenoxyd in Ger),



$(O_2N)_2C_6H_2-O-C_6H_2(NO_2)_2$

; mw 348.18, N 16.09%; plts (from xylene), mp 283° & when pure 285-86°; sol in hot glac acet ac, xylene & acet; sl sol in alc; diffc sol in benz; prepd by nitrating 2,6-dinitrodibenzofuran with nitrosyl hydrogen sulfate (Refs 2,4,6 & 7)

1,3,7,9-Tetranitrodibenzofuran (called 1.3.6.8-Tetranitro-diphenylenoxyd in Ger), dk-brn ndls (from acet + alc), mp 252.5° & 255°; prepd by heating 3,5,3',5'-tetranitro-2,2'-dihydroxybiphenyl with p-toluenesulfonylchloride & dimethylaniline on a w bath (Refs 1,3,5,6 & 7)

The expl props of the tetranitro derivs were not reported. Yamasiro (Refs 6 & 7) also prepd 2,3,5,7-; 2,4,6,8-; and 1,3,7,8-tetranitro derivs

Refs: 1) Beil **17**, (30) 2) Beil **17**, [70] 3) W. Borsche & B.G.B. Scholten, Ber **50**, 606(1917) & CA **11**, 2901(1917) 4) H. Ryan & N. Cullinane, SciProcDublinSoc **17**, 321-26 (1924) & CA **18**, 1655(1924) 5) J. van Alphen, Rec **51**, 179-84(1932) & CA **26**, 1273(1932); Rec **51**, 715-25 (1932) & CA **26**, 5554(1932) 6) S. Yamasiro, JChemSocJapan **59**, 945-53 (1938) & CA **33**, 603(1939) 7) S. Yamasiro, BullChemSocJapan **16**, 6-15 (1941) & CA **35**, 3640(1941) 8) F.H. Case & R.V. Schock,Jr, JACS **65**, 20(1943) & CA **38**, 89(1944)(Report a tetranitro deriv as 3,3',5,5'-Tetranitrobiphenylene Oxide, mp 249-50°) 9) H. Zahn & H. Zuber, ChemBer **86**, 172-80 (1953) & CA **48**, 1297(1954)(Report a tetranitro deriv as 3,3',5,5'-Tetranitrodiphenyl Oxide, mp 252°; appears to be the same compd as the 1,3,7,9-Tetranitro deriv)

NOTE: Higher nitrated derivs of Dibenzofuran were not found in Beil or in CA thru 1961

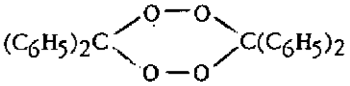
Dibenzolsulfonyl Peroxide. See Benzenesulfonyl Peroxide in Vol 2, p B62-R

1,1'-Di(benzoperoxy)-cyclohexane. See 1,1'-Bis(benzoylperoxycyclohexane in Vol 2 of Encycl, p B135-L

9,9'-Di(benzoperoxy)-fluorene. See 9,9'-Bis(benzoylperoxyfluorene in Vol 2, p B135-R

9,9'-Di(benzoperoxy) 9,9'-fluorene-peroxide. See 9,9'-Bis(benzoylperoxyfluorenyl)-peroxide in Vol 2, p B136-L

Dibenzophenoneperoxide Dimer,



$(C_6H_5)_2C<(O-O)_2>C(C_6H_5)_2$

; mw 396.42, O 16.14%; crysts (from acet, benz or ethyl acetate), mp 206.5-07.5° & 214.5-15.5°, depending on cryst size, rate of heating & solv used; fairly sol in hot benz & CCl_4; diffc sol in eth, acet, eth acet & acet ac; insol in alc; was obtd by ozonolysis of 1,1-diphenyl-1-alkenes in CCl_4 and hydrolysis of the ozonide

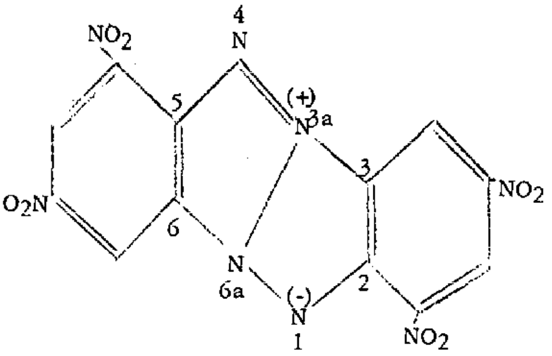
No expln of this compd occurred when heated to its mp, but when heated for 5 mins at 214-15°, it decompd completely to benzophenone

Refs: 1) Beil-not found 2) C.S. Marvel & V.E. Nichols, JACS **60**, 1455-57(1938) & CA **32**, 5815(1938); JOC **6**, 299, 302(1941) 3) S. Kambara et al, JSocRubberIndJapan **21**, 160(1948) & CA **43**, 5622(1949)(Reports same compd (mp 70°) obtd by oxidation of thiobenzophenone by H_2O_2 & concd H_2SO_4); JChemSocJapan (Pure Chem Sect) **70**, 31(1949) & CA **45**, 2916(1951); ChemHighPolymers **5**, 201 (1948) & CA **46**, 1795(1951)(Compd appears to be Dithiobenzophenoneperoxide, mp 70°) 4) Tobolsky & Mesrobian (1954), 172 5) N.A. Miles et al, JACS **77**, 2540-41(1955) & CA **50**, 5512(1956) [Obtd product as crysts (from benz + ethyl acetate 50/50), mp 206-08°]

Dibenzotetrazapentalene and Derivatives

Dibenzotetrazapentalene, $C_{12}H_8N_4$, may be considered as the parent compd of its tetranitro deriv:

Tetranitrodibenzo-1,3a,4,6a-tetrazapentalene (TACOT)



mw 388.21, N 28.86%; oxygen bal to CO_2 -74.2, to CO -24.7; orn to red crysts (from HNO_3), mp 378° (dec), d 1.84, Q_{form} 128 kcal/mol, Q_{comb} 3575 cal/g; sol in nitric acid; sl sol in nitrobenz or dimethyl formamide; v sl sol in acet; insol in w, bases & most acids; method of prepn is not known

The expl and other props of TACOT are as follows (Ref 2):

Brisance by Sand Test, 88% of PETN

Compatibility with metals, compatible with lead, aluminum, copper & steel

Compressive strength, 6000 psi

Detonation rate, 7250 m/sec at d 1.64 g/cc

Explosion temp, 337° in 15 mins and 388-400° in 0.1 sec

Impact sensitivity, 50% point is greater than 56 inches with 5kg wt; 50% point is 102cm on Type 12 apparatus

Power by ballistic mortar, 96% of TNT

Sensitivity to electrostatic discharge, no deton occurred when 3 grains of unconfined loose chge was subjected to a 30,000 volt discharge from a 2000 micro-micro-farad condenser

Sensitivity to initiation, 0.40 grams = 0.0258 gram of LA reqd to initiate

Shaped charge penetration, 85% of RDX

Uses, TACOT is a secondary HE patented by the DuPont Co as a high-temp, heat resistant expl (Ref 3). See also Refs 4, 5 & 6)

Refs: 1) M.F. Murphy & N.L. Coleburn, "A Preliminary Evaluation of TACOT, a New Heat Resistant Explosive", NOLTR **61-155** (Nov 1961) (Conf, not used as a source of info) 2) DuPont Technical Info Data Sheet on "TACOT" Properties (no date) 3) Anon, Kemixon Reporter, Barcelona, Item 12-2123 (Feb 1963) 4) R.A. Carboni, GerP 1164900 (1964) & CA **61**, 1701 (1964) (Describes prepn, props & uses) 5) J.F. Kenney, USP 3131102 (1964) & CA **61**, 5446 (1964) (Priming compn: $KClO_3$ 53, Sb_2S_3 25, $CaSi_2$ 12 & TACOT 10%, stable & sensitive to 400°F) 6) G.A. Noddin, USP 3326731 (1967) & CA **67**, 5265-66 (1967) (TACOT detonating sheet expl)

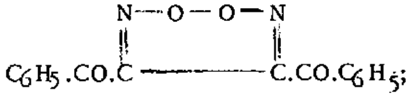
Dibenzoyl and Derivatives

Dibenzoyl, Bibenzoyl, Diphenylglyoxal or Diphenyldiketone. See Benzil in Vol 2 of Encycl, p B64-L

Nitrated Derivatives. See under Benzil and Derivatives, Vol 2, p B64-L to B65-R

Dibenzoyl Peroxide. See as separate entry in this Volume

4,5-Dibenzoyl-1,2-dioxa-3,6-diazine (called Diphenyldinitrosacyl in Ger by Holleman (Ref 2) and Dibenzoylglyoximeperoxyde or Diphenyl-dinitrosacyle in Fr by Böeseken (Ref 2a)

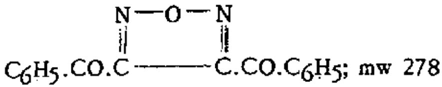


; mw 294.26, N, 9.52%; crysts (from chlf), mp 188°, explodes at higher temp with a luminous flame; was obtd when the oil which separates in the prepn of $C_6H_5CH{:}N.OH$ was treated with ether and the ppt purified from hot acet ac. Its *Azine*, orn-yel plts, mp - dec 207°, was also prepd

Refs: 1) Beil-not found 2) A.F. Hollemann, Ber **20**, 3361 (1887); **21**, 2840 (1888); and Rec **11**, 258 (1892) 2a) J. Böeseken, Rec **29**, 275 (1910) & CA **4**, 2450 (1910) 3) O. Widman & E. Virgin, Ber **42**, 2794 (1909) & CA **3**, 2694-95 (1909) 4) G. Ponzio et al, Gazz **61**, 589 (1931) & CA **26**, 712 (1932); **62**, 633 (1932) & CA **26**, 5564 (1932) 5) S. Cusmano, Gazz **68**, 129 (1938) & CA **32**, 6234 (1938) 6) R. Scarpati & M. Rippa, Gazz **88**, 804 (1958) & CA **53**, 18893 (1959)

NOTE: Compare with Dibenzoylfuroxan

3,4-Dibenzoylfurazan (called 3.4-Dibenzoylfurazan or 3.4-Dibenzoyl-oxidiazol in Ger),



$$\begin{array}{c} N-O-N \\ \| \qquad \| \\ C_6H_5.CO.C-C.CO.C_6H_5 \end{array}$$

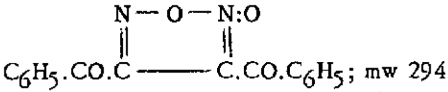
; mw 278.26, N 10.07%; col crysts (from alc), mp 118°; diffc sol in alc; was prepd by reducing the low-melting form of Dibenzoylfuroxan with $ZnCl_2$ & concd HCl in glacial acetic acid below 40-50°

Refs: 1) Beil **27**, 684 & [773] 2) I. de Paolini, Gazz **57**, 658(1927) & CA **22**, 578(1928)

NOTE: Nitrated derivs of 3,4-Dibenzoylfurazan were not found in Beil or in CA thru 1961

Dibenzoylfuroxan and Derivatives

3,4-Dibenzoylfuroxan; 3,4-Dibenzoylfurazan-2-oxide; or 3,4-Bis (benzoyl)-1,2-dioxa-2,5-diazene (called 3.4-Dibenzoyl-furazan-oxyd or Dibenzoylfuroxan in Ger),



$$\begin{array}{c} N-O-N{:}O \\ \| \qquad \| \\ C_6H_5.CO.C-C.CO.C_6H_5 \end{array}$$

; mw 294.26, N 9.52%; crysts: exists as a high-melting form, mp 188° and as a low-melting form (from eth or glac acet ac), mp 86-87°; deflgr when heated in a test tube; readily sol in alc & eth; sl sol in petr eth; insol in w; both forms are obtd by reaction of acetophenone & HNO_3 and by other methods

NOTE: Compare with 4,5-Dibenzoyl-1,2-dioxa-3,6-diazine

Ref: Beil **27**, 684, (605) & [773]

3,4-Di (p-nitrobenzoyl) -furoxan or 3,4-Bis(p-nitro-benzoyl) -furoxan,

$$\begin{array}{c} N-O-N{:}O \\ \| \qquad \| \\ O_2N.C_6H_4.CO.C-C.CO.C_6H_4.NO_2 \end{array}$$

; mw 384.26, N 14.58%; wh crysts (from 2-nitropropane), mp 154°; was prepd by reacting p-nitroacetophenone with concd HNO_3 and warming cautiously to 55° (Ref 3)

By a similar procedure, Snyder & Boyer (Ref 3) prepd *3,4-Bis(m-nitrobenzoyl)-furoxan*, wh crysts, mp 150°. They also prepd the respective *Azines* of the p-nitro deriv, orn-yel crysts, mp 252°; and of the m-nitro deriv - bright yel crysts, mp 251°

The IR absorption spectra of the m- & p-nitro derivs are found in Ref 4. Okuda (Ref 6) also reported the prepn of these derivs

Refs: 1) Beil-not found 2) ADL Synthesis HE's **3rd** Rept(1953), 369, 372 3) H.R. Snyder & N.E. Boyer, JACS **77**, 4235(1955) & CA **50**, 4922(1955) 4) N.E. Boyer et al, JACS **77**, 4240(1955) & CA **49**, 15487(1955) 5) ADL Synthesis HE's **4th** Rept(1956), 54-56 6) T.O. Kuda, Yakugaku-Zasshi **78**, 808-09 (1958) & CA **52**, 18382(1958)

NOTE: See also 4,5-Dibenzoyl-1,2-dioxa-3,6-diazine

Dibenzoyl-hydrazine. See Bis(benzoyl)-hydrazine in Vol 2 of Encycl, p B134-R

DIBENZOYL PEROXIDE AND DERIVATIVES

Dibenzoyl Peroxide or Bis(benzoyl)-peroxide (called Dibenzoylperoxyd, Benzoylsuperoxyd or Benzoylperoxyd in Ger), $C_6H_5CO.O.O.CO.C_6H_5$; mw 242.22, O 26.42%; wh rhomb cryst (from eth); odorless & tasteless; mp 104-06° (dec), bp - explodes; Q^{v}_{comb} 1552kcal/mol (Ref 6) & 6417cal/g (Ref 8); n_D 1.545 at 20°; sol in acet, eth, glac acet ac, benz, pyridine, CS_2 & chlf; sl sol in w, MeOH, cold alc & petr eth; was first prepd by Brodie (Ref 2) from benzoyl chloride & hydrated barium peroxide. Other methods of prepn are given in Beil (Ref 1) & other Refs. A coml method of prepn from benzoyl chloride & Na peroxide is described by Schwyzer (Ref 7)

Dibenzoyl peroxide is flammable, expl, oxidizing substance. It is dangerous to handle dry; has been known to explode during prepn, purification & storage. A sample exploded mildly at 100° even under water. The crude product has also exploded at RT over H_2SO_4 in a desiccator. A mixt of Dibenzoyl peroxide & $KMnO_4$ will deflgr, and a mixt with Na_2O_2 is flammable. The peroxide may be rendered insensitive to expln by mixing it with an excess of NaCl or Amm phosphate while still wet and then drying the mixt. A dry mixt of peroxide 20% & NaCl 80% was reported stable after storage for at least 20 days at 45-50°

The *explosion temp* value of a sample contg 10% H_2O_2 is 125° (smoke); *impact test* with PA App & 2kg wt 4 inches for a 16 mg sample; and *Brisance* by sand test 5.2 g

Uses. Dibenzoyl peroxide is widely used as an oxidizing agent in the food industry, as a polymerization initiator in the manuf of plastics, as a bleaching agent in the decolorization of oils, fats & waxes; and in pharmaceutical prepns for applications to the skin. It was patented (Ref 4) as a component of elec detonators (MF 60 & Dibenzoyl peroxide 40) and in admixture with Pb or Cuprammonium thiosulfate & $KClO_3$

Commercial Dibenzoyl peroxide is manufd in this country under the name "**Lucidol**" by the Lucidol Division of Wallace & Tiernan Inc, Buffalo, NY. Other trade names of the coml product are "**Luperco**" (finely dispersed with an org or inorg filler) and "**Luperox**" (pastes, dispersed in water or oils of various types)

Shipping Regulations. ICC regulations req a yellow label (flammable solid & oxidizing material) and one pound net fiber packing containers. Max shipping wt by rail express is 25-lbs per shipping case (Ref 23)

For addnl info on the props, reactions and nature of Dibenzoyl peroxide, see the following Refs

Refs: 1) Beil **9**, 179, (93) & [157] 2) C. Brodie, JCS **17**, 268 (1864) 3) H. von Pechmann & L. Vanio, Ber **27**, 1511 (1894); **30**, 2003 (1897); **33**, 1046 (1900) (Prepn from benzoyl chloride, H_2O_2 & NaOH 4) A. Jaques, BritP 23450 (1912); JSCI **32**, 674 (1913) & CA **8**, 1347 (1914) 5) R.C. Farmer, JSCI **40**, 84T-86T (1921) & CA **15**, 2356 (1921) (Prepn & props) 6) Anon, Jahresber CTR **4**, 72 (1924-25) 7) J. Schwyzer, "Die Fabrikation Pharmazeutischer und Chemisch-Technischer Produkte", J. Springer, Berlin (1931) (Reproduced by Edwards Bros, Ann Arbor, Mich) 8) A. Schmidt, SS **29**, 263 (1934) 9) K. Nozaki & P.D. Bartlett, JACS **68**, 1686-92 (1946) and W.E. Cass, JACS **68**, 1976-82 (1946) (Kinetics of decompn in solvs) 10) Kirk & Othmer **2** (1948), 479-83 (F.V. Hooft, Benzoyl Peroxide) 11) J. Pratt, MSCE **34**, 385-87 (1948) & CA **44**, 5593 (1950) (Sur une Explosion de Peroxyde de Benzoyle) 12) G.R. Lappin & D.L. Davidson, C & EN **26**, 3518 (1948) (An expln is described with suggested explanation & precautionary measures; in handling of Dewar flasks wrapping with friction tape is advised) 13) D. Taub C & EN **27**, 46 (1949) & CA **43**, 1570 (1949) (Safe purification of Dibenzoyl Peroxide) 14) C.G. Swain et al, JACS **72**, 5426-34 (1950) (Spontaneous thermal decompn) 15) D.A. Sutton, Chem & Ind **1951**, 272 & CA **45**, 8985 (1951) (Expln on attempted reduction with $LiAlH_4$) 16) K.S. Bagdasar'yan & R.I. Milyutinskaya, ZhurFizKhim **27**, 420-32 (1953) & CA **49**, 5393 (1955) (Decompn in various solvs) 17) L. Horner & C. Betzel, ChemBer **86**, 1071-72 (1953) & CA **49**, 10671 (1954) (Expl decompn by dimethylaniline) 17a) Tobolsky & Mesrobian (1954), 169 18) D.F. De Tar & L.A. Carpino, JACS **77**, 6370-71 (1955) & CA **50**, 9330-31 (1956) (A new method of prepn) 19) H. Kracker & T. Jacobs, GerP 934592 (1955) & CA **50**, 12112 (1956) (Method of manuf) 20) T.D. Manly, IndChemist **32**, 271-76 (1956) (Props & industrial uses) 21) T. Sumimoto, JapP 5667 (1959) & CA **53**, 21818-19 (1959) (Manuf) 22) Hawkins, OrgPeroxides (1961), 310 23) Sax (1963), 495-96

Analysis of Dibenzoyl Peroxide. The purity of coml Dibenzoyl peroxide can be detd by dissolving a 0.5 g sample in 15 ml of chlf. The soln is cooled to -5° and 25 ml of 0.1N ice-cold soln of Na methylate is added at once with cooling & shaking. After 4 to 5 mins at -5°, 100 ml of ice water, 5 ml of 10% H_2SO_4 and 2 g KI in 20 ml of 10% H_2SO_4 are added in the order given, with violent stirring. The liberated iodine is titrated with 0.1N Na thiosulfate soln. One cc of 0.1N Na thiosulfate is equiv to 0.0121 g of Dibenzoyl peroxide (Ref 1). See also Refs 2 & 3

Refs: 1) G. Braun, OrgSynth **13** (1933), 87-88 2) P.A. Giguère & D. Lamontagne, CanJChem **29**, 54-59 (1951) (Polarographic detn of Dibenzoyl peroxide) 3) A.T. Blomquist & A.J. Buselli, JACS **73**, 3887 (1951)

Dinitrodibenzoyl Peroxide or Bis(nitrobenzoyl)-peroxide, $[O_2N.C_6H_4.CO]_2O_2$; mw 332.22, N 8.43%. The following isomers are known:

2,2'-Dinitrodibenzoyl Peroxide or Bis(o-nitrobenzoyl)-peroxide, crysts (from acetophenone at 50°), mp 145° dec (Ref 5) or col plates (from chlf), mp 147° expl decompn (Ref 4); was prepd by shaking o-nitrobenzoyl chloride in chlf at 2° with H_2O_2 & aq NaOH (Ref 4)

3,3′-Dinitrodibenzoyl Peroxide or Bis(m-nitrobenzoyl)-peroxide, ndls (from ethyl acet), mp 134°, 137° & 139-40° (dec); readily sol in eth, acet & benz; decomp explosively in contact with concd HNO_3 or H_2SO_4; prepd as above (Refs 1, 4 & 5)

4,4′-Dinitrodibenzoyl Peroxide or Bis(p-nitrobenzoyl)-peroxide, yel ndls (from toluene), mp 151° (dec) & 156° (dec); prepd by the method of Price & Krebs (Ref 2, 3, 4 & 5)

Refs: 1) Beil **9**, 381, (154) & [252] 2) Beil **9**, 394 & [270] 3) C.C. Price & E. Krebs, OrgSynth **23**, 65-67 (1943) 4) D.H. Hey & E.W. Walker, JCS **1948**, 2216 & CA **43**, 3394 (1949) & CA **46**, 2523 (1952) 5) A.T. Blomquist & A.J. Buselli, JACS **73**, 3886, 3888 (1951)

Tetranitrodibenzoyl Peroxide or Bis(dinitrobenzoyl)-peroxide,
$[(O_2N)_2C_6H_3.CO]_2O_2$; mw 422.22, N 13.27%. Only one isomer is known:

3,5,3′,5′-Tetranitrodibenzoyl Peroxide or Bis(3,5-dinitrobenzoyl)-peroxide, crysts (from boiling chlf, cooled and MeOH added), mp 161-62° (dec) (Ref 3), mp 158° (Ref 2); prepd by the method of Price & Krebs (See Ref 3 above)

Refs: 1) Beil-not found 2) W. Cooper, JCS **1951**, 3112 & CA **47**, 4125 (1953) 3) A.T. Blomquist & A.J. Buselli, JACS **73**, 3886, 3888 (1951)

NOTE: Higher nitrated derivs of Dibenzoyl Peroxide were not found in Beil or in CA thru 1961

Di(benzoylperoxy)-cyclohexane. See Bis (benzoylperoxy)-cyclohexane in Vol 2 of Encycl, p B135-L

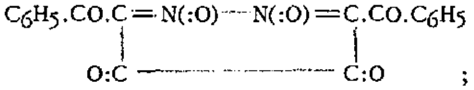
Di(benzoylperoxy)-dicyclohexylperoxide. See Bis(benzoylperoxy)-dicyclohexylperoxide in Vol 2, p B135-L

Di(benzoylperoxy)-dimethyl-hexane. See Bis (benzoylperoxy)-dimethyl-hexane in Vol 2, p B135-R

Di(benzoylperoxy)-fluorene. See Bis(benzoylperoxy)-fluorene in Vol 2, p B135-R

Di(benzoylperoxyfluorenyl)-peroxide. See Bis(benzoylperoxyfluorenyl)-peroxide in Vol 2, p B136-L

3,6-Dibenzoyl-1,2,4,5-tetraketo-4,5-dihydropyridazine or 3,6-Dibenzoyl-4,5-pyridazinedione-1,2-dioxide



$C_6H_5.CO.C{=}N(:O){-}N(:O){=}C.CO.C_6H_5$; O:C——C:O

mw 350.28, N 8.00%; brick-red crysts: rhmb plates (from acet-eth) or ndls (on evapn from acet) + 2 mols solv, mp - explodes on heating in a capillary tube or on Pt block; sol in acet, benz & anisole; sol in H_2SO_4 with decompn; sl sol in benz, chlf & AcOH; was prepd by treating 1,4-dibenzoyl butane-2,3-dione $(C_6H_5COCH_2CO)_2$ in ether with N_2O_4 at 25° for 20 mins

Refs: 1) Beil-not found 2) E.J. Virgin, Diss, Univ Uppsala **1914**, 63pp & CA **14**, 1319 (1920)

Dibenzyl or sym-Diphenylethane. See Bibenzyl in Vol 2, p B110-L

Dibenzylamine and Derivatives

Dibenzylamine or Bis(benzyl)-amine (called Dibenzylamin in Ger), $(C_6H_5.CH_2)_2NH$; mw 197.27, N 7.10%; col to lt-yel liq, fr p -25.6 to -26.6°, bp 215° at 39 mm press & 268-71° at 250 mm press; d 1.0281; n_D 1.5743 at 21.6°; Q^v_{comb} 1851 kcal/mol; readily sol in alc & eth; insol in w; methods of prepn & other props are given in Beil

Ref: Beil **12**, 1035, (453) & [553]

Mononitrodibenzylamine,
$O_2N.C_6H_4.CH_2.NH:CH_2.C_6H_5$; mw 242.27, N 11.56%. Only the 2-*Nitro deriv* is described in Beil **12**, 1078

Dinitrodibenzylamine, $(O_2N.C_6H_4.CH_2)_2NH$; mw 287.27, N 14.63%; the following isomers are known:

2,2'-Dinitrodibenzylamine, crysts (from alc), mp 99-102°; forms a Hydrochloride salt, $C_{14}H_{13}N_3O_4$ + HCl, dec at 238°; and a Nitrate, $C_{14}H_{13}N_3O_4 + HNO_3$, lt-yel ndls (from w), mp 200-03° dec (Ref 1)

3,3'-Dinitrodibenzylamine, citron-yel prisms (from alc or eth), mp 83.5°; diffc sol in w; forms a Hydrochloride salt, yel prisms (from dil HCl), mp 253°; and a Nitrate, lt-yel prisms (from dil HNO_3), mp 163° dec (Ref 2)

4,4'-Dinitrodibenzylamine, yel plts (from alc), mp 93°; sol in hot alc; insol in w & eth; forms a Hydrochloride, ndls (from w), mp 217-18°; and a Nitrate, yel plts (from dil HNO_3), mp dec 210-11° (Ref 3)

Refs: 1) Beil **12**, 1078, (466) & [577] 2) Beil **12**, [579] 3) Beil **12**, 1086, (467) & [581]

Trinitrodibenzylamine, $C_{14}H_{12}N_4O_6$, not found in Beil or in CA thru 1961

2,6,2',6'-Tetranitrobenzylamine or Bis-(2,6-dinitrobenzyl)-amine, $[(O_2N)_2C_6H_3.CH_2]_2NH$; mw 377.27, N 18.56%; dk-yel ndls (from alc), mp 194°; obtd by introducing NH_3 into a benzene soln of 2,6-dinitrobenzylbromide; forms a *Chloroplatinate salt,* mp 236°; *Hydrobromide,* mp 202°; and a *Sulfate,* mp 235°

Equimolar mixts of 2,6-dinitrobenzylbromide & K phthalimide, when heated above 130°, lead to explosions

Refs: 1) Beil **12**, (467) 2) S. Reich & A. Oganessian, BullFr **21**, 118 (1917) & CA **11**, 3028-29 (1917)

NOTE: Higher nitrated derivs of Dibenzylamine were not found in Beil or in CA thru 1961

ω,ω'-Dibenzylidene-diaminoguanidine. See Bis(benzylideneamino)-guanidine in Vol 2 of Encycl, p B136-L

Dibenzylidene Diperoxide. See Dibenzal Diperoxide

Dibenzylmethane and Derivatives

α,γ-Dibenzylmethane or 1,3-Diphenylpropane (called 1.3-Diphenylpropan or Dibenzylmethan in Ger), $C_6H_5.CH_2.CH_2.CH_2.C_6H_5$; mw 196.28, H 8.22%; liq, fr p 6°, bp 301-03°, 160° at 18 mm press, d 0.9855, n_D 1.5634; other props & many methods of prepn are given in Beil

Ref: Beil **5**, 613, (288), [517] & {1864}

Nitroso-nitro-dibenzylmethane (called 2-Nitroso-2-nitro-1,3-diphenyl-propan in Ger); $C_6H_5.CH_2.C(NO)(NO_2).CH_2.C_6H_5$; mw 270.28, N 10.37%; blue rhmb crysts (from cold eth + alc), mp 88° (dec) & 72-73°; readily sol in eth, chlf & benz; less sol in glac acet ac; diffc sol in alc & petr eth; insol in w; was prepd by treating dibenzylketoxime with ethyl nitrite & HNO_3 in ether under cooling, or with N_2O_4 in eth at 0° (Refs 1 & 2)

Refs: 1) Beil **5**, [517] & {1865} 2) H. Reinboldt & M. Dewald, Ber **60B**, 250-51 (1927) & CA **21**, 1626 (1927)

Dinitro-dibenzylmethane, $C_6H_5.CH_2.C(NO_2)_2.CH_2.C_6H_5$; mw 286.28, N 9.79%; 127.5° & 132°; readily sol in chlf; less sol in Nitrobenz; sl sol in eth, glac acet ac & benz; v sl sol in alc & petr eth; insol in w; was prepd from the nitrosonitro deriv by oxidizing in glac acet acid with fuming HNO_3 (Refs 1 & 2)

Refs: 1) Beil **5**, [517] & {1865} 2) W. Charlton et al, JCS **1932**, 36 (Dibenzyldinitromethane)

X,X-Dinitrodibenzylmethane, $C_{15}H_{14}(NO_2)_2$; ndls (from chlf), mp 139°; obtd by treating dibenzylmethane in glacial acet acid with fuming HNO_3 (Refs 1 & 2)

Refs: 1) Beil **5**, 613 2) A. Michaelis & A. Flemming, Ber **34**, 1293 (1901) & JCS **80 I**, 438 (1901)

2,2-Dinitrodibenzyl-nitromethane or Bis(2-nitrobenzyl)-nitromethane, $O_2N.C_6H_4.CH_2.CH(NO_2).CH_2.C_6H_4.NO_2$; mw 331.28, N 12.69%; dk-yel crysts (from chlf + eth), mp 140-41.5°; readily sol in chlf; sl sol in alc, eth, w & glac acet ac; was prepd from nitromethane and o-nitrobenzylchloride in alc soln in the presence of Na ethoxide (Refs 1 & 2)

Refs: 1) Beil **5**, 613 2) T. Posner, Ber **31**, 657 (1898) & JCS **74 I**, 361 (1898)

X,X,X,X-Tetranitrodibenzylmethane or 1,3-Bis(X,X-dinitrophenyl) -propane,, $C_{15}H_{12}(NO_2)_4$; mw 376.28, N 14.89%; ndls (from chlf + alc), mp 162-69°; obtd by treating dibenzylmethane with fuming HNO_3 (Refs 1 & 2)
Refs: 1) Beil **5**, 613 & {1866} 2) A. Michaelis & A. Flemming, Ber **34**, 1293(1901) & JCS **80** I, 438(1901)

NOTE: Higher nitrated derivs of Dibenzylmethane were not found in Beil or in CA thru 1961

Di(p-biphenylyl)-amine or 4,4'-Diphenyl-diphenylylamine. See Bis(p-biphenylyl)-amine in Vol 2, p B136-R

Diborane. See Vol 2, p B253-R, under Boron Hydrides and p B255-L, under Boron Hydride Fuels. See also the following addnl Refs:
Refs: 1) F.R. Price, JACS **72**, 5361-65 (1950) & CA **45**, 2755(1951)(First & 2nd pressure limits of expln of Diborane-Oxygen mixts) 2) R.P. Clarke & R.N. Pease, JACS **73**, 2132-34(1951) & CA **45**, 7418(1951) (Kinetics of pyrolysis of Diborane) 3) J.K. Bragg et al, JACS **73**, 2134-40(1951) & CA **45**, 7418(1951)(Kinetics of pyrolysis of Diborane) 4) A.T. Whatley & R.N. Pease, JACS **76**, 1997-99(1954) & CA **48**, 8543(1954) (Thermal expln of Diborane-oxygen mixts) 5) W.G. Parker & H.G. Wolfhard, Fuel **35**, 323-32(1956) & CA **50**, 12437(1956)(Props of Diborane flames) 6) E.L. Poling & H.P. Simons, IEC **50**, 1695-98(1958) & CA **53**, 9795(1959)(Expl reaction of Diborane in dry & water-saturated air) 7) P.E. Sample & H.P. Simons, IEC **50**, 1699-1702(1958) & CA **53**, 9795(1959)(Expl reactions of Diborane in benzene-saturated air) 8) P. Breisacher et al, "Flame Front Structures of Lean Diborane-Air and Diborane-Hydrocarbon-Air Mixtures", pp 894-902 in 7thSymp Deton (1959) 9) F.J. Martin et al, "Condensation of Products in Diborane-Air Detonations", pp 633-44 in 8thSympCombstn (1962)

Di(bornylamino)diazido-copper, $(N_3)_2Cu(H_2N.C_{10}H_{17})_2$; mw 454.10, N 24.68%; crysts, bp - explodes 207-08°, burns quietly in a flame; insensitive to impact; insol in w, but slowly decomp; prepd by dissolving $Cu(N_3)_2$ in 2 mols of bornylamine in the presence of acid, or by adding NaN_3 to an amine soln of the Cu salt
Refs: 1) Beil-not found 2) A. Cirulis & M. Straumanis, JPrakChem **162**, 310-11(1943) & CA **38**, 1969(1944)

Dibromoacetylene or Dibromomethyne (called Dibromacetylen or Dibromäthin in Ger), Br.C:C.Br; mw 230.74; oily liq having a penetrating odor, fr p -25 to -23° forming ndls, bp 76-76.5°(in atmosphere of CO_2); explodes on heating in air; dec in damp air or in light; very toxic; prepd by interaction of Mg dibromoacetylene and an ethereal soln of cyanogen bromide, or by interaction of tribromoethylene & alc KOH
Refs: 1) Beil **1**, 246, [222] & {919} 2) CondChemDict (1961), 356 3) Sax (1963), 675

Dibromoaminobenzene Nitro Derivs. See under Dibromophenylamine

Dibromoaniline. Its *Mononitro & Dinitro derivs* are not expl. See under Dibromophenylamine and Derivatives.

Dibromoanilino-ethane, See Bis(bromoanilino)-ethane in Vol 2 of Encycl, p B136-R

Dibromoanilino-propane. See Bis(bromoanilino)-propane in Vol 2, p B137-L

DIBROMOBENZENE AND DERIVATIVES

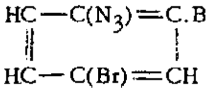
Dibromobenzene (called Dibrombenzol in Ger), $C_6H_4Br_2$; mw 235.92. All isomers are known: *1,2(or ortho)-Dibromobenzene,* liq having a characteristic odor, fr p 6.4°, bp 223-24° at 760 mm, d 1.9759 at 25°, n_D 1.6086 at 25°; other props & methods of prepn in Ref 1

1,3(or m)-Dibromobenzene, liq, fr p -6.7°, bp 218-18.6°, d 1.957 at 23°, n_D 1.5914 at 55°; other props & methods of prepn are given in Ref 2

1,4(or p)-Dibromobenzene, sublimable plts (from alc, petr eth or acet) or monoclinic prisms, mp 87.5°, bp 220.4°, d 2.2836 at 24.2°, d(liq) 0.9641 at 99.6°; other props & methods of prepn are given in Ref 3

Refs: 1) Beil **5**, 210, (116), [162] & {564} 2) Beil **5**, 211, (116), [162] & {565} 3) Beil **5**, 211, (116), [163] & {566}

1-Azido-2,3(or m)-Dibromobenzene or 2,4-Dibromophenyl Azide (called 2.4-Dibrom-phenylazid or 2.4-Dibrom-1-azido-benzol in Ger),



; mw 276.95, N 15.18%; yel ndls (from acet, alc or benz), mp 62°, probably explodes when heated rapidly to higher temps; v sol in eth; sol in hot alc; sl sol in w; prepd by introducing NH_3 into 2,4-Dibromobenzene-1-diazonium tetrachloroiodide in the cold (Refs 1 & 2). The *2,4-Dichlorophenyl Azide* explodes when heated rapidly or when sharply struck (See Vol 2 of Encycl, p B44-R)

Refs: 1) Beil **5**, 278 & [209] 2) F.D. Chattaway et al, JChemSoc **125**, 1988 (1924)

Mononitrodibromobenzene, $C_6H_5Br_2NO_2$; mw 280.92, N 5.00%. All possible isomers are known:

1-Nitro-2,3-dibromobenzene, monoclinic prisms, mp 85°, d 2.358, d 1.9764 at 109.5°; readily sol in acet & chlf; sol in eth, eth acet & 3 pts of hot acet ac (Ref 1)

1-Nitro-2,4-dibromobenzene, yel plts or triclinic prisms (from alc), mp 61-62°, vol on steam bath, sublimes w/o decompn, d 1.9581 at 111°; sol in hot alc; diffc sol in cold (Ref 2)

1-Nitro-2,5-dibromobenzene, lt-yel plts (from acet, acet + petr eth, or eth-alc), mp 84-85°, d 1.9146 at 111° (Ref 3)

1-Nitro-2,6-dibromobenzene, monoclinic prisms (from alc), mp 82-84°, sublimes on steam bath, d 1.9211 at 111° (Ref 4)

1-Nitro-3,4-dibromobenzene, monoclinic prisms (from alc or glac acet ac), mp 58-59°, bp 296°, volatile on steam bath, d 1.9835 at 111°; readily sol in glac acet ac (Ref 4)

1-Nitro-3,5-dibromobenzene, ndls (from alc), prisms or plts (from eth), mp 106°, volatile on steam bath, d 1.9341 at 111°; insol in KOH (Ref 3)

Other props & methods of prepg the Mononitro derivs are found in Beil

Refs: 1) Beil **5**, 249 & [190] 2) Beil **5**, 250, [190] & {620} 3) Beil **5**, 250, [190] & {621} 4) Beil **5**, 250 & {621}

Dinitrodibromobenzene, $C_6H_2Br_2(NO_2)_2$; mw 325.93, N 8.60%. All possible isomers are known:

1,2-Dinitro-3,4-dibromobenzene, greenish crysts (from CS_2), mp 109°, d 2.375; obtd with other isomers by nitrating 1-nitro-2,3-dibromobenzene with mixed acids (Ref 1)

1,2-Dinitro-3,5-dibromobenzene, mp 84.8-86°, trimorphic: stable form, monoclinic prisms (from concd soln of eth-alc or alc, d 2.274; metastable form, monoclinic prisms (from cooling a saturated soln in eth acet), d 2.317; and labile form, rhmb bipyramidal crysts (from seeding an alc-eth soln); prepd by nitrating 1-nitro-3,5-dibromobenzene with mixed acid on a water bath (Ref 1)

1,2-Dinitro-3,6-dibromobenzene, monoclinic prisms (from CS_2 or glac acet ac), mp 159-60°; readily sol in hot abs alc; insol in w; prepd by nitrating p-dibromobenzene with mixed acid, but a p-dibromo-dinitrobenzene isomer is also obtd (Ref 2)

1,2-Dinitro-4,5-dibromobenzene, rhmb ndls (from glac acet ac) or crysts (from CS_2), readily sublimes by heating on a w bath, mp 114-15°, d 2.313; mod sol in eth, chlf & CS_2; diffc sol in cold alc, petr eth & glac acet ac; obtd by warming o-dibromobenzene or 1-nitro-3,4-dibromobenzene with mixed acid (Ref 1)

1,3-Dinitro-2,4-dibromobenzene, almost col ndls or yellowish-grn plts (from alc), mp 83°; prepd by nitrating either m-dibromobenzene or 1-nitro-2,6-dibromobenzene (Ref 3)

1,3-Dinitro-2,5-dibromobenzene, ndls (from CS_2, glac acet ac or alc), mp 119-20°; obtd with other isomers by nitrating p-dibromobenzene with mixed acid (Ref 2)

1,3-Dinitro-4,5-dibromobenzene, monoclinic prisms (from CS_2), mp 71°, d 2.373; readily sol in alc & glac acet ac; obtd with other isomers by nitrating 1-nitro-2,3(or 3,4)-dibromobenzene (Ref 4)

1,3-Dinitro-4,6-dibromobenzene, mp 117°; yel dimorph: stable form, rhmb crysts, d 2.295; sol in common solvs; and metastable form, monoclinic prisms (from a cooled saturated in eth + a little alc or by seeding a saturated soln in eth acet); prepd by nitrating m-dibromobenzene or 1-nitro-2,4-dibromobenzene with mixed acid (Ref 5)

1,4-Dinitro-2,3-dibromobenzene, monoclinic prisms (from CS_2), mp 156.5°; diffc sol in alc & eth; sl sol in CS_2; obtd with other isomers by heating 1-nitro-2,3-dibromobenzene with mixed acid at 100° (Ref 4)

1,4-Dinitro-2,5-dibromobenzene, pale-yel prisms (from alc or benz + alc), mp 126-27°; sol in benz, chlf & CS_2; sl sol in eth & cold alc; insol in w & petr eth; obtd with other isomers by nitrating p-dibromobenzene (Ref 6)

1,4-Dinitro-2,6-dibromobenzene, prisms (from alc), mp 130°; prepd from 4-nitro-2,6-dibromobenzenediazonium nitrate & $NaNO_2$ soln at 0° (Ref 7)

Other props of the dinitro derivs are given in Beil

Refs: 1) Beil **5**, 267 2) Beil **5**, 267 & {641} 3) Beil **5**, 267 & (139) 4) Beil **5**, 268 5) Beil **5**, 268, [202] & {641} 6) Beil **5**, 268 & {641} 7) Beil **5**, (139)

1,3,5-Trinitro-2,4-dibromobenzene, $C_6HBr_2(NO_2)_3$; mw 370.92, N 11.33%; lt-yel prisms having a greenish shimmer, mp 135°; readily sol in eth, chlf & acet; sl sol in alc; insol in w; can be prepd by nitrating 1-nitro-2,6-dibromobenzene, or 1,3-dinitro-2,4(or 4,6)-dibromobenzene; or by heating 1,3,5-trinitro-2,4,6-tribromobenzene with Na_2SO_3 in benzene contg alc (Refs 1 & 2). Its expl props were not reported

Refs: 1) Beil **5**, 275 & (141) 2) W. Körner & A. Contardi, AttiAccadLinceiRend [5] **22** II, 629 (1913) & CA **8**, 1761 (1914)

NOTE: No higher nitrated derivs of Dibromobenzene were found in Beil or in CA thru 1961

2,6-Dibromo-1,4-benzoquinone-4-oxime. See Vol 2, p 86-L and 4-Nitroso-2,6-dibromophenol under Dibromophenol in this Vol

Dibromodiaminoanthraquinone and Derivatives

Dibromodiaminoanthraquinone (called Dibromdiamino-anthrachinon in Ger), $C_{14}H_8N_2O_2Br_2$; mw 398.05, N 7.04%. Several isomers are described in Beil:

2,3-Dibromo-1,4-diaminoanthraquinone, $C_6H_4(CO)_2.C_6Br_2(NH)_2$ (Ref, p 203)

2,6-Dibromo-1,5-diaminoanthraquinone, $H_2N.C_6H_2Br(CO)_2C_6H_2Br.NH_2$, steel-blue ndls (from Nitrobenz), mp 274° [Ref, pp 208 & (470)]

1,5-Dibromo-2,6-diaminoanthraquinone, orn-colored powd [Ref, p (472)]

3,7-Dibromo-2,6-diaminoanthraquinone, yel ndls, mp - above 360° [Ref, p (472)]

3,6-Dibromo-2,7-diaminoanthraquinone, brn crysts (from aniline); sol in common org solvs; insol in w & alkalies [Ref, p (473)]

Ref: Beil **14**, 203, 208 & (470, 472, 473)

4,8-Dinitro-2,6-dibromo-1,5-diaminoanthraquinone, $H_2N.C_6H.Br(NO_2)(CO)_2C_6HBr(NO_2).NH_2$, lt-red crysts having a metallic luster (from boiling phenol or Nitrobenz), mp - above 360°; was prepd by treating the nitramino deriv with phenol & H_2SO_4 at RT (Refs 1 & 4)

4,8-Dinitro-2,6-dibromo-1,5-dinitraminoanthraquinone, $O_2N.HN.C_6H.Br(NO_2)(CO)_2.C_6HBr(NO_2).NH.NO_2$; mw 576.05, N 14.59%; diamond-shaped crysts, mp - deflgr 142-43°; diffc sol in boiling acet & glac acet ac; almost insol in other solvs; sol in concd H_2SO_4 giving a yel soln; was prepd by adding 2,6-dibromo-1,5-diaminoanthraquinone to colorless HNO_3 (d 1.52) and cooling

It forms an *Ammonium salt,* $(NH_4)_2C_{14}H_2N_6O_{10}Br_2$, red-brn ndls (from w), deflgr at 187°; *Sodium salt,* $Na_2C_{14}H_2N_6O_{10}Br_2$, red-brn crysts (from hot w), mp - deflgr on rapid heating; and other salts (Refs 2, 3 & 4)

Refs: 1) Beil **14**, 211 2) Beil **16**, 680 & (401) 3) R. Scholl et al, Ber **37**, 4441 (1904) & JCS **88** I, 70-71 (1905) 4) R. Scholl & A. Krieger, Ber **37**, 4682, 4683, 4686 (1904) & JCS **88** I, 145 (1905)

Dibromodiazophenol (called Dibrom-diazophenol or Dibrom-chinon-diazid in Ger), $C_6H_2N_2Br_2O$; mw 277.93, N 10.08%. Three isomers are found in Beil:

3,5-Dibromo-2-diazophenol, orn-yel prisms (from eth) or brn-yel ndls (from petr eth), sensitive to light, mp 130-40°(dec); very sol in chlf, benz, eth & glac acet ac; v sl sol in cold w & petr eth (Ref 1, p 523)

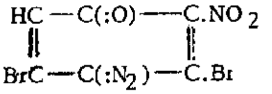
2,6-Dibromo-4-diazophenol, brn-yel prisms, deflgr at 145° or golden-yel ndls (from alc), deflgr at 156°; sol in concd HCl {Ref 1, pp 530 & [288]}

3,5-Dibromo-4-diazophenol, yel slanted prisms (from w), deflgr at 142°; readily sol in hot alc; sl sol in hot w, eth & chlf; almost insol in cold w (Ref 1, p 531)

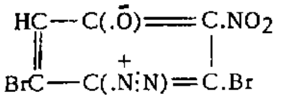
Other props & methods of prepn are given in Beil. The expl props of Dibromodiazophenols were not reported

Refs: 1) Beil **16**, 523, 530, 531 & [288]
2) See original papers by authors as given in Beil

2-Nitro-3,5-Dibromo-4-diazophenol,



or

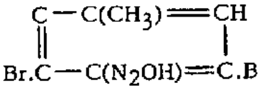


; mw 322.92, N 13.01%; golden-yel plts (from alc), mp dec 196°; obtd by contact of an aq soln of 2,4,6-tribromo-3-nitrobenzene-1-diazoniumsulfate with Na acetate (Refs 1 & 2). Its expl props were not reported

Refs: 1) Beil **16**, 531 2) K.J.P. Orton, JCS **83**, 810(1903)

NOTE: See also Dibromophenol and Derivatives

3,5-Dibromo-4-diazotoluene [called 3.5-Dibrom-toluol-diazoniumhydroxyd-(4) in Ger],



; known in the form of its salts:

Bromide, $C_7H_5Br_2.N_2.Br$; obtd as yel crysts when the alcoholic soln in which the base has been diazotized is immediately treated with eth; if allowed to stand for some time, eth ppts the salt as brn crysts; in dry state both salts are very expl, detonating at 97-98° (Refs 1 & 2)

Thiocyanate, $C_7H_5Br_2.N_2.SCN$, orn-colored crysts, dec w/o expln on heating to 40-50°, explodes by friction; obtd when a soln of 3,5-dibromo-4-diazotoluene sulfate is treated with an aq soln of KSCN (Refs 1 & 3)

Refs: 1) Beil **16**, 505 2) A.R. Hantzsch, Ber **30**, 2347(1897) & JCS **74** I, 19(1898) 3) B. Hirsch, Ber **31**, 1261(1898) & JCS **74** I, 474(1898)

3,5-Dibromo-2-diazotoluene-4-sulfonic Acid. See under Diazotoluenesulfonic Acid Halogenated Derivatives in this Vol

Dibromodimethylether. See item p in Vol 2 of Encycl, p C169-R, under CHEMICAL AGENTS

Dibromoethylsulfide. See item m in Vol 2, p C169-R under CHEMICAL AGENTS

Dibromofumaric Acid (called Dibromofumarsäure in Ger), HOOC.CBr:CBr.COOH; mw 273.89, O 23.37%; crysts (from w), mp 225°, bp - dec 230°;readily sol in alc & eth; was obtd with other products by introducing bromine into an aq soln of acetylenedicarboxylic acid

Its *Silver salt,* $Ag_2C_4O_4Br_2 + \frac{1}{2}H_2O$, ndls, explodes violently on heating (Refs 1 & 2)

Refs: 1) Beil **2**, 747, (303), [641] & {1910}
2) E. Bandrowski, Ber **12**, 2213-14(1879)

1,4-Di(bromoimino)-p-quinone. See Benzoquinone-1,4-di(bromoimine) in Vol 2 of Encycl, p B83-L

Dibromopentaerythritol, $(BrCH_2)_2C(CH_2OH)_2$, and its *Dinitrate,* $(BrCH_2)_2C(CH_2ONO_2)_2$, crysts, mp 70°, were prepd by Kouba et al (Ref 3). They also reported the prepn of *Monobromopentaerythritol,* $BrCH_2C(CH_2OH)_3$, and its *Trinitrate,* $BrCH_2.C(CH_2ONO_2)_3$, crysts, mp 91°. These derivs were prepd by reaction of pentaerythritol with hydrobromic acid in the presence of acetic anhydride, followed by nitration with HNO_3. Elrick et al (Ref 2) earlier prepd *Monobromopentaerythritol trinitrate,* wh crysts (from alc), mp 89-90°, by nitrating the dinitroderiv with 100% white HNO_3

Refs: 1) Beil-not found 2) D.E. Elrick et al, JACS **76**, 1374-75 (1954) & CA **49**, 3811 (1955) 3) D.L. Kouba et al, "High Explosives, Hercules Powd Co Monthly Progress Rept (4 Jan 1952) (Contract Nord-11280, TaskA)

DIBROMOPHENOL AND DERIVATIVES

Dibromophenol (called Dibrom-phenol or Dibrom-1-hydroxy-benzol in Ger), $C_6H_3Br_2.OH$; mw 251.92. All possible isomers are known:

2,3-Dibromophenol, prisms (from petr eth), mp 68-69° (Ref 1)

2,4-Dibromophenol, ndls (from petr eth), mp 40°, bp 243-46° at 740 (sl decompn) (Ref 2)

2,5-Dibromophenol, ndls (from petr eth), mp 73-74°, bp 256-57° at 755 mm press (Ref 6)

2,6-Dibromophenol, ndls (from petr eth, alc or w), mp 56-57°, bp 162° at 21 mm, 255-56° at 740 mm & 138° at 10 mm press (Ref 3)

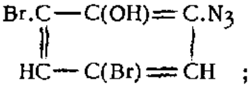
3,4-Dibromophenol, ndls (from CCl_4 or w), mp 79-80° (Ref 4)

3,5-Dibromophenol, monoclinic ndls (from petr eth), mp 81°, bp 274-76° at 750 mm press (Ref 5)

Other props & methods of prepn are given in Refs

Refs: 1) Beil **6**, [188] & {753} 2) Beil **6**, 202, (106), [188] & {753} 3) Beil **6**, 202, (106, [189] & {755} 4) Beil **6**, 203 & {756} 5) Beil **6**, 203, [189] & {756} 6) Beil **6**, {755}

2-Azido-4,6-Dibromophenol or 2-Hydroxy-3,5-dibromophenyl Azide,



$$\begin{array}{l} Br.C - C(OH) = C.N_3 \\ \| \qquad\qquad\qquad | \\ HC - C(Br) = CH \end{array}$$

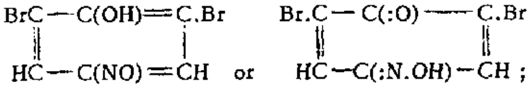
; mw 292.93, N 14.35%, crysts (from cyclohexane), mp 73-74°; was obtd when 4,6-dibromo-2-diazo-o-quinone reacted with $Al(N_3)_3$ in tetrahydrofuran at low temp, the soln washed with benz & w, and the benz residue recrystd (Ref 2). Its expl props were not investigated

Refs: 1) Beil-not found 2) R. Huisgen & I. Ugi, ChemBer **90**, 2915, 2925 (1957) & CA **52**, 15455 (1958)

NOTE: See also Dibromodiazophenol

Nitroderivatives of Dibromophenol

4-Nitroso-2,6-dibromophenol (called 4-Nitroso-2.6-dibrom-phenol or 2.6-Dibrom-chinon-oxim-(4) in Ger),



$$\begin{array}{l} BrC - C(OH) = C.Br \\ \| \qquad\qquad\qquad | \\ HC - C(NO) = CH \end{array} \quad \text{or} \quad \begin{array}{l} Br.C - C(:O) - C.Br \\ \| \qquad\qquad\qquad \| \\ HC - C(:N.OH) - CH \end{array}$$

; mw 280.92, N 4.99%; brn ndls (from tol, alc or glac acet ac) or plts (from dil alc), mp - darkens at 160° & detonates betwn 168-75° (Ref 1); Henry (Ref 2) obtd tan ndls (from MeOH), mp 168-69° (dec); readily sol in alc, eth & ethyl acetate; sol in NaOH & concd H_2SO_4; sl sol in chlf, glac acet ac & w; v sl sol in benz & petr eth; recently prepd by reaction of $NaNO_2$ on 3,5-dibromo-4-hydroxybenzoic acid in aq alc (Ref 2)

NOTE: This compd is described in Vol 2 of this Encycl, p B86-L, as 2,6-Dibromo-1,4-benzoquinone-4-oxime. It is listed here with addnl info and under alternate name with parent compd Dibromophenol

Refs: 1) Beil **7**, 641, (349) & [584] 2) R.A. Henry, JOC **23**, 650 (1958) & CA **52**, 17166 (1958)

Mononitrodibromophenol, $HO.C_6H_2Br_2NO_2$; mw 296.92, N 4.72%. Most possible isomers are known:

2-Nitro-3,6-dibromophenol, golden-yel ndls, mp 77° (Ref 1)

2-Nitro-4,6-dibromophenol, yel plts (from alc, eth or glac acet ac), mp 117-19°; forms numerous salts with metals (Ref 2)

2-Nitro-5,6-dibromophenol, yel ndls (from alc), mp 105° (Ref 3)

3-Nitro-4,6-dibromophenol, yel ndls (from petr eth), mp 77-78°; forms a Hydrate, $C_6H_3Br_2NO_3.H_2O$, yel ndls (from aq acet), mp 92-94° (after fusing) (Ref 3)
3-Nitro-5,6-dibromophenol, ndls (from benz), mp 106°; readily sol in alc & glac acet acid; v sl sol in chlf, petr eth (Ref 4)
3-Nitro-X,X-dibromophenol. See Ref 3
4-Nitro-2,5-dibromophenol, yel ndls (from 1,2-dichloroethane), mp 110°; not volatile on steam bath (Ref 5)
4-Nitro-2,6-dibromophenol, prisms or plts (from alc, petr eth or glac acet ac), mp 141-46°, volatile on steam bath, dec on heating above its mp (Ref 6)

Other props & methods of prepg the mononitro derivs are found in Beil
Refs: 1) Beil **6**, 245 & {847} 2) Beil **6**, 246, (123), [234] & {848} 3) Beil **6**, {848} 4) Beil **6**, [234] 5) Beil **6**, {849} 6) Beil **6**, 247, (123), [234] & {849}

Dinitrodibromophenol, $HO.C_6HBr_2(NO_2)_2$; mw 341.93, N 8.20%. Several isomers are known:
2,4(or 2,6)-Dinitro-3,5-dibromophenol, lt-yel ndls (from aq alc or w), mp 146-48°; v sol in alc & eth; almost insol in petr eth; obtd by heating 2,4(or 2,6)-dinitro-3,5-dibromoanisole with concd H_2SO_4 at 180-90°, or by heating 1,3-dinitro-2,4,6-tribromobenzene with NaOH (Ref 1)
2,4-Dinitro-3,6-dibromophenol, mentioned in Ref but no info is given (Ref 4)
2,5-Dinitro-4,6-dibromophenol, ndls (from aq alc), mp 137°; prepd by reacting 2,5-dinitrophenol with an excess of Br in 25% alc (Ref 3)
3,4-Dinitro-2,6-dibromophenol, ndls (from w), mp 142°; was prepd by reacting 3-nitro-2,4,6-tribromophenol with dil HNO_3 at 12°, or 3,4-dinitrophenol with Br (Ref 3)
X,X-Dinitro-2,4-dibromophenol, ndls (from alc), mp 146-46.5°; prepd by reacting propionic acid-2,4-dibromophenyl ester with 2/1 HNO_3/H_2SO_4; forms Ba & K salts (Ref 2)
Refs: 1) Beil **6**, 262 & [251] 2) Beil **6**, 262 3) Beil **6**, {872} 4) S.S. Joshi & T.C. Mathur, JIndianChemSoc **35**, 610-12 (1958) & CA **53**, 12224 (1959)

2,4,6-Trinitro-3,5-dibromophenol or Dibromopicric Acid, $HO.C_6Br_2(NO_2)_3$; mw 386.92, N 10.86%; crysts (from chlf, CCl_4 or benz), mp 165°, 173°, explosive; gives a yel color in alc or w; can be prepd by nitrating 3,5-dibromophenol with HNO_3 in glac acet ac, or with mixed acid
Refs: 1) Beil **6**, 292 & {973} 2) H. Willstaedt & F. Reuter, JPraktChem **135**, 211, 214 (1932) & CA **27**, 968 (1933)

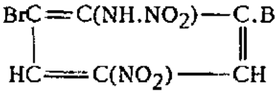
3,5-Dibromophenol-4-diazoniumhydroxide [called also 3.5-Dibrom-1-oxy-benzol-diazoniumhydroxyd-(4) in Ger], $HO.C_6H_2Br_2.N(:N).OH$; known as its *Nitrate,* which deflgr on heating; separated as an amorphous ppt when 2,4,6-tribromobenzene-1-diazoniumnitrate was heated with benz on a w bath (Refs 1 & 2)
Refs: 1) Beil **16**, 531 2) H. Silberstein, JPraktChem **27**, 107 (1883) 3) No later refs were found in the literature

Dibromophenylamine and Derivatives

Dibromophenylamine, Dibromoaminobenzene or Dibromoaniline, $C_6H_3NH_2Br_2$, may be considered as the parent compd of its nitro derivs.

The *Mononitro* and *Dinitro derivs* of Dibromoaniline are not expl. The following nitro derivs of Dibromophenylnitramine are of interest:

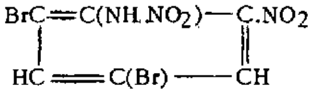
2,6-Dibromo-4-nitrophenylnitramine,



$$\begin{array}{lll} BrC= & C(NH.NO_2)- & C.Br \\ \;| & & \;\| \\ HC= & C(NO_2)- & CH \end{array}$$

; mw 340.94, N 12.35%; almost col ndls or yel-brn crysts (from benz or chlf), mp - explodes at 122°; readily sol in benz, chlf, eth & glac acet ac; less sol in alc & w; almost insol in petr eth & HCl; was prepd by nitrating either 2,6-dibromoaniline-4-sulfonic acid (Ref 3) or 3,5-dibromo-4-aminobenzaldehyde at low temp (Ref 4)

4,6-Dibromo-2-nitrophenylnitramine,



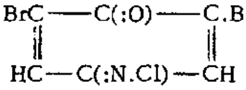
$$\begin{array}{lll} BrC= & C(NH.NO_2)- & C.NO_2 \\ \;| & & \;\| \\ HC= & C(Br)- & CH \end{array}$$

; orn prisms (from chlf + petr eth), mp 91-92°; was obtd by treating an acetic acid soln of 4,6-dibromo-2-nitroaniline with HNO_3, followed by addn of acetic anhydride in the cold. A *Barium salt,* $Ba(C_6H_2N_3O_4Br_2)_2$, yel plts, was also prepd.

Their expl props were not investigated (Ref 2)
Refs: 1) Beil **16**, 667 2) K.J.P. Orton, JCS **81** I, 811 (1902) 3) T. Zincke & A. Kuchenbecker, Ann **339**, 227 (1905) 4) L. Ellion, Rec **42**, 159-60 (1923)

Dibromophenyl Azide. See under Dibromobenzene

2,6-Dibromoquinonechlorimide [called 2,6-Dibrom-p-chinon-chlorimid-(4) in Ger],



$$\begin{array}{ccc} BrC & —C(:O)— & C.Br \\ \| & & \| \\ HC & —C(:N.Cl)— & CH \end{array}$$

; mw 299.38; yel prisms (from glac acet ac or alc), mp 80-83°, dec at 121°; was prepd by adding dropwise a 10% aq chloride of lime soln to a sl acidic soln of the Sn double salt of 2,6-dibromo-4-aminophenol (Ref 1)

It is reported by B.T. Taranto of Schering Corp, Bloomfield, NJ (Ref 2) that this reagent, used for spot visualization in chromatographic systems, presents a potential expln hazard. Tests on the compd were conducted after a small quantity exploded on a reagent shelf. In one test, 0.5 g was sealed in a thermal stability bomb and the temp was raised 5°C/min. At ca 120°, a violent exothermic reaction occurred, rupturing the burst diaphragm of the app at 1800 psi. Another test conducted on a 0.5 g sample at 60°C resulted in sudden decompn after 3 hrs at that temp. The sample temp rose sharply and exceeded the scale limit of 250°C. The same tests on the 2,6-*Dichloro deriv* (qv) showed similar, but less severe, instability.
Refs: 1) Beil **7**, 640, (349) & [584] 2) Anon, C & EN **45**, No 52, p 54 (11 Dec 1967)

Dibromoxylene. See item q in Vol 2, p C169-R, under CHEMICAL AGENTS

Di(butylamino)diazo- and triazo-copper Compounds.
Di(n-butylamino)diazo-copper,
$[(N_3)_2Cu(C_4H_9.NH_2)_2]$, green ndls, mp 113-14°, deflgr at 196°, does not expl under impact; was prepd by addn of NaN_3 to an amine soln of n-butyl Cu salt (Ref 2)
Di(iso-butylamino)diazo-copper,
$(N_3)_2Cu[(CH_3)_2CH.CH_2.NH_2]_2$, grn crysts, mp 139-40°, explodes at 198-200°, does not expl under impact; insol in w, alc & eth; prepd by above method using isobutylamine (Ref 2)
Di(iso-butylammonium)-triazo-copper,
$[(CH_3)_2CH.CH_2]_2NH[Cu(N_3)_3]$, grn-brn crysts, mp - explodes at 195-96°, but not under impact; prepd by dissolving $Cu(N_3)_3$ in isobutylamine in the presence of acid (Ref 3)
Refs: 1) Beil-not found 2) M. Straumanis & A. Cirulis, ZAnorgChem **251**, 346-47 (1943) & CA **37**, 6574 (1943) 3) M. Straumanis & A. Cirulis, ZAnorgChem **252**, 9-23 (1943) & CA **38**, 3563 (1944)

Di(iso-butyl)-benzidine (called Di-isobutylbenzidin in Ger),
$[-C_6H_4.NH.CH_2CH(CH_3)_2]_2$; may be considered the parent compd of its nitro derivs:
3,5,3',5'-Tetranitro-N,N'-diisobutylbenzidine,
$[-C_6H_2(NO_2)_2.NH.CH_2CH(CH_3)_2]_2$; mw 476.44, N 17.64%; dk red crysts (from ethyl ester of benzoic acid), mp 194°; obtd by heating 3,5,3',5'-tetranitro-4,4'-dimethoxy (or diethoxy) biphenyl with isobutylamine in alc in a sealed tube (Refs 1 & 2)
3,5,3',5',N,N'-Hexanitro-N,N'-diisobutylbenzidine,
$[-C_6H_2(NO_2)_2.N(NO_2).CH_2CH(CH_3)_2]_2$; mw 566.44, N 19.78%; ndls (from concd HNO_3), mp-dec at 205°; obtd from the tetranitro deriv by treating with concd HNO_3 (Refs 1 & 2)
Refs: 1) Beil **13** [109, 110] 2) G. van Romburgh, Rec **41**, 42, 43 (1922) & CA **16**, 1238 (1922)

N,N'-Dibutylcarbanilide. See Centralite, Butyl in Vol 2 of Encycl, p C140-L

Dibutyldiphenylurea. See Butyl Centralite under Centralites in Vol 2, p C140-L

Dibutyl Magnesium, $Mg(CH_2.CH_2.CH_2.CH_3)_2$; mw 138.54; clear, dk-amber liq; a 20% soln in toluene ignites on contact with red fuming HNO_3; was prepd by pptn of Mg chloride from butyl Mg chloride in ether soln with dioxane. This compd was prepd for evaluation as a fuel, or fuel additive in ram jets & rockets

Ref: I.J. Schaffner & P.O. Tawney, US Rubber Co March Summary Rept (1948), p 2 (Contract Nord 10129)

Di-t-butyl Peroxide or t- Butyl Peroxide, $(CH_3)_3C.O.O.C(CH_3)_3$; mw 146.22, O 21.88%; col liq, fr p -18° & -40°, bp 12-13° at 20 mm, 109-11° at 760 mm press; d 0.793 at 20°, n_D 1.3872 at 20°; stable in the presence of concd HCl & strong bases; can be prepd from t-butyl alcohol & H_2O_2; t-butyl hydrogen sulfate & t-butyl hydroperoxide; and by reaction of t-butyl alcohol or isobutylene, sulfuric acid & H_2O_2 (Ref 2). The latter reaction produces a mixt contg 50-70% t-butyl hydroperoxide which can be separated by fractionation under reduced pressure or extraction with strong alkali or several times with water

The pyrolysis of Di-t-butyl Peroxide shows that acetone & ethane are the only products formed up to 300° w/o expln; while t-Butyl Hydroperoxide decomposes explosively at 250° forming acetone, methanol, t-butyl alc, formaldehyde & water (Ref 2). The props & uses of these peroxides are tabulated by Perry & Seltzer (Ref 3)

A coml product gave the following props as detd at Picatinny Arsenal:

Brisance by Sand Test, 6.1 g sand crushed vs 49 g for TNT

Explosion temperature, 130° in 5 secs (smoke)

Impact sensitivity, PA App & 2 kg wt (sample 12 mg) +50 inches; Bur Mines App & 2 kg wt (sample 20 mg) 100^{+}cm

Di -t-butyl Peroxide is a very active high-temp polymerization catalyst. Vaughan & Rust (Ref 1a) patented the use of this & other di-t-alkyl peroxides as polymerization catalysts & diesel oil additives

Refs: 1) Beil-not found 1a) W.E. Vaughan & F.F. Rust, USP 2403771 (1946) & CA **40**, 5757 (1946) 2) N.A. Milas & D.M. Surgenor, JACS **68**, 205-08 (1946) 3) R.P. Perry & K.P. Seltzer, Modern Plastics **25**, 134-36, 216, 218, 220, 222 (1947) & CA **42**, 1557 (1948) 4) F.H. Dickey et al, IEC **41**, 1673-79 (1949) (Prepn, analysis & props); Q.T. Wiles et al, IEC **41**, 1679-82 (1949) (Applications) & CA **44**, 534-35 (1950) 5) Tobolsky & Mesrobian (1954), 18-20 & 178 6) F. Welch et al, JACS **77**, 551-54 (1955) & CA **50**, 780 (1956) (Prepn & props of Di-n-butyl, Di-sec-butyl & Diisobutyl Peroxides) 7) T.D. Manley, IndChemist **32**, 271-76 (1956) & CA **50**, 15991 (1956) (Props & industrial applications) 8) CondChemDict (1961), 360 9) Sax (1963), 681-82

Dibutylperoxy-butane. See 2,2-Bis(t-butylperoxy)-butane in Vol 2 of Encycl, p B137-R

Dibutylperoxy-propane. See 2,2-Bis(t-butylperoxy)-propane in Vol 2, p B137-R

Dibutyl-o-phthalate (DBP or DBuPh) [called Phthalsäure-dibutylester or Dibutylphthalat in Ger], $C_6H_4[COO.(CH_2)_3.CH_3]_2$; mw 278.34, O 22.99%; col odorless, non-vol, non-toxic, stable, oily liq, fr p -35°, bp 206° at 20 mm, 210° at 29 mm & 340.7° at 763 mm pressure; d 1.047 at 20°, n_D 1.4900 at 20°, viscosity 0.203 poise at 20°; miscible with common org solvs; v sl sol in w; prepd by treating n-butyl alcohol with phthalic anhydride in the presence of H_2SO_4 (Ref 1)

Dibutylphthalate is used as a solv for aromatic nitrocompds, such as DNT & Dinitroethylbenzene. Silk (Ref 2) patented its use for the coating of NC & NG propellants to serve as a deterrant, solvent, plasticizer & stabilizer. It is added during the mixing process of the propellant colloid and replaces a portion of the volatile solvent, thus reducing the possibility of the loss of vol solv. It functions in the burning of propellants to cool the gases of combustion below their kindling point and thereby prevents muzzle flash (Ref 3)

Analytical Procedures. The detn of the Dibutylphthalate content of propellants is reported by a number of investigators (Refs 5, 6a,7, 8, 9, 10, 11, 12, 13, 14, 15 & 16a). Following are two methods used by US military installations in analysis of propellants:

A. Zinc Reduction-Volumetric Procedure (Ref 16a, Method 203.5). This method is used for determining the DBuPh and DEtPh content of proplnts that do not contain interfering esters such as other phthalates, dimethyl sebacate, triacetin, or sucrose octaacetate

Procedure. a) Prepare a specimen of proplnt as described in Method 509.3 of Ref 16a and extract a 5 g portion (weighed to within 0.2 mg) as described in method 104.13, using diethyl ether (JAN-E-199, Grade B) as solvent. These procedures are described also in Vol 2 of Encycl, p C131-L, under CENTRALITES

b) Evaporate the ether extract in the flask using a stream of dry air and add 6 g of Zn dust (ACS grade)

c) Add 20 ml of glacial acetic acid (JAN-A-465) around the sides of the flask and swirl to dissolve the dried residue. Add 15 ml of distd w while swirling, cover with a watch glass and heat to incipient boil at moderate heat. Set the switch of hot plate to OFF or to LOW

d) Remove the flask from the hot plate, lift and put aside the watch glass. Swirl the flask and add a small portion of Zn dust; swirl again and continue adding the Zn dust, with swirling, until the 6 g have been added

e) Cover with the above watch glass and place the flask on a partially cooled hot place. Heat gently for 5 mins with swirling until effervescence ceases. Be sure that the soln effervesces strongly during the 5-minute period

f) Remove the flask from the hot plate, cool to RT and wash down the watch glass

g) Direct a stream of w from a wash bottle to wash down any loose Zn on the walls of the flask, add 200 ml of w, swirl and allow to settle. Decant into a 500 ml separatory funnel, leaving the Zn in the flask (a little Zn in the funnel will do no harm)

h) Rinse the flask and Zn by swirling with 50 ml of w, allow to settle, and decant into the same separatory funnel. Repeat with another 50 ml of w

i) Add 60 ml of petroleum ether (ACS Grade, bp 30° to 60°) to the flask, swirl, allow to settle and decant into the separatory funnel. Repeat with another 60 ml of petr ether

j) Close the separatory funnel, shake it for a minute and allow the layers to separate

k) Drain the aqueous layer, with occasional swirling, into a 400 ml beaker and allow ca 0.5 ml of petr layer to follow the w into beaker

l) Transfer petr ether extract thru the top of separatory funnel into an alkali-resistant 300 ml Erlenmeyer flask with 24/40 ground glass joint (Corning Glass Works, Catalogue LG-1, Pyrex Laboratory Glassware 75000 or equivalent) while leaving ca 0.5 ml of petr eth in the funnel

m) Transfer contents of 400 ml beaker into the above funnel and rinse it twice with w into funnel

n) Add 60 ml of petr eth to the original (extraction) flask contg Zn and decant into the funnel. Repeat the extraction twice using new 60 ml portions of petr ether

o) Evaporate petr ether from 300 ml Erlenmeyer by blowing hot air and add 10 ml of 95% ethyl alcohol (MIL-E-463, Grade 1) directing it around the sides, with swirling

p) Add 3-4 drops of phenolphthalein indicator. Prepare it by dissolving 0.1 g of phpht (reagent grade) in 100 ml of 95% ethyl alcohol (MIL-E-463, Grade 1), neutralizing the acidity by adding 0.5N NaOH soln drop by drop until a pink color develops and then adding one drop of 0.5N HCl to discharge the pink color

q) Add alcoholic KOH soln (ca 0.45N) dropwise with a medicine pipet until the soln turns just pink, and then add 0.5N HCl soln dropwise until the pink color just disappears. Prepare alcoholic KOH soln by dissolving 30 g of ACS grade KOH (85%) pellets in a mixt of 600 ml ethyl alc and 50 ml w, filtering thru a Whatman No 41 filter paper, diluting to 1 liter with alc in a Pyrex bottle and covering it with a tight fitting rubber stopper

r) By means of a delivery pipet, add a volume of KOH soln calcd to be at least twice that required to saponify the DBuPh present

s) Reflux for 30 mins under water condenser and, if a ppt forms during heating, add a small amt of w thru the top of condenser
t) Stop refluxing, add 50 ml of w thru the top, disconnect the flask and cover it with a watch glass
u) After cooling the flask to RT, add 3-4 drops of phpht indicator soln and titrate rapidly with 0.1N HCl soln to the disappearance of pink color. Take the reading of sample titration (B)
v) For the blank, neutralize 10 ml of ethyl alcohol, as described in procedure q, and then follow procedures r, s, t and u. Take the reading of blank titration (A)
w) Calculate as follows:

$$\%\ DBuPh = [13.92(A-B)N]/W,$$

A=Ml of hydrochloric acid for blank titration
B=Ml of hydrochloric acid for sample titration
N=Normality of hydrochloric acid
W=Weight of sample in grams, corrected for total volatility

The same procedure is used for determination of Diethylphthalate (qv)

B. Difference Method (Ref 16a, Method 204,1.2)
a) Prepare and extract the 5 g sample as described in previous method, using methylene chloride for extraction
b) Evaporate the solvent and determine the total percentage of extraction matter in propellant on a volatile-free base
c) Determine the percentage of all methylene chloride extractive components in the propellant (except DBuPh), using appropriate method given,PA Tech Memo **GL-2-59**. Propelnt components sol in methylene chloride include triacetin, sucrose octaacetate, DNT, TNT, nitrate esters (such as NG, TEGN, PETN, BTTN, PETRIN), DPA, 2-NDPA, ethyl centrlite, ethylene dimethacrylate, and Candelilla wax
d) Subtract the sum of the determined percentages of all extractable matter (except DBuPh)(proc c) from the total percentage of extraction matter (proc b) to determine the percentage of DBuPh

Refs: 1) Beil **9**, [586] 2) C.E. Silk, USP 2335779(1943) & CA **38**, 3130(1944) 3) All&EnExpls(1946), 41-42 4) S. Berman et al, IEC **40**, 1312-19(1948)(Reaction rate of catalytic esterification of DBP) 5) S. Wachtell, PAChemLabRept **129161** (17 Jan 1950)(Determination of the DBP content of Solventless Powder, Composition 17: NC (12.6%N) 53.2, NG 36.6, DBP 8.0, 2-Nitrodiphenylamine 1.0 & Pb stearate 1.2% with carbon black 0.03% added) 6) L .Brissaud, MP **34**, 341-50(1952) & CA **49**, 2735(1955) (Prepn of spherical NC granules using DBP) 6a) L.J. Bellamy, JApplChem(London) **3**, 421-25(1953)(Detn of DBP in Cordite) 7) J. Tranchant, MP **35**, 308-16(1953) & CA **50**, 2173(1956)(Detection, distinction & detn of phthalic esters in proplnts) 8) C. Ribaudo, PATR **1939**(1953)(Detn in DBP coated IMR Propellant) 9) A.H. Castelli et al, PATR **2094**(1954)(Detn of DBP by IR in M1 & M6 Propellants) 10) C. Ribaudo, PATR **2119**(1955)(Polarographic detn of DBP in M18 Propellant) 11) M.A.C. Mullaly, Analyst **80**, 237-39(1955) & CA **49**, 7249(1955)(Detn of DBP in proplnts) 12) J. Sourd MP **38**, 371-82(1956) & CA **51**, 11719(1957)(Detn of DBP in proplnts) 13) G. Norwitz, AnalChimActa **19**, 216-23 (1958)(Detn of diethyl & DBP's in proplnts) 14) L. Marvillet & J. Tranchant, MP **41**, 179-82(1959)(Analysis of phthalic esters in proplnts) 15) L. Marvillet, MP **42**, 247-60 (1960) & CA **55**, 14917(1961)(Analysis of phthalic esters in proplnts) 16) CondChemDict(1961), 360 16a) MIL-STD-286A(Aug 1961) 17) Proplnt Manual SPIA/M2(1962), Unit Nos 280, 464, 523 & 640(Uses of DBuPh in rocket proplnts)(Conf) 18) Sax(1963), 683 19) US Specification **TT-D-3018** (Dibutylphthalate for use in org coatings) and Specification **MIL-D-218A** (Dibutylphthalate, technical)(See also Refs under Diethylphthalate)

Dibutylsebacate (called Sebacinsäure-dibutylester or Dibutylsebacinat in Ger), $C_4H_9OCO(CH_2)_8OCOC_4H_9$; mw 314.45, O 20.35%; clear col, odorless liq, fr p - 11°, bp 349°, d 0.936, n_D 1.4399 at 25°;obtd by reaction of sebacic or sebacylic acid with butyl alcohol & HCl in benz (Refs 1 , 2 & 3)

Hopper (Ref 2) detd the props of Dibutylsebacate and found it to be equal to DBP as a

gelatinizing agent for NC. It is currently used in some proplnts (Ref 4)
Refs: 1) Beil **2**, 719 & {1809} 2) J.D. Hopper, PATR **952** (1939) 3) CondChemDict (1962), 360 4) ProplntManual SP1A/M2 (1962), Unit No 462 (Conf) 5) Sax (1963), 683

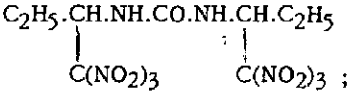
Dibutyltartrate (called L_g or d-Weinsäure-dibutylester or Dibutyl L_g-tartrat in Ger), $(CHOH.COO.C_4H_9)_2$; mw 230.30, O 27.79%; liq, fr p 21.8°, bp 320° at 765 mm of Hg, flash p 132°, d 1.0909 at 20°, n_D 1.4451 at 20°; viscosity at 18° 1.0587, at 40° 0.2532, at 80° 0.0455 g/cm sec; may be prepd by distilling tartaric acid with butyl alcohol

Dibutyltartrate is a solv & plasticizer for NC. DuPont (Ref 2) obtd a patent for the use of Dibutyltartrate as a constituent of proplnts which produce no flash from the gun barrel. An example of such a compn is: NC, DNT & ester of an org acid, such as dialkyl phthalate, dibutyltartrate or ethyl palmitate
Refs: 1) Beil **3**, 518, (178), [332] & {1021} 2) DuPont, GerP 567878 (1930) & CA **27**, 2814 (1933) 3) CondChemDict (1961), 361 4) Sax (1963), 683 5) US Specification-none

Dibutylurea and Derivatives

Dibutylurea (called Dibutyl-harnstoff in Ger), $C_9H_{20}N_2O$, mw 172.27, N 16.26%. Several isomers are described in Beil:
N,N'-Dibutylurea, $C_4H_9.HN.CO.NH.C_4H_9$; crysts (from eth & petr eth), mp 75°, bp 115° at 0.03 mm of Hg (Ref 1)
N,N-Dibutylurea, $(C_4H_9)_2N.CO.NH_2$; crysts, hygro, solidification point 22-25°, bp 118° at 2-3 mm; forms a Picrate salt, $C_9H_{20}N_2O + C_6H_3N_3O_7$, mp 82-83° (Ref 2)
N,N'-Di-iso-butylurea, $[(CH_3)_2CH.CH_2.NH]_2CO$; ndls (from dil alc), mp 134-36°; insol in cold w; readily sol in hot w, alc or eth (Ref 3)
N,N-Di-iso-butylurea, $[(CH_3)_2CH.CH_2]_2N.CO.NH_2$; crysts, mp 72-74°, bp 180° at 25 mm; readily sol in common org solvs; forms an Oxalate salt, $2C_9H_{20}N_2O + C_2H_4O_4$, prisms (from w), mp - dec 115° (Ref 4, p 170)
N,N'-Di-(d-sec-butyl) urea, $[CH_3.CH_2.CH(CH_3).NH]_2CO$; ndls; readily sol in alc; v sl sol in w (Ref 4, p 160)
N,N'-Di-(dl-sec-butyl)urea, $[CH_3.CH_2.CH(CH_3).NH]_2CO$; ndls (from dil alc), mp 137-38° (Ref 5)
N-Butyl-N'-(d-sec-butyl)urea, $CH_3.CH_2.CH(CH_3).NH.CO.NH.C_4H_9$; ndls, mp 47° (Ref 4, p 160)
N-(d-sec-Butyl)-N'-(dl-sec-butyl)urea, $[CH_3.CH_2.CH(CH_3).NH]_2CO$; crysts, mp 132° (Ref 4, p 162)
N-iso-Butyl-N'-tert-butyl-urea, $(CH_3)_3C.NH.CO.NH.CH_2.CH(CH_3)_2$; crysts, mp 163° (Ref 4, p 174)
N,N'-Di-tert-butylurea, $(CH_3)_3C.NH.CO.NH.C(CH_3)_3$; crysts (from alc), mp 243°, sublimes at 250°; sol in alc & eth; insol in w (Ref 6)
Refs: 1) Beil **4**, [634] & {299} 2) Beil **4**, (372), [635] & {301} 3) Beil **4**, 168 & {319} 4) Beil **4**, 160, 162, 170 & 174 5) Beil **4**, 162 & (372) 6) Beil **4**, 175 & {325}

Di(1,1,1-trinitro-iso-butyl)-urea or Bis(1,1,1-trinitro-iso-butyl)-urea,



$$\underset{C(NO_2)_3}{C_2H_5.CH}.NH.CO.NH.\underset{C(NO_2)_3}{CH}.C_2H_5$$

; mw 442.26, N 25.34%; wh solid, mp 110-12°; detonated with difficulty be impact; was obtd when urea in acet ac was added over a period of 15 mins to an acet ac soln of nitroform & propionaldehyde, and the reaction mixt heated for 20 mins and diluted with water
Ref: US Rubber Co Quarterly Progress Rept **14**, "Synthesis of New Explosives and Propellants"; Res & Develop Dept, Gen Labs, Passaic, NJ (16 May 1951) (Contract NORD 10129)

NOTE: No other nitrated derivs of Dibutylurea were found in Beil or CA thru 1961

Di-n-butyryl Peroxide (called Dibutyrylperoxyd or "Butyrylperoxyd" in Ger), $C_3H_7.CO.OO.CO.C_3H_7$; mw 174.19, O 36.74%; gas, crysts at -80°; Q_{form} in liq state at 25° -161.0 & in gaseous state at 25° -60 kcal/mol (Ref 3); $Q^{25°}_{comb}$ -4475.4 kg/mol (liq): 1 cal=

4.1840 joules (Ref 3); E (energy of activation) for decompn is 29.6 kcal/mol (Ref 2); obtd by reaction of Na_2O_2 & butyryl chloride in eth at 0°, or butyric anhydride in petr eth at -15° (Ref 1)

Refs: 1) Beil **2**, 274 & {614} 2) A. Reinbaum & M Szwarc, JChemPhys **23**, 912 (1955) & CA **49**, 14448 (1955) 3) L. Jaffe et al, JChemPhys **27**, 416-19 (1957) & CA **52**, 71-72 (1958)

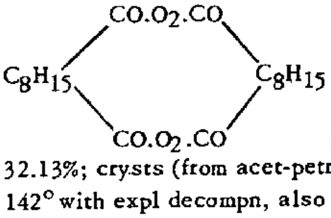
Di-iso-butyryl Peroxide (called Diisobutyrylperoxyd in Ger), $(CH_3)_2CH.CO.OO.CO.CH(CH_3)_2$; mw 174.19, O 36.74%; gas, dec vigorously at 110-20° (20° higher than the n-butyryl deriv) when heated in a tube (Ref 2); was prepd by reacting isobutyryl chloride with Na peroxide in cold eth or petr eth (Ref 1). Smid & Szwarc (Ref 3) studied its kinetics of decompn

Refs: 1) Beil **2**, {653} 2) A.E. Oxford, JCS **1942**, 578 3) J. Smid & M Szwarc, "Kinetics of Decompostion of Iso-buryrl Peroxide and Reactions of Iso-propyl Radicals", Syracuse-UnivRept (1958) (Contract DA-30-115-ORD-678)

Dicadmium-acetato-perchlorate-acetate, $[Cd_2(CH_3COO)_2](CH_3COO).ClO_4.3H_2O$; mw 555.4, ClO_4 17.9%; col ndls, mp - deflgr violently on heating; was obtd when a soln of Cd acetate in $HClO_4$ was allowed to evap slowly in the presence of CH_3COOH

Refs: 1) Beil-not found 2) R. Weinland & H. Schlaich, ZAnorgChem **150**, 41 (1925) & CA **20**, 720 (1926)

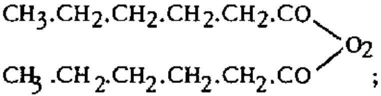
Dicamphoryl Peroxide,



; mw 398.44, O 32.13%; crysts (from acet-petr eth 4/1), mp 142° with expl decompn, also explodes when heated in a flame; sol in acet, chlf & eth; sl sol in benz & petr eth; v sl sol in water; was prepd by adding slowly, with vigorous stirring, chopped ice & Na_2O_2 to an ethereal soln of d-camphoric anhydride at 0°. Compare with d-Camphoric Acid Peracid in Vol 2 of Encycl, p C23-R

Refs: 1) Beil-not found 2) N.A. Milas & A. McAlvey, JACS **55**, 350 (1933) & CA **27**, 972 (1933)

Di-n-caproyl Peroxide or Bis(n-caproyl)-peroxide (called Di-n-caproylperoxyd in Ger),



$CH_3.CH_2.CH_2.CH_2.CH_2.CO\diagdown O_2 \diagup CO.CH_2.CH_2.CH_2.CH_2.CH_3$

; mw 230.30, O 27.79%; oil having a sl unpleasant odor, fr p - does not freeze, bp - dec 64-65° & deflgr at 84-85°; readily sol in alc, eth & petr eth; insol in w; was prepd by treating an ethereal sol of n-caproic anhydride with BaO_2 in the presence of a little water

Refs: 1) Beil **2**, [286] 2) F. Fichter & R. Zumbrunn, Helv **10**, 870 (1927) & CA **22**, 1136 (1928) 3) F. Fichter, Helv **12**, 571 (1929) & CA **23**, 3410 (1929) 4) Tobolsky & Mesrobian (1954), 179

Dicarbamylamine. See Biuret in Vol 2, p B164-L

N,N'-Dicarboethoxy-1,3-diamino-isopropanol or N,N'-Isopropanol Diurethane

$(C_2H_5OOC)HN.CH_2\text{-}CH(OH)\text{-}CH_2NH(COOC_2H_5)$ and its nitrated derivative, $(C_2H_5OOC)(O_2N)N.CH_2\text{-}CH(ONO_2)\text{-}CH_2.N(NO_2)(COOC_2H_5)$, serve as intermediates in the prepn of Dinitramino-iso-propanol Nitrate, described under Diaminohydroxypropane and Derivatives

Dicarboethoxyethylenediamine and Derivatives

N,N'-Dicarboethoxyethylenediamine; 1,4-Dicarboxy-1,4-diazabutane or Ethylenediurethane (called N.N'-Dicarbäthoxyäthylendiamin; Äthylen-bis-carbamidsäureäthylester; or Äthylendiurethan in Ger), $C_2H_5.OOC.NH.CH_2.CH_2.NH.COO.C_2H_5$; mw 204.22, N 13.72%; ndls (from w), mp 112° or prisms (from alc), mp 113°; readily sol in alc or eth; sl sol in w; obtd by heating ethylenediisocyanate with abs alc, or by reaction of ethylenediamine & chloroformic acid ethyl ester at 0°

Ref: Beil 4, 254, [693] & {532}

N,N -Dinitro-N,N'-dicarboethoxyethylenediamine; 1,4-Dinitro-1,4-dicarboxy-1,4-diazabutane or Ethylene-bis-nitrourethane (called N.N'-Dinitro-N.N'-dicarbäthoxy-äthylendiamin or N.N'-Dinitro-äthylendiurethan in Ger), $C_2H_5OOC.N(NO_2).CH_2.CH_2.N(NO_2).COOC_2H_5$; mw 294.22, N 19.04%; fine ndls (from w), mp 83-84°; sol in chlf & benz; sl sol in alc; almost insol in cold w; prepd by nitrating ethylenediurethane with the strongest concd HNO_3

Ref: Beil 4, 257 & {547}

4,7-Dicarbethoxy-1,4,7,10-tetrazadecane. See Bis(aminoethyl)-ethanediurethane in Vol 2, p B129-R

Dicarbomethoxyethylenediamine and Derivatives

N,N'-Dicarbomethoxyethylenediamine [called N.N'-Dicarbomethoxy-äthylendiamin; Äthylendiurethylan or α,β-Bis(carbomethoxy-amino)-äthan in Ger], $CH_3.OOC.NH.CH_2.CH_2.NH.COOCH_3$; mw 176.17, N 15.90%; crysts (from MeOH), mp 133.5°; sol in hot w, alc & chlf; less sol in eth & benz; was prepd by reaction of 1 mol ethylenediamine with 2 mols of the dimethyl ester of carbonic acid at 100°

Ref: Beil 4, 254 & (416)

N,N'-Dinitro-N,N'-dicarbomethoxyethylenediamine (called N.N-Dinitro-N.N'-dicarbomethoxy-äthylendiamin or N.N'-Dinitro-äthylendiurethylan in Ger), $CH_3OOC.N(NO_2).CH_2.CH_2.N(NO_2).COOCH_3$; mw 266.17, N 21.05%; ndls (from w or MeOH), mp 132-33° (dec); readily sol in benz or chlf; sol in alc; sl sol in petr eth or alc; almost insol in cold w; was prepd by nitrating the parent compd with the highest concn of HNO_3. Its expl props were not investigated

Refs: 1) Beil 4, 257 & (417) 2) H.J. Backer, Rec **31**, 170 (1912) & JChemSoc **102** I, 730 (1912)

Dicarboxamide-acetylene. See Acetylenedicarboxamide in Vol 1, p A65-L

Dicarboxanilide-acetylene; Di(N-phenylcarboxamide)-acetylene or Bis(carboxanilide)-acetylene, $C_6H_5HNOC.C{\equiv}C.CONHC_6H_5$; mw 264.27, N 10.60%; pinkish-white crysts (from MeOH-water), mp 194-96°; was prepd from acetylenedicarboxylic acid by a series of reactions to give bromofumaranilide which was dehydrohalogenated by a dil soln of methanolic KOH.

Its hexanitro deriv, **N,N'-Di(2,4,6-trinitrophenyl)acetylene-dicarboxamide,** $(NO_2)_3C_6H_2HNOC.C{\equiv}C.CONHC_6H_2(NO_2)_3$; mw 534.27, N 20.97%; wh crysts, mp 272-80°; was prepd by nitrating the parent compd. No expl props were reported

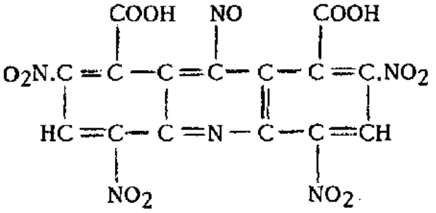
Ref: ADL, Synthesis HE's, 2nd Rept (1951), pp 142-43 & 153-54

Dicarboxylic Acid Halogen Amides, Their Salts and Use in Cross-Linking of Cellulose Fibers Thru Urethane Formation. This problem was investigated by Eckert et al (Ref), who isolated some expl products. For example, *N,N,N',N'-Tetrachloroadipamide or N,N,N',N -Tetrachlorohexane-diamide,* $Cl_2NOC(CH_2)_4CONCl_2$, obtd by reacting adipic amide & aq $NaHCO_3$ at 0° with Cl gas for 2hrs, dissolves in boiling water with degradation, giving a highly expl oil (di- or trichloroamine). The *Sodium salt* of adipic acid dibromoamide decompd at 30-32°

Ref: P. Eckert et al, Reyon, SyntheticaZellwolle **29**, 2-8, 89-99 (1951) & CA **45**, 7527, 8761 (1951)

Dicarboxy-phenazine-N-oxide, $C_{14}H_4N_6O_3$, may be assumed to be the parent compd of its tetranitro deriv although it was not used to prep it:

1,9-Dicarboxy-2,4,6,8-tetranitrophenazine N-oxide (also called "White Compound") $C_{14}H_4N_6O_{13}$; mw 464.22, N 18.11%;



assumed structure, since it was not definitely established; wh expl compd; obtd in small quantity as one of the by-products in the manuf of TNT. This compd should not be confused with the White German Powder or Augendre Powder described in Vol 1, p A507-L

Ref: British Advisory Council Rept **AC-3775/HE I**

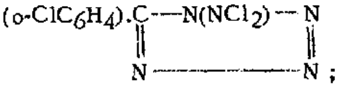
***a,a'*-Dicarboxypimelyl Tetraazide** (called *a,a'*-Dicarboxypimelinsäure Tetraazid in Ger), $(N_3.OC)_2CH.(CH_2)_3.CH(CO.N_3)_2$; mw 348.25, N 48.27%; wh powd (from eth), explodes by friction, impact or rapid heating; melts under benz at 50-55°; stable in dry state; readily sol in abs alc; less sol in eth, benz or chlf; insol in water; was prepd by reaction of the tetrahydrazide with HCl

Refs: 1) Beil **2**, (334) 2) T. Curtius, JPraktChem **94**, 346(1916)

Dicarboxypropionyl-peroxide. See Bis(succinyl)-peroxide in Vol 2 of Encycl, p B156-R

Dichloralperoxydhydrat. A Ger name for the compd Bis(*a*-hydroxy-*β,β,β*-trichloroethyl)-peroxide described in Vol 2, p B148-R

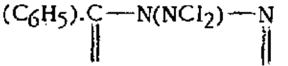
1,1-Dichloramino-5-(o-chlorophenyl)-*a*-tetrazole or 1,1-Dichloramino-5-(2'-chlorophenyl)-1H-tetrazole,



$(o\text{-}ClC_6H_4).C{=}N{=}N \;\; N(NCl_2)\text{—}N{=}N$; mw 264.52, N 26.47%; granular solid (from alc), exploded violently when dried by spreading and rubbing on a porous porcelain plate: was prepd by treating 1-amino-5(o-chlorophenyl-*a*-tetrazole (qv) with hypochlorous acid

Refs: 1) Beil-not found 2) R. Stollé et al, JPraktChem **138**, 2 & 10(1933) & CA **27**, 4798(1933)

1,1-Dichloramino-5-phenyl-*a*-tetrazole or 1,1-Dichloramino-5-phenyl-1H-tetrazole,



$(C_6H_5).C\text{—}N(NCl_2)\text{—}N$; mw 230.1, N 30.44%; OB to CO_2 -147.3%; solid, extremely violent and sensitive expl; was prepd by treating 1-amino-5-phenyl-*a*-tetrazole with hypochlorous acid

Refs: 1) Beil-not found 2) R. Stolle et al, JPraktChem **138**, 2 & 4-5(1933) & CA **27**, 4798(1933)

1,1-(N-Dichloramino)-5-(p-tolyl)-*a*-tetrazole. See in Vol 1, p A266-R

Dichloroacetylene or Dichloroethyne (called Dichloracetylen or Dichloräthin in Ger), Cl.C⁚C.Cl; mw 94.93; oil, fr p -64 to -68°, bp 32-33° at 748 mm press, forms ndls at temps below its fr p; explodes on contact with air; d 1.261 at 20°; n_D 1.4279 at 20°; miscible with many org solvs; can be stored at RT in ether soln, with which it forms an addn compd. Dichloroacetylene is narcotic & poisonous, causing nerve & eye symptoms

Dichloroacetylene can be formed by bubbling acetylene into an ice-cold soln of K hypochlorite; on treating either trichloro ethylene or tetrachloroethane with KOH; by heating Ba trichloroacrylate or Ba chloropropiolate; by the IGFarbenind continuous process using acetylene diluted with N & a strongly alkaline hypochlorite soln; and by the recently patented (Ref 4) process of reacting phosgene with hydroxyacetylene, in the presence of substituted amides or lactams

Refs: 1) Beil **1**, 245, (106), [222] & {918} 2) E. Ott, Ber **75B**, 1517-22(1942) & CA **38**, 1468(1944) 3) Kirk & Othmer **3**(1949), 799-800 4) H. Pasendach & R. Fischer, assigned to Badische Aniline-und Soda-Fabrik AG, GerP 1135893(1962) & CA **58**, 7829(1963) 5) Sax(1963), 687 6) Kirk & Othmer, 2nd ed **5**(1964), 204-05

Dichloroacetyl Peroxide or Chloroacetyl Peroxide (called Bis-chloracetyl-peroxyd in Ger), $ClCH_2.CO.OO.CO.CH_2Cl$; mw 186.98; small hygroscopic ndls, mp 35-36°, dec > 70°; explodes violently on impact, friction, or when mixed with Al, Pb peroxide, S or $KClO_3$; readily sol in most solvs, except petr eth; can be prepd from chloroacetyl chloride & 3% H_2O_2 in the presence of Na acetate under cooling and shaking of the mixt, and from chloroacetylchloride & Na_2O_2 in water at 0°, or 3% H_2O_2 in pyrindine in the cold

Refs: 1) Beil **2**, 199 & {449} 2) L. Vanino & E. Uhfelder, Ber **33**, 1043(1900) & JCS **78** I, 371(1900) 3) Tobolsky & Mesrobian (1954), 169

N,N-Dichloroalkylamines or Alkyldichloroamines, $RNCl_2$ were patented by Riedel(Ref) for use as liq expls. They can be detonated by MF or other initiators. Kieselguhr or other similar material can be used as an absorbent for the liq dichloroalkylamine

See also N,N-Dichloroethylamine and others

Ref: J.D. Riedel, GerP 301799(1914) & CA **15**, 1996(1921)

1,1-Dichloroamino-5-(o-chlorophenyl)-α-tetrazole. See 1,1-Dichloramino-5-(o-chlorophenyl)-α-tetrazole

1,1-Dichloroamino-5-phenyl-α-tetrazole. See 1,1-Dichloramino-5-phenyl-α-tetrazole

1,1-(N-Dichloroamino)-5-(p-tolyl)-α(or 1H)-tetrazole. See 1,1-(N-Dichloramino)-5-(p-tolyl)-α(or 1H)-tetrazole in Vol 1, p A266-R

Dichloroanilino-ethane or Dichlorophenylaminoethane. See Bis(chloroanilino)-ethane in Vol 2, p B138-L

Dichloroanilino-propane or Dichlorophenylamino-propane. See Bis(chloroanilino)-propane in Vol 2, p B138-R

Dichloroazidobenzene. See under Dichlorobenzene

Dichloroazobenzene and Derivatives

Dichloroazobenzene (called Dichlor-azobenzol in Ger), $Cl.C_6H_4.N:N.C_6H_4Cl$, mw 251.11, N 11.16%. Its *2,2'-Dichloro-* orn ndls (from petr eth or alc), mp 136-37°; *3-3'-Dichloro-* orn-red ndls (from alc,) mp 101°; *4,4'-Dichloroderivs,* lt-yel ndls (from acet), mp 188° (Ref 1); and *2,4-Dichloroazobenzene,* $Cl_2C_6H_3.N:N.C_6H_5$, orn crysts (from alc), mp 105°(Ref 2) are described in the literature

Refs: 1) Beil **16**, 35, 36, (222, 223) & [12, 13] 2) Beil **16**, (223)

Mononitrodichloroazobenzene, $C_{12}H_7Cl_2N_3O_2$; mw 266.10, N 15.79%. Two isomers are found in Beil

2'-Nitro-2,4-dichloroazobenzene, $Cl_2C_6H_3.N:N.C_6H_4.NO_2$; dk-salmon colored ndls (from alc), mp 155.5°(Ref, p 51); and *2 (or 3)-Nitro-4,4'-dichloroazobenzene,* $(O_2N)Cl.C_6H_3.N:N.C_6H_4.Cl$; pale-yel crysts, mp 210°(Ref, p 58)

Ref: Beil **16**, 51, 58

Dinitrodichloroazobenzene, $(O_2N)Cl.C_6H_3.N:N.C_6H_3Cl(NO_2)$; mw 337.11, N 16.62%. Three isomers are found in the literature:

4,4'-Dinitro-2,2'-dichloroazobenzene, brn-red prisms (from solv naphtha), mp 274°(Ref 1) or red ndls (from benz), mp 265°(Ref 3), mp 275-76°(Ref 4); readily sol in benz; mod sol in glac acet acid; sl sol in acet; insol in alc; was prepd by oxidization of 4-nitro-2-chloroaniline with alk Na hypochlorite soln (Ref 1) and in 87% yield by oxidation of 4-nitro-2-chloroaniline with iodosobenzene diacetate at 35°(Ref 5)

2,2'-Dinitro-3,3'-dichloroazobenzene, orn prisms (from benz), mp 266.5-67.2°; obtd by oxidizing 2-nitro-3-chloroaniline in dry benzene with iodosobenzene diacetate (Ref 5)

2,2'-Dinitro-6,6'-dichloroazobenzene, orn ndls (from aq alc), mp 158.5°-59°; prepd by oxidizing 2-nitro-6-chloroaniline with $Pb(OAc)_4$ in benz (Ref 5)

Refs: 1) Beil **16**, (227) 2) A.H. Cook & D.G. Jones, JChemSoc **1939**, 1314 & CA **34**, 85(1940) 3) G.B. Barlin et al, JChemSoc **1954**, 3123 & CA **49**, 11608(1955) 4) P.S. Raman, ProcIndianAcadSci **45A**, 65(1957) & CA **51**, 13795(1957) 5) L.K. Dyall & K.H. Pausacker, AustralianJChem **11**, 491 (1958) & CA **53**, 7147-48(1953)

Tetranitrodichloroazobenzene, $(O_2N)_2Cl.C_6H_2.N:N.C_6H_2Cl(NO_2)_2$; mw 431.11, N 19.50%. Two isomers are known :

2,4,2',4'-Tetranitro-5,5'-dichloroazobenzene, yel crysts (from 50% acet ac) turning greenish gradually on exposure to light, mp 93°, explodes on further heating; readily sol in alc, eth, petr eth, glac acet ac & benz; was obtd on warming *a*-nitroso-5-chloro-2,4-dinitrophenylhydrazine with 50% acetic acid (Refs 2 & 4)

2,4,2',4'-Tetranitro-6,6'-dichloroazobenzene, orn ndls (from boiling chlorobenz + alc), mp 244-45°; was obtd when 2,4,6,2',4',6'-hexanitroazobenzene was heated with alc HCl in a sealed tube at 130-50° for 1½ hrs (Refs 1 & 3). Its expl props were not reported

Refs: 1) Beil **16**, 59 2) Beil **16**, [18] 3) H. Leemann & E. Grandmougin, Ber 41, 1298(1908) & JChemSoc **94** I, 479(1908) 4) E. Müller & G. Zimmermann, JPraktChem **111**, 290(1925) & CA **20**, 750(1926)

NOTE: Higher nitrated derivs of Dichloroazobenzene were not found in Beil or in CA thru 1961

N,N'-Dichloroazodicarboxamidine. See *a,a*-Azobis(chloroformamidine) in Vol 1, p A652-R

Its salts, especially those of silver & mercury, are expls very sensitive to heat, easily ignited by a low electric current and, hence, are suitable for use in elec blasting cap compns with Nitrostarch as a gelatinizer (Refs 1 & 2)

Mercury salt, flocculent red ppt; was prepd by adding while stirring an aq soln of Hg acetate (1 mol) to a hot aq soln of N,N'-dichloroazodicarboxamidine with NH_3 added from time to time to keep the mixt just sl acid at the start of the reaction & sl alkaline towards the end

Silver salt, black ppt; was prepd by adding while stirring an aq soln of ammoniacal $AgNO_3$ (2 mols) to a hot aq soln of N,N'-dichloroazodicarboxamidine. A dark red ppt forms immediately, but it turns black on continued stirring at 90°. In order to decrease the sensitiveness to friction, the salt may be coprecipitated with materials such as dextrin

Refs: 1) L.A. Burrows, USP 2086533(1937) & CA **31**, 6467(1937) 2) Blatt, OSRD **2014** (1944)

Dichloroazoxybenzene and Derivatives

Dichloroazoxybenzene (called Dichlor-azoxybenzol in Ger), $Cl.C_6H_4.N(:O):N.C_6H_4Cl$; mw 267.11, N 10.49%. Its *2,2'-Dichloro-*, lt-yel nds (from alc), mp 56°; *3,3'-Dichloro-*, yel ndls (from alc), mp 94-97° and *4,4'-Dichloro-* derivs, lt yel ndls (from alc), mp 157-58° are described in Beil. Other props & their methods of prepn are given in the Ref

Ref: Beil **16**, 625, (376) & [314, 315]

Mononitrodichloroazoxybenzene, $Cl.C_6H_4.N(:O):N.C_6H_3(NO_2)Cl$; mw 312.10, N 13.46%. Several isomers are known, all of which are yel ndls, such as:

2-Nitro-3,3'-dichloro-, mp 112° (Ref 3) & 116° (Ref 2); *4-Nitro-3,3'-dichloro*, mp 145° (Ref 2); *5-Nitro-3,3'-dichloro-*, mp 105° (Ref 2); *6-Nitro-3,3'-dichloro-*, mp 116° (Ref 2); and *2-Nitro-4,4'-dichloro-*, mp 137° ((Refs 1, 3 & 4) . Some of their reduction products (amines) are extremely expl, especially that of the 2-Nitro-3,3'-dichloro deriv by Na arsenite reduction (Ref 3)

Refs: 1) Beil **16**, 628 2) H.E. Bigelow & W.H. Steeves, CanJRes **17B**, 161(1939) & CA **33**, 8583(1939) 3) H.E. Bigelow & K.F. Keirstead, CanJRes **24B**, 234(1946) & CA **41**, 406(1947) 4) C.D. Houghton & W.A. Waters, JChemSoc **1950**, 1018 & CA **44**, 7256(1950)

Dinitrodichloroazoxybenzene, $Cl(O_2N)C_6H_3.N(:O):N.C_6H_3(NO_2)Cl$; mw 357.11, N 15.69%. Only two isomers are known: *5,5 -Dinitro-2,2'-dichloro-*, reddish-yel ndls (from alc), mp 163-64° (Refs 1 & 3); and *3,4-Dinitro-3,3'-dichloro-azoxybenzene,* mp 157° (dec) (Ref 2). Other props & methods of prepn are given in the Refs

Refs: 1) Beil **16**, (378) 2) H.E. Bigelow & K.F. Keirstead, CanJRes **24B**, 235 (1946) & CA **41**, 406 (1947) 3) P.E. Gagnon et al, CanJChem **35**, 1304, 1318 (1957) & CA **52**, 6225 (1958)

Trinitrodichloroazoxybenzene, $Cl(O_2N)_2C_6H_2.N(:O):N.C_6H_3(NO_2)Cl$; mw 402.11, N 17.42%. Three isomers are known: *Trinitro-3,3'-dichloroazoxybenzene,* crysts, mp 182° (Refs 1 & 2)

2,4,6-Trinitro-3,3'-dichloroazoxybenzene, crysts, mp 165° (Refs 1 & 2)

Trinitro-4,4'-dichloroazoxybenzene, yel crysts (from glac acet ac), mp 186-87° (Ref 3) & 189° (dec rapidly at 193°) (Ref 2). Trinitro derivs are prepd by nitrating the dichloro deriv. Their expl props were not reported

Refs: 1) H.E. Bigelow & W.H. Steeves, CanJRes **17B**, 161, 164 (1939) & CA **33**, 8583 (1939) 2) H.E. Bigelow & K.F. Keirstead, CanJRes **24B**, 234, 235 (1946) & CA **41**, 406 (1947) 3) R.C. Fuson & S. Melamed, JOC **13**, 694 (1948) & CA **43**, 3802 (1949)

3,5,3',5'-Tetranitro-4,4'-dichloroazoxybenzene, $Cl(O_2N)_2C_6H_2.N(:O):N.C_6H_2(NO_2)_2Cl$; mw 447.11, N 18.80%; crysts, mp 226-28°; was obtd by treating 3,3',5,5'-tetranitro-4,4'-dihydroxy-azoxybenzene (mp 186-87°) with p-toluene-sulfonyl chloride (Refs 1& 2). The expl props of this compd were not reported

Refs: 1) T. Urbański & J. Urbański, BullAcadPolonSci, SerSci, ChimGeol et Geograph **6**, 307-08 (1958) (in English) & CA **52**, 19994 (1958) 2) T. Urbański & J. Urbański, RocznikiChem **33**, 693-702 (1959) (English summary) & CA **54**, 2222 (1960)

NOTE: Higher nitrated derivs of Dichloroazoxy benzene were not found in Beil or in CA thru 1961

DICHLOROBENZENE AND DERIVATIVES

Dichlorobenzene (called Dichlor-benzol in Ger), $C_6H_4Cl_2$; mw 147.01. This compd exists as ortho, meta & para derivs. A mixt of these isomers is obtd when benzene vapor & chlorine gas, preheated to 400-500°, are passed thru a reaction chamber at 650-700° (Ref 4):

o(or 1,2)-Dichlorobenzene, col liq, fr p -17°, bp 180.5°, flash p 155°F (Ref 5); d 1.3003 at 25°, n_r 1.5501 at 20° (Ref 1)

m(or 1,3)-Dichlorobenzene, liq, fr p -24.8°, bp 173°, d 1.2828 at 25°, n_D 1.5464 at 20° (Ref 2)

p(or 1,4)-Dichlorobenzene, white crysts, exists in two modifications: α-monoclinic, mp 53.5° and β-triclinic, mp 54° which is interconvertible with α-form at 30.8°, bp 174.1°, flash point (cc) 65° or 155°F (Ref 3), d 1.5039 at 25° (Ref 3)

Prolonged exposure to high concentrations of Dichlorobenzenes, can produce paralysis of the central nervous system, and also damage the liver. These compds have local irritant effects on the skin. The recommended MAC of o-dichlorobenzene is 50ppm (by vol). Long exposure to the p-deriv is reported capable of producing cataracts (Ref 6)

Other props & methods of prepn are given in Beil

Refs: 1) Beil **5**, 201, (111), [153] & {537} 2) Beil **5**, 202, (111), [154] & {541} 3) Beil **5**, 203, (111), [154] & {542} 4) T. Wheeler, BritP 388818 (1931) & BritishAbstract **52B**, 521 (1933) 5) Sax (1963), 687-88 6) Kirk & Othmer, 2nd edit **5** (1964), 258-67

Azido & Diazo Derivatives Dichlorobenzene

1-Azido-2,4-dichlorobenzene. See Vol 2, p B44-R

1-Azido-2,5-dichlorobenzene, $N_3.C_6H_3Cl_2$; mw 188.01, N 22.34%; yel crysts, mp 30°; readily sol in common org solvs; was prepd by reaction of aq ammonia with a paste of 2,5-dichlorobenzenediazonium tetrachloroiodide & crushed ice (Ref 1, p [208] & Ref 3)

1-Diazo-2,5-dichlorobenzene or 1-Diazonium-hydroxide-2,5-dichlorobenzene, $Cl_2C_6H_3.N\vdots N).OH$; known only in the form of its salts: *Tribromide,* $Cl_2C_6H_3.N_2.Br_3$, yel plts (from acet + eth), mp 160°(dec), deflgr on heating on Pt foil; insol in w & eth (Ref 2)

1-Azido-2-nitro-4,5-dichlorobenzene, $Cl_2C_6H_2.(NO_2).N_3$; mw 233.02, N 24.05%; yel-brn ndls (from alc), mp 56°; sol in benz, alc, glac acet ac & chlf; insol in w & petr eth (Ref 1, p 209 & Ref 4)

Refs: 1) Beil **5**, [208, 209] 2) Beil **16**, 467 3) F.D. Chattaway et al, JChemSoc **125** II, 1988(1924) 4) E. Müller & W. Hoffmann, JPraktChem **111**, 299(1925) & CA **20**, 750(1926)

Nitroso & Nitro Derivatives of Dichlorobenzene

1-Nitroso-dichlorobenzene, $Cl_2C_6H_3.NO$; mw 176.00, N 7.96%. Three isomers are described in Beil: *1-Nitroso-2,4-dichloro-*, not obtd pure; *1-Nitroso-2,5-dichloro-*, col crysts, mp 101°; and *1-Nitroso-3,4-dichlorobenzene*, mp 88°, dec on heating above it mp (Ref)

Ref: Beil **5**, {589}

Mononitrodichlorobenzene, $Cl_2C_6H_3NO_2$; mw 192.01, N 7.30%. Several isomers are known: *1-Nitro-2,3-dichloro-*, mp 61°, bp 257°; *1-Nitro-2,4-dichloro-*, mp 34°, bp 132-33° at 13-14 mm press; *1-Nitro-2,5-dichloro-*, mp 55°; *1-Nitro-2,6-dichloro-*, mp 71-72°, bp 105-07° at 3 mm press; *1-Nitro-3,4-dichloro-*, mp 43° bp 188.5-89° at 100 mm press; and *1-Nitro-3,5-dichlorobenzene*, mp 65°, d 1.400 at 100° (Ref). None of these compds is expl

Ref: Beil **5**, 245, 246, (130, 131), [185, 186] & {615, 616, 617}

Dinitrodichlorobenzene, $Cl_2C_6H_2(NO_2)_2$; mw 237.00, N 11.82%. All possible isomers are known, some of which may be of interest in the expl industry:

1,2-Dinitro-3,4-dichlorobenzene, ndls (from petr eth), mp 97°; can be prepd by diazotizing with $NaNO_2$ 2-nitro-5,6-dichloroaniline (Ref 1)

1,2-Dinitro-3,5-dichlorobenzene, crysts (from alc + benz), mp 95-98°; obtd by nitrating with mixed acids 1-nitro-3,5-dichlorobenzene (Ref 2)

1,2-Dinitro-3,6-dichlorobenzene, ndls (from alc), mp 101-03°, bp 318°(dec), d 1,6945 at 16°; can be prepd by diazotizing 2-nitro-3,6-dichloroaniline (Ref 3); an expl compd (Ref 13)

1,2-Dinitro-4,5-dichlorobenzene, crysts (from dil acetic acid), mp107° or plts (from alc), mp 109-10°; prepd by nitrating with mixed acids at 100° 1-nitro-3,4-dichlorobenzene (Ref 3)

1,3-Dinitro-2,4-dichlorobenzene, ndls or prisms (from alc), mp 70-71°; prepd by nitrating 1-nitro-2,6-dichlorobenzene (Ref 4)

1,3-Dinitro-2,5-dichlorobenzene, lfts (from alc) or monoclinic prisms (from CCl_4), mp 104-05°, bp 302°(dec), d 1,7103 at 16°; was prepd by nitrating 1-nitro-2,5-dichlorobenzene (Ref 5); an expl compd proposed for use in mixts with PA

1,3-Dinitro-4,5-dichlorobenzene, listed in Ref 16 as 1,2-Dichloro-3,5-dinitrobenzene, grn-yel crysts (from alc), mp 56°; was obtd together with 1,2-dinitro-3,4-dichlorobenzene by nitrating 1-nitro-2,3-dichlorobenzene with mixed acids (Ref 6); an expl 77% as powerful as PA & 85% as powerful as TNT (by Trauzl test) and less sensitive to impact than TNT or PA (120% PA by Brit FI test)

1,3-Dinitro-4,6-dichlorobenzene, listed in Ref 16 as 1,3-Dichloro-4,6-dinitrobenzene, yel ndls (from alc), mp 103-04°; 102°(in Ref 16); can be prepd by nitrating 1,3-dichlorobenzene with KNO_3 & concd H_2SO_4 at 120-40° (Ref 7); an expl 85% as powerful as TNT (by Ballistic Mortar test) and less sensitive to impact than TNT or PA (127% PA by Brit FI test)

1,4-Dinitro-2,3-dichlorobenzene, no props given (Ref 8)

1,4-Dinitro-2,5-dichlorobenzene, citron-yel crysts (from chlf) or lt-yel ndls (from alc), mp 117.5-19°, bp 304°; obtd with other isomers on nitrating 1,4-dichlorobenzene (Ref 9)

1,4-Dinitro-2,6-dichlorobenzene, ndls or prisms (from alc), mp 114°; was prepd by diazotizing 4-nitro-2,6-dichloroaniline with $NaNO_2$ soln at 0°(Ref 10)

Commercial Dinitrodichlorobenzene or Parazol. A mixt of dinitrodichlorobenzene isomers, mp 65-85°, d 1.694 (cast). Its expl props were summarized by Blatt (Ref 16) and they are as follows:

Brisance by Pb block compression test, 70% of TNT

Explosion temperature no ignition up to 340°

Impact sensitivity - less sensitive than TNT

Power by Trauzl test, 86% of TNT

Sensitivity to initiation, requires a powerful detonator when confined

Thermal stability by 65.5° Heat test, 60 min; by 135° German test, 120 min

Its toxicity is similar to that of TNT but the coml product is more poisonous than TNT, causing severe dermatitis. Vapors of the product are also very toxic (Refs 16 &17)

Uses. It was used in Germany as an expl filler during WWI. When mixed with TNT, it detonates only partially forming a very fine dust which causes itching of the skin. The gases of expln contain phosgene ($COCl_2$) and are very poisonous. Babcock (Ref 12) proposed for military use an expl filler composed of a dinitro-p- & dinitro-o-dichlorobenzene with or w/o Picric Acid, thus producing a mixt castable below 100°

Refs: 1) Beil **5**, [197] & {637} 2) Beil **5**, 264, [198] &{637} 3) Beil **5**, 265, [198] & {638} 4) Beil **5**, 265 & [198] 5) Beil **5**, 265, (138), [198] & {638} 6) Beil **5**, (138), [198] & {638} 7) Beil **5**, 265, (138), [199] & {638} 8) Beil **5**, [199] 9) Beil **5**, 265 & [199] 10) Beil **5**, (138), [199] & {639} 11) W.C. Cope, IEC **12**, 870 (1920) 12) J.H. Babcock, USP 1366048 (1921) & CA **15**, 947 (1921) 13) Marshall **3** (1932), 72, 238 14) L. Wöhler & O. Wenzelberg, ZAngChem **46**, 173 (1933) & CA **27**, 2579 (1933) 15) W.F. Von Oettingen, US Public HealthServBull **271** (1941), 105 16) Blatt, OSRD **2014** (1944) 17) Sax (1963), 755

Trinitrodichlorobenzene, $Cl_2C_6H(NO_2)_3$; mw 282.01, N 17.04%. Only one isomer is known:

1,3,5-Trinitro-2,4-dichlorobenzene or 2,4,6-Trinitro-1,3-dichlorobenzene, col prisms (from alc), mp 129°; was prepd by heating 2,4,6-trinitro-3-chlorophenol with p-toluenesulfonyl chloride & N,N-diethylaniline and by other methods (Refs 1 & 2)

This expl compd has not found any practical military application

Refs: 1) Beil **5**, 275 & {646} 2) H.B. Haas et al, JACS **72**, 2282 (1950) & CA **44**, 9928 (1950) (A new method of prepn) 3) H. Feuer et al, JACS **73**, 3575 (1951) & CA **46**, 6614 (1952) (Reaction with amino acids) 4) K. Namba & T. Yamashita, KógyôKayakuKyôkaishi **19**, 86-94 (1958) & CA **53**, 11279 (1959) (Prepns with Na azide and kinetics of the reactions)

(5,7-Dichlorobenzo-6-nitro)-benzotriazol-1-ol. See Vol 2, p B88-L, under Benzotriazol and Derivatives

5,6-Dichloro-benzotriazol-1-ol. See Vol 2, p B88-L, under Benzotriazol

Di(chlorobenzoyl) Peroxide, Bis(chlorobenzoyl) Peroxide or Dichlorodibenzoyl Peroxide [called Bis(chlor-benzoyl)-peroxyd or Dichlor-dibenzoyl-peroxyd in Ger],

$ClC_6H_4CO.OO.CO.C_6H_4Cl$; mw 311.11, O20.57%. Three derivs are known:

Di(o-chlorobenzoyl) Peroxide, crysts (from chlf + MeOH), mp 101-02° dec; was prepd by oxidizing 2-chlorobenzoyl chloride with H_2O_2 in alkaline soln (Ref 3)

Di(m-chlorobenzoyl) Peroxide, crysts (from hydrocarbon solvs), mp 123° dec; was prepd by reaction 3-chlorobenzoyl chloride in dry toluene with an excess of aq Na_2O_2 (Ref 4)

Di(p-chlorobenzoyl) Peroxide, crysts (from CS_2), mp 142° dec; explodes in a steel bomb when heated to 180° giving 4-4'-dichlorodiphenyl, 4-chlorobenzoic acid, CO_2 & other products; more sensitive to impact & friction than Dibenzoyl Peroxide; was prepd by reacting 4-chlorobenzoyl chloride in acetone with either 5% H_2O_2 & 25% NaOH below 6° or a soln of Na_2O_2 in ice water (Refs 1 & 2)

Refs: 1) Beil **9**, [227] 2) F. Fichter & M. Adler, Helv **9** 284-86 (1924) & CA **21**, 318 (1927) 3) J.W. Breitenbach & G. Bremer, Ber **76B**, 1129 (1943) & CA 4935 (1944) 4) M. Takebayashi & T. Shingakik BullChemSocJapan **26**, 139 (1953) (in English) & CA **49**, 895 (1955)

Dichlorodiaminoethane, $Cl.NH.CH_2.CH_2.NH.Cl$, may be considered as the parent compd of its dinitro deriv, although not used to prep it:

N,N'-Dichloro-N,N'-dinitro-1,2-diaminoethane, Dichloro-EDNA or Dichloro-Haleite;

$(O_2N)Cl.N.CH_2.CH_2.N.Cl(NO_2)$; mw 218.99, N 25.58%; crysts (from alc), mp 41-41.4°; detonates violently when heated to 100°; it decomp with gas formation in 10% NaOH soln;

reduced by $NaHSO_3$ soln to Ethylene Dinitramine ($O_2N.NH.CH_2.CH_2NH.NO_2$); was prepd when a 5% excess of N,N,N',N'-tetrachloro-1,2-diaminoethane ($Cl_2N.CH_2.CH_2N.Cl_2$) and 99% HNO_3 were added in 30 mins to stirred acetic anhydride at 35°, distilling the reaction mixt at 15 mm pressure, and chilling the oily residue at -75°

Refs: 1) Beil-not found 2) G.N.R. Smart & G.F. Wright, CanJRes **26B**, 289(1948) & CA **42**, 5844(1948) 3) ADL, SynthHE's, 2nd Rept(1951), 203

2,2'-Dichlorodiethylamine. See under item HN in Vol 2, p C168-L

Dichlorodiethylenediamine Complexes of Cobalt. See Cobalt (III) dichlorodiethylenediamine Chlorate and Perchlorate in Vol 3, p C384-L

Dichlorodifluoromethane (Freon 12) (called Difluordichlormethan in Ger), CCl_2F_2; mw 120.93; liq, fr p -158°, bp -29.8°, liq d 1.311 at 25°, liq n_D 1.287 at 25°; crit temp 112°, crit press 40.6 atm, crit vol 1.792 ml/g; heat capacity 0.240 cal/g (°C) at 25°; viscosity 0.26 cp at 25°; dielectric constant 2.13 at 29°; sol in water at 1 atm 0.028% by wt (Ref 2). Other props & methods of prepn are given in Beil (Ref 1)

Freon-12 is used principally as a refrigerant, as a propellant for producing aerosol spray, and in some military applications which are classified

Refs: 1) Beil 1, 61 & {48} 2) Kirk & Othmer **6**(1951), 754; 2nd edit **9**(1966), 744

Dichlorodimethylether. See item 0 in Vol 2, p C169-R

N,N'-Dichloro-N,N'-dinitro-1,2-diaminoethane. See under Dichlorodiaminoethane in this vol

Dichloro-EDNA; Dichloro-Haleite. See N,N'-Dichloro-N,N'-dinitro-1,2-diaminoethane under Dichlorodiaminoethane

DICHLOROETHANE AND DERIVATIVES

Dichloroethane, Ethylene Dichloride or Ethylidene Dichloride, $C_2H_4Cl_2$; mw 98.97. Two isomers are possible:

1,1-Dichloroethane, as-Dichloroethane, or Ethylidene Dichloride (called 1,1-Dichloräthan or Äthylidenchlorid in Ger), $CH_3.CHCH_2$, col liq having a smell & taste resembling chloroform, fr p -97.6°, bp 57.3°, flash p -8.5°, d 1.1601 at 30°, n_D 1.41638 at 20°, Q_{comb} 267.4 kcal/mol at C_v; expl limits in air 5.9-15.9% by vol; less narcotic but more toxic than chloroform; can be produced by reacting HCl with vinyl chloride in the presence of Al, Ferric or Zn chloride catalyst, or by reacting HCl with acetylene in the presence of mercuric-ferric chloride catalyst at RT (Refs 1 & 3, p 148)

1,2-Dichloroethane, s-Dichloroethane, or Ethylene Dichloride, (also known as "Oil of the Dutch Chemists") (called 1,2-Dichloräthan or Äthylenchlorid in Ger), $CH_2Cl.CH_2Cl$, col liq with an odor resembling that of chloroform, turns yel on long exposure to sunlight, fr p -35.4°, bp 83.5°, flash p 17°(CC) & 21°(OC), d 1.2383 at 30°, n_D 1.4449 at 20°, Q_{comb} 296.4 kcal/mol at C_p; expl limits in air 6.2-16.9% by vol; its toxicity over short periods is less than that of chlf, but the fatal dose is less than that of chlf; inhalation of the vapors causes irritation of the respiratory tract, salivation, sneezing & clouding of the cornea; regular exposure to low concns causes headache, vertigo, nausea, vomiting, insomnia & loss of wt; its MAC in working spaces is less than 50 ppm; can be manufd by reacting Cl_2 with ethylene in either liq or gaseous phase (Refs 2 & 3)

Uses. 1,1-Dichloroethane is used as an extractant for heat-sensitive substances, as a solv for rubber & silicone grease, and for dewaxing mineral oils (Ref 3, p 149). 1,2-Dichloroethane is used as a starting material for the manuf of vinyl chloride, as a compon-

ent of antiknock gasoline, in chemical synthesis of ethylenediamine & ethylene glycol diacetate, and as an insecticidal fumigant (Ref 3, p 153)
Refs: 1) Beil **1**, 83, (23), [52] & {139} 2) Beil **1**, 84, (24), [52] & {141} 3) Kirk & Othmer **5**, 2nd edit (1964), 148-54

Azido, $C_2H_3Cl_2N_3$, and *Diazido*, $C_2H_2Cl_2N_6$, derivs of Dichloroethane were not found in Beil or in CA thru 1961

Nitro- & Nitrosoderivatives of Dichloroethane

1-Nitroso-1,1-dichloroethane, $CH_3.C.Cl_2(NO)$; mw 127.96, N 10.95%; dk-blue oil, bp 68° at 763 mm, d 1.2521 at 19°; insol in w; other props & method of prepn are given in Beil **1**, 99 & [70]

Mononitrodichloroethane, $C_2H_3Cl_2NO_2$; mw 143.96, N 9.73%. The following isomers are known:

1-Nitro-1,1-dichloroethane, $CH_3.CCl_2(NO_2)$, liq, bp 120-21° at 756 mm, flash p 57.8°, d 1.4260-1.4271 at 20° (Ref)

1-Nitro-1,2-dichloroethane, $CH_2Cl.CHCl(NO_2)$, lt-yel pungent smelling oil, bp 124° at 10 mm (Ref)

1-Nitro-2,2-dichloroethane, yel liq, bp 45-46° at 5 mm, d 1.4900 at 15°, n_D 1.4765 at 15° (Ref)

Other props & methods of prepn are given in Beil
Ref: Beil **1**, {202}

Dinitrodichloroethane, $C_2H_2Cl_2N_2O_4$; and **Trinitrochloroethane**, $C_2HCl_2N_3O_6$; derivs were not found in Beil or in CA thru 1961

1,1,2,2-Tetranitro-1,2-dichloroethane (called 1.1.2.2-Tetranitro-1.2-dichlor-äthan in Ger), $(O_2N)_2ClC.CCl(NO_2)_2$; mw 278.96, N 20.09%; snow-wh amor powd, sublimes at RT to form star-shaped crysts, mp - begins to decomp at 60-65° with evolution of N oxides, melts sharply at 107°; volatile in steam with partial decompn; very sol in org solvs with decompn; slowly converted by boiling w into CO_2, oxides of N, HCl & dichloro-dinitromethane; forms with KOH or KI, *Dipotassium tetranitroethane*, a yel cryst salt, explosion temp 268°; was prepd by bubbling Cl thru a soln of the dipotassium salt in the presence of Na acetate & cooling the mixt (Refs 1 & 2)

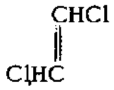
Expl props of parent compd were not reported
Refs: 1) Beil **1**, [70] 2) L. Hunter, JChemSoc **125**, 1482(1924) & CA **18**, 3168(1924) 3) S.S. Novikov et al, IzvestAkadNaukSSSR, OtdelKhimNauk **1961**, 672 & CA **55**, 22097 (1961) (Prepd compd in 86% yield & reports mp 95-96°)

N,N-Dichloroethylamine or Ethyldichloroamine, $C_2H_5.NCl_2$, mw 113.98, N 12.29%; irritating smelly yel oil which does not freeze even at -30°; bp; d 1.2300 at 15°. Was prepd by Tscherniak (Ref 2) by treating monoalkylamine hydrochloride with $CaOCl_2$. Other methods of prepn are given in Ref 1. It hydrolyzes readily in water and explodes on heating. It is one of the Dichloroalkylamines (qv), patented by Riedel (Ref 3)
Refs: 1) Beilstein **4**, 127, (358), [616] & {230} 2) J. Tscherniak, Ber **9**, 143-150 (1876) & **32**, 3582(1900) 3) J.D. Riedel, GerP 301799(1914) & CA **15**, 1996(1921)

Dichloroethylene, $C_2H_2Cl_2$; mw 96.95. Three isomers exist:

1,1-Dichloroethylene, Vinylidene Chloride or as-Dichloroethylene (called 1.1-Dichloräthylen or Vinylidenchlorid in Ger), $CH_2:CCl_2$; clear, mobile, volatile liq, possessing a sweet odor, fr p - 122°, bp 31.8°; flash p -23°; flammable, burning with a green-edged flame; d 1.213 at 20°, n_D 1.4249 at 20°; explosive limits in air 5.6-13% by vol; can be produced by dehydrochlorination of 1,1,2-trichloroethane by agitating with an aq suspension of $Ca(OH)_2$ (Refs 1 & 4, p 178). It is used in the manuf of vinyl compds & for the production of 1,1,1-trichloroethane (Ref 4)

1,2-Dichloroethylene; Acetylene Dichloride; Dioform or s-Dichloroethylene (called 1.2-Dichlor-äthylen or Acetylendichlorid in Ger), $ClHC:CHCl$; known in trans & cis forms which are colorless, mobile liquids with a sweetish, sl irritant odor resembling that of chlf (Refs **2** & 4, p 180)

Trans form: 
$\begin{matrix} CHCl \\ \| \\ ClHC \end{matrix}$
, fr p -49.4°, bp 47.7°, d 1.2631 at 10°, n_D 1.4462 at 20°; forms a ternary azeotrope contg ethanol 1.4 & water 1.1% boiling at 44.4°C

Cis form: 
$\begin{matrix} CHCl \\ \| \\ CHCl \end{matrix}$
, fr p -81.5°, bp 60.2°, d 1.2917, n_D 1.44900 at 20°; forms a ternary azeotrope contg ethanol 6.65 & water 2.85% which boils at 53.8°

The tech product contains varying proportions of trans & cis isomers depending upon the conditions of manuf. A typical tech product boils in the range 45-60°, has a flash p of 6°, and expln limits in air 9.7-12.8% by vol. This compd burns with diffic but it forms expl mixts with air. It is produced by reduction of 1,1,2,2-tetrachloroethane or by direct chlorination of acetylene

It is used as a low-temp, special purpose solv, and in the manuf of rubber solns. It is reported to have been used in Germany for the extraction of NG from Dynamites (Ref 3)

Refs: 1) Beil **1**, 186, (77), [158] & {647} 2) Beil **1**, 187, (78), [159] & {651} 3) Naoúm, NG(1928), 447 4) Kirk & Othmer, 2nd edit **5**(1964), 178, 180 5) ChemikerZtg **88**, No 10 (1964), U35 Cited by Dr A. Langhans in Explosivst **1964**, 246 (Reports Dichloroethylene to be combustible)

Dichloroethylsulfide. See item H (or HS) in Vol 2, p C168-L

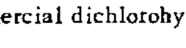
Dichloro-Haleite. See N,N'-Dichloro-N,N'-Dinitro-1,2-diaminoethane under Dichlorodiaminoethane in this Vol

DICHLOROHYDRIN AND DERIVATIVES

Dichlorohydrin or Glycerol Dichlorohydrin, $C_3H_6Cl_2O$; mw 128.99; exists in two isomers, both of which are present in the commercial product:

α,β-(or 2,3)Dichlorohydrin or 2,3-Dichloro-1-propanol [called 2.3 Dichlor-propan ol-(1); 2.3-Dichlor-propylalkohol; Glycerin-1.2-dichlorhydrin; or β-Dichlorhydrin in Ger], $ClH_2C.CHCl.CH_2OH$; col sl viscous liq, bp 182-85°, d 1.3607-1.3616 at 20°, n_D 1.4819-1.4849 at 20°; Q_{comb} 3184 cal/g at C_v, flash p 195°F (OC); miscible with org solvs; also forms azeotropes (Refs 1 & 4). For prepn, toxicity & uses, see below under Commercial Dichlorohydrin

α,γ-(or 1,3)Dichlorohydrin or 1,3-Dichloro-2-propanol [called 1.3-Dichlor-propanol-(2); β.β'-Dichlor-isopropylalkohol; Glycerin-1.3-dichlorhydrin; or α-Dichlorhydrin], $CH_2Cl.CH(OH).CH_2Cl$; col sl viscous liq having a sweet taste, bp 175°, d 1.3645 at 20°, n_D 1.48375 at 20°, Q_{comb} 3151 cal/g; solubility in water 15.6% by wt; miscible with alcohols, acet, benz & eth; not miscible with petr eth; forms azeotropes with w & some org solvs (Refs 2 & 4). For prepn, toxicity & uses, see below under Commercial Dichlorohydrin

Commercial Dichlorohydrin consists of the above two isomers, the proportions of which depend on method of prepn; d 1.36-1.39, bp 175-80°, flash p 74°. Glycerol was the main source for the prepn of glycerol chlorohydrins until the process for direct substitutive chlorination of propylene to allyl chloride paved the way for synthesis by chlorohydrination of allyl chloride. In the synthesis from glycerol, excess HCl is used in the presence of 4% acetic acid. The reaction is run at 130° to yield 90% of product which is mainly the α,γ-form. Synthesis from propylene yields a mixt of approx 70% α,β-form & 30% α,γ-form. Addn of HCl to epichlorohydrin,  $CH_2\text{—}CHCH_2Cl$ (with O bridge), at -10° yields 99.6% α,γ-dichlorohydrin & 0.4% α,β-form. The α,β-form may be obtd in 92.5% yield by chlorination of acrolein, followed by reduction, or by chlorination of allyl alcohol (Ref 5). Naoúm (Ref 4) states that a mixture of two dichlorohydrins, sym bp 174° & asym bp 182°-183°, has been used to prep commercial dichlorohydrin nitrate

Toxicity. Dichlorohydrin has a narcotic & depressant action on the heart, circulation & respiration. It is harmful when taken internally or when absorbed thru the skin
Uses. According to Daniel (Ref 3), Dichlorohydrin was proposed at the end of the 19th century as a solv for NC used in commercial expls, since it is less volatile than some other solvs (such as ether-alc, acetone, ethyl or amyl acetate). Dichlorohydrin is used in the production of epichlorohydrin, as intermediates in the manuf of glycerol derivs, sulfur-contg polymers & textile finishing agents (Ref 5)
Refs: 1) Beil 1, 356, (181), [371] & {1426}
2) Beil 1, 364, (185), [383] & {1471}
3) Daniel (1902), 301 (under Fleming)
4) Naoúm, NG (1928), 191 5) Kirk & Othmer, 2nd edit **5**(1964), 314ff

Nitrite- & Nitrate Derivatives of Dichlorohydrin

2,3-Dichlorohydrin-1-nitrite (called 2.3-Dichlor-propyl-nitrit in Ger), $ClH_2C.CHCl.CH_2O.NO$; mw 141.99, N 9.86%; lt yel lachrymatory liq, bp 55° at 14 mm press, d 1.323 at 22°, n_D 1.456 at 16°; was prepd by reacting 2,3-dichloro-1-propanol with $NaNO_2$ & dil HCl
Ref: Beil **1**, {1427}

2,3-Dichlorohydrin-1-nitrate (called β,γ-Dichlorpropyl-nitrat in Ger), $ClH_2C.CHCl.CH_2O.NO_2$; mw 173.99, N 8.05%; liq, bp 180°, d 1.3 at 7°; was prepd by nitrating 2,3-dichlorohydrin with HNO_3
Ref: Beil **1**, 356

1,3-Dichlorohydrin-2-nitrate [called (β,β'-Dichlor-isopropyl)-nitrate in Ger], $ClH_2C.CH(O.NO_2).CH_2Cl$; mw 173.99, N 8.05%; col very mobile liq, having an aromatic odor, fr p below -20°, bp 180-90° (dec), d 1.459 at 15°; was prepd by nitrating 1,3-dichlorohydrin with mixed acids (Ref 1). Naoum (Ref 2) reports some addnl props and compares this compd with NG & Chlorohydrin Dinitrate (qv)
Refs: 1) Beil 1, 364 & {1473} 2) Naoum, NG (1928), 191-93

Commercial Dichlorohydrin Nitrate consists of the above two isomers; col very mobile liq having a spicy aromatic odor, bp 176° (dec) & 88° at 15 mm; d 1.45 at 15°; insol in w; sol in NG & in solvs which dissolve NG; more volatile than either NG or Chlorogydrin Dinitrate. Naoúm (Ref) recommends nitration by introducing, with cooling, dichlorohydrin into a mixt of HNO_3 (60%) & H_2SO_4 (40%), collecting & washing the ester with water, followed by a weak soda soln and finally with water. After drying a yield of 90% of theory is obtd

This compd is a weak expl, extremely insensitive to initiation by shock. It cannot be exploded in the liq state by a No 8 cap when in a Trauzl block; but if mixed with kieselguhr (10/3 by wt) it can be exploded by a No 8 cap giving a net expansion of 45 cc in Pb block test

It decomp according to one of the following equations:

$$2C_3H_5Cl_2(ONO_2) \rightarrow 6CO + 4HCl + 3H_2 + N_2$$
$$\rightarrow 4CO + 2H_2O + 4HCl + 2C + H_2 + N_2$$

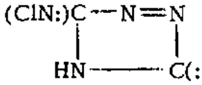
The heat of combustion for first equation is 259 cal/g and for second 440 cal/g at C_v with H_2O vapor. Heat of formation is 81.9 kcal/mole

It has been recommended that coml Dichlorohydrin Nitrate be used as an antifreeze addn to dynamites as a substitute for Chlorohydrin Dinitrate. According to Naoúm (Ref), the coml product offers no advantage because it possesses low viscosity, high volatility, high chlorine content, and does not gelatinize Colloidon cotton; it therefore would not prevent exudation of gelatinized expls
Ref: Naoúm, NG (1928), 191-93

NOTE: No higher nitrated derivs of Dichlorohydrin were found in Beil or in CA thru 1961

1,4-Dichloroimino-benzoquinone. See Vol 2, p B83-L under Benzoquinonediimine

3,5-Dichloroimino-3,5-dihydro-1,2,4-triazole [called Di-(chlorimino-3.5-dihydro-3.5-triazol-1.2.4 by Stollé & Dietrich],



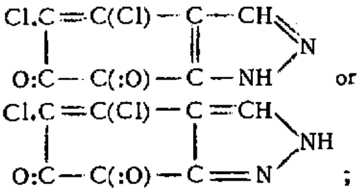
$$\begin{array}{l}(ClN:)C-N=N\\ \quad\ \ |\qquad\quad\ |\\ \quad HN-C(:NCl)\end{array}$$

; mw 165.98, N 42.20%; yel amorphous powd; mp - detonates violently

at 135°; sol in eth; insol in w; was prepd by adding an aq soln of guanazole, $H_2NC:N.N:C(NH_2).NH$, to bleaching powder contg 7% "Perchloron "(Pennsalt Chemicals Corp trademark for high-test calcium hypochlorite)(Ref 2)
Refs: 1) Beil-not found 2) R. Stollé & W. Dietrich, JPraktChem **139**, 207-08(1934) & CA **28**, 2714(1934)

1,4-Di(chloroimino)-p-quinone. See Benzoquinone-1,4-di(chloroimine) in Vol 2, p B83-L

4,5-Dichloroindazole-6,7-quinone [called 4.5-Dichlor-indazol-(6.7) in Ger],



; mw 217.02, N 12.94%; red lfts (from glac acet ac + HNO_3), mp - deflgr above 350°; sol in cold dil caustic soda soln; sl sol in acet & alc; v sl sol in benz & benzine; was prepd by heating 4,5,7-trichloro-6-hydroxyindazole with concd HNO_3 in glac acet ac, or by heating 4,5,7-trichloro-6,6,7-trihydroxy-6,7-dihydroindazole with water (Refs 1 &2)
Refs: 1) Beil **24**, [192] 2) K. Fries, Ann **454**, 315, 319(1927) & CA **21**, 2693(1927)

Dichloro-isonicotinyl Azide. See 2,6-Dichloropyridine-4-carboxylic Acid Azide in this vol

Dichloromethane and Derivatives

Dichloromethane, Methylene Chloride or Methylene Dichloride (called Dichlormethan or Methylenchlorid in Ger), CH_2Cl_2, colorless, mobile liq having a pleasant ethereal odor, fr p -96.7°, bp 40.4°, d 1.326 at 20°, n_D 1.4246 at 20°(liq), Q_{comb} 144.5 kcal/mol; expl limits in O_2 15.5-66% by vol; forms binary azeotropes with many solvs; can be produced by chlorinating either methane or methyl chloride, or by reduction of chloroform or CCl_4. It is the least toxic of the chloromethanes. It is used in paint strippers, as a degreasing solv, in the manuf of aerosols, photographic film & synthetic fibers (Refs 1 &2)
Refs: 1) Beil **1**, 60, (8), [13] & {43}
2) Kirk & Othmer, 2nd edit **5**(1964), 111ff
3) US Specification **MIL-D-6998B** (June 1967)

Mononitrodichloromethane, $CHCl_2.NO_2$; mw 129.94, N 10.78%; oil, bp 106-07°; was prepd by passing Cl into an aq soln of K salt of nitro-acetic acid ($O_2N.CH_2.COOK$) (Refs 1 & 2)
Refs: 1) Beil **1**, {113} 2) W. Steinkopf & M. Kühnel, Ber **75B**, 1939(1942) & CA **37**, 4687(1943)

Dinitrodichloromethane, $CCl_2(NO_2)_2$; mw 174.94, N 16.02%; colorless oil, bp 46° at 20 mm, d 1.6124 at 20°, n_D 1.4575 at 20°; sol in alc, eth, chlf, benz & CS_2; insol in w; can be prepd by heating 2,4,6-trichloroaniline or trichloroethylene with concd HNO_3, and by other methods (Ref 1). See also Refs 2 & 3

Its expl props were not investigated
Refs: 1) Beil **1**, 78, (21), [44] & {115}
2) R.A. Gotts & L. Hunter, JChemSoc **125**, 442(1924) 3) E. Gryzskiewicz-Trochimowski et al, BullFr **1948**, 597 & CA **41**, 8165(1948)

Dichloromethyl-chloroformate. See under item b in Vol 2, p C169-L

Dichloromethyl-thallium-chloride. See Bis(chloromethyl)-thallium Chloride in Vol 2, p B139-L

Dichloro-iso-nicotinyl Azide. See 2,6-Dichloropyridine-4-carboxylic Acid Azide in this vol

DICHLOROPHENOL AND DERIVATIVES

Dichlorophenol or Hydroxydichlorobenzene (called Dichlor-phenol or Dichlor-hydroxy-benzol in Ger), $C_6H_3Cl_2.OH$; mw 163.00. All possible isomers are known:

2,3-Dichlorophenol, prisms (from petr eth), mp 56-58°, readily volatile; of no coml importance (Refs 1 & 6)

2,4-Dichlorophenol, hexagonal ndls (from benz), mp 43°, bp 206-08° at 753 mm press, vol with steam; forms some unstable salts; most important use is in the manuf of 2,4-D (2,4-dichlorophenoxyacetic acid), a powerful plant-growth regulator (Refs 2 & 6)

2,5-Dichlorophenol, crysts (from benz), mp 59°, bp 209-11°, volatile with steam; of no coml importance (Refs 3 & 6)

2,6-Dichlorophenol, crysts (from petr eth), mp 66-68°, bp 220°, volatile with steam; no coml application (Refs 4 & 6)

3,4-Dichlorophenol, ndls (from petr eth) mp 64-66°, bp 145-46°, volatile with steam; no coml utility (Refs 5 & 6)

3,5-Dichlorophenol, crysts (from petr eth), mp 68°, bp 233° at 757 mm press; can be nitrated & bromonated to produce interesting derivs, but its high starting cost has precluded coml develop (Refs 5 & 6)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **6**, (102) & {699} 2) Beil **6**, 189, (103) & [178] 3) Beil **6**, 189, (103), [178] & {712} 4) Beil **6**, 190, (103), [179] & {713} 5) Beil **6**, 190, (103), [179] & {115} 6) Kirk & Othmer, 2nd edit **5**(1964), 332-33

Nitroderivatives of Dichlorophenol

Mononitrodichlorophenol, $O_2N.C_6H_2Cl_2.OH$; mw 208.00, N 6.74%. The following isomers are described in Beil:

2-Nitro-3,4-dichlorophenol, yel ndls (from petr eth), mp 76° (Ref 1)

2-Nitro-3,5-dichlorophenol, yel ndls (from petr eth), mp 51° (Ref 1)

2-Nitro-3,6-dichlorophenol, yel prisms (from petr eth), mp 70° (Ref 1)

2-Nitro-4,6-dichlorophenol, lt-yel lfts,(from alc), plts (from chlf), prisms (from eth), mp 121-24°, sublimes below 100°, deflgr on rapid heating, d 1.822 at 19°; sol in alc, eth, chlf & benz; sl sol in w; its Ammonium salt, $H_4N.C_6H_2Cl_2O_3N$, orn-red ndls, sublimes at 100° & deflgr on rapid heating (Ref 2)

3-Nitro-2,4-dichlorophenol, yel-grn ndls (from petr eth), mp 85-87° (Ref 3)

3-Nitro-4,6-dichlorophenol, ndls (from w), mp 105-06° (Ref 4)

4-Nitro-2,5-dichlorophenol, shiny ndls (from w), prisms (from alc), ndls (from benzine), mp 115-17°, volatile with steam (Ref 5)

4-Nitro-2,6-dichlorophenol, almost col ndls (from w), lfts (from glac acet ac), pltlts (from eth) & crysts (from petr eth); mp 125-27° (dec), only sl volatile with steam; forms many colored salts (Ref 6)

4-Nitro-3,5-dichlorophenol, lt grn-yel prisms (from w), mp 150-51°, not volatile with steam (Ref 7)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **6**, [229] 2) Beil **6**, (122), [230] & {841} 3) Beil **6**, [230] & {841} 4) Beil **6**, [230] 5) Beil **6**, 241 & [231] 6) Beil **6**, 241, (122), [231] & {841} 7) Beil **6**, (122) & [231]

Dinitrodichlorophenol, $(O_2N)_2C_6HCl_2.OH$; mw 253.01, N 11.07%. Three isomers are described in the literature:

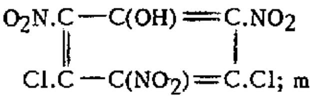
??-Dinitro-??-dichlorophenol, ndls, mp 105-06°; was prepd by nitrating the 2,4-dichlorophenyl ester of propionic acid with mixed acids; forms colored salts (Ref 1)

2,4-Dinitro-3,5-dichlorophenol, yel plts (from w), mp 120°; sol in alc; sl sol in eth & benz; was prepd by heating 1,3-dinitro-2,4,6-trichlorobenzene with acetamide & anhyd Na acetate at 160° (Refs 2 & 4)

2,4-Dinitro-3,6-dichlorophenol, lt yel prisms (from benz), mp 146°; was prepd by nitrating 2,5-dichlorophenol with an excess of HNO_3 in the cold (Refs 3 & 5) and by other methods (Ref 6)

Refs: 1) Beil **6**, 261 2) Beil **6**, [248] 3) Beil **6**, [249] 4) W. Borsche & W. Trautner, Ann **447**, 9(1926) & CA **20**, 1222 (1926) 5) K. Fries & W. Buchler, Ann **454**, 247 (1927) & CA **21**, 2692(1927) 6) P.I. Petrovich, ZhurVsesoyuzKhimObshch im D.I. Mendeleeva **5**, 106(1960) & CA **54**, 20937(1960)

2,4,6-Trinitro-3,5-dichlorophenol (called 3.5-Dichlor-2.4.6-trinitro-phenol or Dichlorpikrinsäure in Ger),



; mw 298.00, N 14.10%; lt-yel prisms (from glac acet ac), mp 139-40°; or col crysts (from chlf or CCl_4), mp 135°, bp - explodes; readily sol in alc & eth; sol in hot w; v sl sol in petr eth; obtd when 3,5-dichlorophenol in glac acet ac was treated with red fuming HNO_3 and heated on a w bath to 70° (Refs 1, 2 & 3)
Refs: 1) Beil **6**, 292 & (141) 2) J.J. Blanksma, Rec **27**, 37 (1908) 3) R. Willstätter & G. Schudel, Ber **51**, 787 (1918) & CA **13**, 578 (1919)

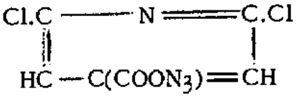
NOTE: It is obvious from above formula that higher nitrated derivs of Dichlorophenol are not possible

Dichlorophenylazide. Same as Azidodichlorobenzene. See under Azido & Diazo Derivatives of Dichlorobenzene in this vol

Dichlorophenyl-azotetrazole. See Bis(chlorophenyl)-azotetrazole in Vol 2, p B139-L

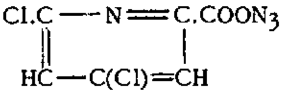
Dichloropicolinyl Azide. See 4,6-Dichloropyridine-4-carboxylic Acid Azide

2,6-Dichloropyridine-4-carboxylic Acid Azide or 2,6-Dichloro-iso-nicotinyl Azide (called 2.6-Dichlor-isonicotinsäure-azid or 2.6-Dichlor-pyridin-carbonsäure-azid-(4), in Ger),



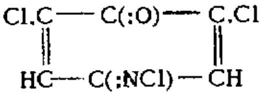
; mw 217.02, N 27.04%; crysts, mp 89°; deflgr very weakly on heating in a free flame; was prepd by treating its hydrazide, $NC_5H_2Cl_2.CO.NH.NH_2$ (mp 184°) with $NaNO_2$ in aq HCl at 0° (Refs 1 & 2)
Refs: 1) Beil **22**, (505) 2) H. Meyer & E.R. vonBeck, Monatsh **36**, 737 (1915) & CA **10**, 180 (1916)

4,6-Dichloropyridine-2-carboxylic Acid Azide or 4,6-Dichloro-picolinyl Azide,



; mw 217.02, N 27.04% crysts, mp 74°; was prepd in a manner similar to that of the 2,6-Dichloro deriv (Ref 2)
Refs: 1) Beil-not found 2) R. Graf, JPraktChem **133**, 36 (1932) & CA **26**, 1933 (1932)

2,6-Dichloroquinonechlorimide [called 2.6-Dichlor-chinon-chlorimid-(4) in Ger],



; mw 210.46; yel ndls (from alc), mp 67-68°; was prepd from the HCl salt of 2,6-dichloro-4-aminophenol by treating it with an acidic chloride of lime soln (Ref 1)

It is reported by B.T. Taranto of Schering Corp, Bloomfield, NJ (Ref 2) that this reagent, used for spot visualization in chromatographic systems, presents a potential expln hazard. Tests of this compd and of the *2,6-Dibromo deriv* (qv) in a thermal stability bomb showed both to be expl, but the dichloro deriv was not as unstable as the dibromo deriv
Refs: 1) Beil **7**, 634 2) Anon, C & EN **45**, No 52, p 54 (11 Dec 1967)

1,7-Dichloro-2,4,6-Trinitro-2,4,6-triazaheptane or GSX (called by Jones & Thorn) or **1,1′-Bis[(chloromethyl)nitramine]-N-nitro-dimethylamine** (CA nomenclature),
$Cl.CH_2.N(NO_2)\ CH_2.N(NO_2).CH_2N(NO_2).CH_2Cl$; mw 314.02, N 31.23%; fine crysts (from benz), mp 144.5-45.5°, bp - explodes; was prepd by saturating with anhyd HCl at 10° a soln of 1-acetoxy-7-trifluoroacetoxy-2,4,6-trinitro-2,4,6-triazaheptane. GSX was also prepd from HCl & the diacetate of 1,7-diacetoxy-2,4,6-trinitro-2,4,6-triazaheptane; 1,7-bis-trifluoroacetoxy-2,4,6-trinitro-2,4,6-triazaheptane & HCl gave a 20% yield of GSX (Ref 3). Jones & Thorn (Ref 2) detd its UV spectra, but did not report its prepn. The expl props of GSX were not detd
Refs: 1) Beil-not found 2) R.N. Jones & G.D. Thorn, CanJRes **27B**, 830 (1949) & CA **44**, 2848 (1950) 3) R. Reed, Jr, JACS **78**, 804 (1956) & CA **50**, 12865 (1956)

Dichromates. See under Chromates, Dichromates, Trichromates and Tetrachromates in Vol 3, p C283ff

Dicinnamoyl Peroxide or Cinnamoyl Peroxide (called Dicinnamoylperoxyd in Ger), $(C_6H_5.CH:CH.CO.O\text{-})_2$; mw 294.29, O 21.75%; col ndls (from acet or alc-acet), mp 133°, 144°, deflgr on heating in a flame; was prepd by slowly addg cinnamoyl chloride in acet to a sl excess of alkaline H_2O_2 (Refs 1 & 2), and by other methods (Refs 3 & 9). It is used as a polymerization catalyst for styrene (Refs 7, 8 & 10) and methyl methacrylate (Ref 9)

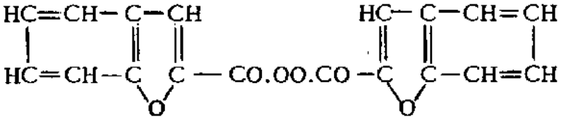
Refs: 1) Beil **9**, [390] 2) H. Wieland & G. Rasuwajew, Ann **480**, 161, 165-66 (1930) & CA **24**, 4013 (1930) 3) D.H. Hey & E.W. Walker, JCS **1948**, 2216 & 2218 & CA **43**, 3394 (1949) 4) J.W. Breitenbach & J. Derkosch, Monatsh **81**, 689 (1950) & CA **45**, 3233 (1951) (Heat of combustion) 5) Ibid, **81**, 530 (1950) & CA **45**, 5021 (1951) (UV absorption spectrum) 6) Ibid **82**, 177 (1951) & CA **45** 10029 (1951) (Recalculated values of $Q_{combstn}$) 7) H.F. Park, USP 2593399 (1952) & CA **46**, 7364 (1952) 8) W. Cooper, JCS **1952**, 2408 & CA **47**, 3087 (1953) 9) N.G. Saha et al, JCS **1956**, 427 & CA **50**, 6833 (1956) 10) I.V. Sukmanskaya & A.I. Yurzhenko, ZhObshKhim **30**, 2108 (1960) & CA **55**, 9944 (1961)

Dickson Powder. A Canadian commercial expl patented in England in 1895: Ba nitrate 40.00, PA 32.00, K chlorate 6.33, wheat flour 6.33, K ferrocyanide 6.34, liquid ammonia 8.00 & lampblack 1.00%

Ref: Daniel (1902), 205

Dicobaltocarbonyl. See Cobalt Tetracarbonyl in Vol 3, p C387-L

Dicoumariloyl Peroxide or Coumariloyl Peroxide



mw 322.26; O 29.79%; small pale-yel nds, mp - exploded at 117°; flashed violently when heated in a flame; was obtd when aq NaOH & a soln of coumariloyl in Na-dried ether were dropped simultaneously into stirred aq H_2O_2 at 0°; decompd by refluxing in benzene, CCl_4 and some other solvs

Refs: 1) Beil-not found 2) M.C. Ford & W.A. Waters, JCS **1951**, 824 & CA **45**, 9027 (1951)

Dicresol; Dihydroxybitolyl or Dihydroxydimethylbiphenyl. See Bicresol in Vol 2, p B112-R

Dicrotonyl Peroxide or Crotonyl Peroxide (called Dicrotonyl-peroxyd or Crotonylperoxyd in Ger), $CH_3.CH:CHCO.OO.CO.CH:CH.CH_3$; mw 170.16, O 37.61%; ndls or plts (from petr eth), mp 41°, explodes mildly on further heating; sol in common solvs; its aq soln slowly hydrolizes; was prepd by action of BaO_2 on an ethereal soln of crotonic anhydride at 0° (Refs 1 & 2). See Refs for addnl props & methods of prepn

Refs: 1) Beil **2**, 411 & {1265} 2) A.M. Clover & G.F. Richmond, AmChemJ **29**, 174-203 (1903) & JCS **84** I, 396 (1903) 3) F. Fichter & J. Rosenzweig, Helv **18**, 238 (1935) & CA **29**, 2094 (1935 (Thermal decompn of Dicrotonyl Peroxide) 4) K.H.W. Tuerck, USP 2379390 (1945) & CA **39**, 4620 (1945) (Prepn from crotonic anhyd & H_2O_2 in aq alk medium) 5) T. Uno, Kobunski Kagaku **17**, 183 (1960) & CA **55**, 15993 (1961) (Use of Dicrotonyl Peroxide in the prepn of graft polymers)

Dicuminoyl Peroxide (called Dicuminoylperoxyd in Ger), $[(CH_3)_2CH.C_6H_4.CO\text{-}]_2O_2$; mw 326.38, O 19.61%; ndls (from eth), mp - explodes on heating; was prepd by reacting Ba peroxide hydrate with cuminoyl chloride (Refs 1 & 2)

Refs: 1) Beil **9**, 547 2) B.C. Brodie, Jahresbericht über die Fortschritte der Chemie **1863**, 317

Dicumyl Peroxide of Bis(α,α-dimethylbenzyl) Peroxide, $[C_6H_5C(CH_3)_2O\text{-}]_2$; mw 270.36, O 11.84%; wh crysts (from MeOH), mp 39°, bp - decomp rapidly at 120°, n_D 1.5360 at 21°(Ref 3), thermally less stable than *Cumyl Hydroperoxide* (qv, Vol **3**, p C574-R); was formed by heating the hydroperoxide in the presence of Norit, acetic acid or dimethylbenzyl alcohol at 100°; or by decompn of acetyl peroxide in a soln of the hydroperoxide & cumene (Ref 2)

Its mononitro deriv, *Bis(α,α-dimethyl-p-nitrobenzyl Peroxide,* $[p\text{-}O_3N.C_6H_4C(NO_3)_2O\text{-}]_2$, crysts(from alc), mp 158°(decompn), was prepd by Hock & Kropf (Ref 4)

Refs: 1) Beil-not found 2) M.S. Kharasch et al, JOC **15**, 753-62(1950) 3) Tobolsky & Mesrobian (1954), 165 4) H. Hock & H. Kropf, ChemBer **88**, 1544(1955) & CA **50**, 16695(1956)

Dicuproacetaldehyde. See Vol 1, p A73-L

Dicy. One of the abbreviations for Dicyandiamide

Dicyanamide (called Dicyanamid, Dicyanimid or Imino-dicarbonsäure-dinitril in Ger), NC.NH.CN; mw 67.05, N 62.67%; ndls + H_2O; obtd by reacting chloramine with an aq soln of KCN and by other methods; forms some unstable salts such as:

Ammonium salt, $NH_4C_2N_3$, ndls (from alc + eth), mp 126°, decomp on heating above 126°

Copper salt, $Cu(C_2N_3)_2$, blue-grn ndls, deflgr on heating

Lead salt, crysts, decomp on rapid heating

Mercury salt, $Hg(C_2N_3)_2$, cryst ppt, deflgr on heating to 110° and increases in vol like a Pharoh's serpent, emitting a squeaky noise

Other props are given in Beil

Ref: Beil **3**, 82, [68] & {154}

Dicyanamideazide. See Cyanoformamidine Azide in Vol 3, p C586-L. This compd was found to be effective in increasing the consumability of cartridge cases at low pressure

Ref: W.A. Abel & M.B. Nelson, ArmourResFoundation Rept **3200-6**(1963)

Dicyandiamide. See Cyanoguanidine in Vol 3, p C587-L

Dicyandiamide Nitrate. See Cyanoguanidine Nitrate, Vol 3, p C588-L

DICYANDIAMIDINE AND DERIVATIVES

Dicyandiamidine, Guanylurea or Amidinourea (CA name);(called Dicyandiamidin, Guanylharnstoff, Carbamoylguanidin or Guanidincarbonsäure-amid in Ger), $H_2H.C(:NH).NH.CO.NH_2$; mw 102.10, N 54.88%; crysts (from alc), mp 104-105.5°, bp dec 160°; v sol in w, warm alc & pyridine; insol in benz, chlf, eth & CS_2; can be prepd by heating dicyandiamide with aq NH_3 at 150°, and by other methods (Ref 1). Its admixtures with oxidizing agents were recommended as powerful expl (Ref 3)

Dicyandiamidine forms numerous cryst salts, addn compds & a nitrocompd.

Its **Perchlorate salt,** $C_2H_6N_4O + HClO_4$, ndls, does not melt at 260°, sublimes at higher temp & explodes on further heating (Ref 1, p {166}). This salt is an expl as powerful as TNT but less sensitive to impact. It has been proposed for use in military & commercial expls (Ref 2): a) Perchlorate 69, $NaNO_3$ 29 & woodmeal 2% b) Perchlorate 10, TNT 10 & AN 80%. Blatt (Ref 4) gives the following props for the perchlorate:

Density 1.8 g/cc

Explosion temperature 378°

Impact sensitivity 117% PA (Brit FI test)

Power 71% PA by Trauzl test and 92% TNN by Ballistic Mortar test

Refs: 1) Beil **3**, 89, (42), [74] & {164} 2) W. Rintoul & E. Beckett, BritP 14706(1915) & CA **13**, 1640(1919); USP 1298793(1915) & CA **13**, 1766(1919) 3) C. Mannelli & B. Luigi, ItalP 49536(1919) & CA **14**, 2555(1920) 4) Blatt, OSRD **2014**(1944)

Nitrodicyandiamidine (called N'-Nitro-N-guanyl-harnstoff or Nitrodicyandiamidin in Ger) $H_2N.C(:NH).NH.CO.NHNO_2$; mw 147.10, N

47.61%; fine crysts, mp - deflgr on heating; $Q^v_{combstn}$ 285.82 kcal/mol & Q_{form} 71.96 kcal/mol (Ref 4); sol in NH_3 & alkalies; v sl sol in w; insol in alc & eth; was prepd by treating Dicyandiamide with mixed acids in the cold (Ref 1)

Its admixtures with oxidizing agents were recommended as powerful expls (Ref 2). Its *Silver salt*, $AgC_2H_4N_5O_3$, amor ppt, deflgr on heating

Refs: 1) Beil **3**, 126, (59) & {236} 2) C. Mannelli & B. Luigi, ItalP 49536(1919) & CA **14**, 2555(1920) 3) A.H. Blatt & F.C. Whitmore, OSRD **1085**(1942), p 62 (This expl is reported to be of low power) 4) L. Médard & M. Thomas, MP **39**, 339-40, 355(1957)

Dicyandiamidine Perchlorate. See under Dicyandiamidine in this vol

Dicyandiazide (called Dimers Cyanazid; "Diazido-cyanimino-methan"; or "Dicyandiazid" in Ger), $NC.N:C(N_3)_2$ or $(NC.N_3)_2$; mw 136.08, N 82.35%; col ndls, mp - decomp at 127° and explodes violently at 143°; also explodes violently on sl friction or shock; readily sol in alc, eth & acet; sol in w; v sl sol in benz; insol in petr eth; was obtd when 3 mols NaN_3 & 1 mol dibromomalonitrile reacted in cold water & ether, and the soln acidified. Ag nitrate ppts from the soln a *Silver salt* which is an expl extremely sensitive to friction (Refs 1 & 2)

Compare with Cyanazide in Vol 3, p C585-L

Refs: 1) Beil **3**, {239} 2) E. Ott & H. Weissenburger, Ber **70B**, 1829, 1832-34 (1937) & CA **31**, 7401(1937)

1,1'-Dicyanobicyclohexyl (called 1.1'-Dicyndicyclohexyl in Ger), $NC.C_6H_{10}.C_6H_{10}.CN$; mw 216.32, N 12.95%; wh crysts (from acet), mp 224.5-225°; was prepd by passing N thru solid 1,1 -azo-bis-1-cyclohexane nitrile and heating to 120° for 1 hr (Ref 3) or by refluxing 1,1'-dicyanoazocyclohexyl with 4 times its weight of dry toluene until the evolution of N ceased (Refs 1 & 2). Overberger et al (Ref 3) warned that heating of the 1,1'-azo-bis-1-cyclohexane nitrile in quantities larger than 1.92 g (0.008 mole) may result in an expln

Refs: 1) Beil **9**.[571] 2) H. Hartman, Rec **46**, 151(1927) 3) C.G. Overberger et al, JACS **71**, 2662-63(1949) & CA **44**, 5799(1950)

1,2-Dicyanoethane or Succinonitrile (called Bernsteinsäure-dinitril; Succinonitril; Äthylencyanid; or Butandinitril in Ger), $NC.CH_2.CH_2.CN$; mw 80.09, N 34.98%; col waxy solid, mp 57.7°, bp 267°, d 1.022 at 25°; sol in w, alc & eth; sl sol in CS_2 & n-hexane; can be prepd by addn of HCN to acrylonitrile ($CH_2:CHCN$) and by other methods (Refs 1 & 3)

Blatt (Ref 2) reports this compd (under NITRILES) to be an expl less powerful & less brisant than PA

Refs: 1) Beil **2**, 615, (265), [554] & {1672} 2) Blatt, OSRD **2014**(1944) 3) Kirk & Othmer **9**(1952), 369

Dicyanomethane or Malononitrile (called Malonsäure-dinitril, Malonitril or Methylencyanid in Ger), $NC.CH_2.CN$; mw 66.06, N 42.41%; col crysts, mp 31.6-32.4°, bp 108-09° at 17 mm press, cryst d 1.191 at 20°, liq d 1.0494 at 35°, n_D 1.4139 at 34.2°; prepd by esterification of cyanoacetic acid, and treatment of the ester with NH_3 which leads to cyanoacetamide which, on reaction with phosphorous oxychloride or pentachloride, gives Dicyanomethane (Ref 1)

Blatt (Ref 2) reports this compd (under NITRILES) to be an expl less powerful & less brissnt than PA

Refs: 1) Beil **2**, 589, (256), [535] & {1634} 2) Blatt, OSRD **2014** (1944) 3) ADL PureExplCompds, Part 1(1947), p 63

Di(cyanomethyl)amine (called Bis-cyanmethylamin, Iminodiessigsäure-dinitril, Iminodiacetonitril or Diglykolamidsäure-dinitril in Ger), $HN(CH_2.CN)_2$; mw 95.10, N 44.19%;

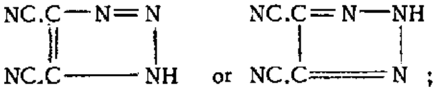
crysts (from w), plts (from alc or eth), mp 75-77°; can be nitrated to form an expl deriv (Ref 1)

Di(cyanomethyl)nitramine, $O_2N.N(CH_2.CN)_2$; mw 140.10, N 39.99%; crysts (from acet + w), ndls (from anhyd benz), mp 96-97°; sl sol in benz, alc, eth & ethyl acet; v sl sol in w & chlf; insol in petr eth; prepd by nitrating Di(cyanomethyl)amine with 99% HNO_3 & acetic anhydride at 10-15° (Ref 2)

This expl is reported by Blatt (Ref 3) under NITRAMINES to be less powerful & less brisant than PA (Refs 3 & 4)

Refs: 1) Beil **4**, 367, (481); [800] & {1176} 2) Beil **4**, (488) & {1207} 3) Blatt, OSRD **2014**(1944) 4) ADL PureExplCompds, Part 1 (1947), p 64

4,5-Dicyano-1,2,3-triazole (called 4.5-Dicyan-1.2.3-triazol in Ger),



mw 119.09, N 58.81%. Exists in two modifications:

Yellow form, yel crysts (from w), mp 145-50° (dec); readily sol in w, alc & eth; insol in ligroin & petr eth; was prepd by reacting $H_2N.CH(CN).C(CN){:}NH$ or $H_2N.C(CN){:}C(CN).NH_2$ (diamino-maleonitrile or tetramer hydrocyanic acid) with HNO_2

Colorless form, crysts (from w), mp 145-50° (dec); readily sol in w, alc & eth; insol in ligroin; obtd on sublimation of the yel form at 140°

This compd forms several cryst salts; its *Silver salt,* AgC_4N_5, deflgr on heating (Refs 1 & 2)

Refs: 1) Beil **26**, [160] 2) E. Griszkiewicz-Trochimowski, RoczChem **1**, 468-78 (1921) & CA **17**, 1425 (1923)

Di(cyclohexanecarbonyl)-peroxide. See Bis(hexahydrobenzoyl)-peroxide in Vol 2, p B143-L

1,8-Dicyclohexylamino-3,6-diazaoctane, $C_{18}H_{42}N_4Cl_4$; crysts (from w), mp 299.4-.7°; isolated as the tetrahydrochloride, is the parent compd of its nitro deriv:

1,8-Dicyclohexylnitramino-3,6-dinitro-3,6-diazaoctane,

$C_6H_{11}[N(NO_2).CH_2.CH_2]_3N(NO_2)C_6H_{11}$; mw 484.47, N 23.13%; crysts (from boiling HNO_3, and then hot pyridine), mp 208.6-209.3°; can be detonated by frictional impact; was prepd from its parent compd by reacting with 95% HNO_3, acetic anhydride & acetic acid at 20-25° (Ref 2)

Refs: 1) Beil-not found 2) J.A. Harpham et al, JACS **72**, 342 (1950) & CA **45**, 1048 (1951)

7,7'-Dicyclohexyl-7,7'-butadiene-6,6'-dihydroperoxide. See under Diacetylenic Dihydroperoxides

Di(cyclohexyl)-ethylenediamine and Derivatives

Di(cyclohexyl)-ethylenediamine or N,N'-Dicyclohexyl-1,2-diaminoethane,

$C_6H_{11}.NH.CH_2.CH_2.NH.C_6H_{11}$, is the parent compd of its nitrated derivs:

N-Nitroso-N'-nitroso-N,N'-dicyclohexyl-ethylenediamine or N,N'-Dicyclohexyl-1,2-dinitrosoaminoethane,

$C_6H_{11}.N(NO).CH_2.CH_2.N(NO).C_6H_{11}$; mw 282.38, N 19.84%; crysts (from nitromethane, ethyl acetate, abs alc, abs eth, or petr eth), mp 144.3-144.6°; was prepd by nitrating N,N'-dicyclohexyl-1,2-diaminoethane dinitrate with 70% HNO_3 & $NaNO_2$ at 25° (Ref 2)

N-Nitro-N'-nitroso-N,N'-dicylohexyl-ethylenediamine or N,N'-Dicyclohexyl-N-nitro-N'-nitroso-1,2-diaminoethane,

$C_6H_{11}N(NO_2).CH_2.CH_2.N(NO).C_6H_{11}$; mw 298.38, N 18.78%; cryst (from benz or alc), mp 148-149.9°; was prepd by treating N,N'-dicyclohexyl-N-nitro-1,2-diaminoethane-mononitrate suspended in aq $NaNO_2$ with chloroform & acetic acid, or by treating a slurry of dicylohexyl-imidazolidine dinitrate with abs HNO_3 & acetic anhydride (Ref 3)

N,N'-Dinitro-N,N'-dicyclohexyl-ethylenediamine or N,N'-Dicyclohexyl-1,2-dinitroaminoethane,

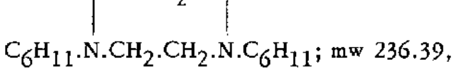
$C_6H_{11}N(NO_2).CH_2.CH_2.N(NO_2).C_6H_{11}$; mw 314.38, N 17.82%; crysts (from 70% HNO_3.

then isopropy ether, and pyridine or chlf, chlf-petr eth or benz-petr eth), mp 212-13°; was obtd by nitrating N,N -dicyclohexyl-1,2-diaminoethane dihydrochloride with 99% HNO_3 at 0° & acetic anhydride in acetic acid (Refs 2 & 3)

The expl props of the nitrated derivs of Dicyclohexyl-ethylenediamine were not investigated

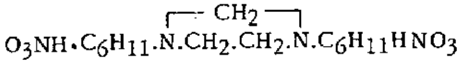
Refs: 1) Beil-not found 2) J.A. Harpham et al, JACS **72**, 342(1950) 3) J.L. Boivin & G.F. Wright, CanJRes **28B**, 221, 223(1950)

1,3-Di(cyclohexyl)-imidazolidine,



$C_6H_{11}.\overbrace{N.CH_2.CH_2.N}^{CH_2}.C_6H_{11}$; mw 236.39, N 11.85%; liq, fr p 13-15°, bp 125-26° at 0.5 mm & 137-38° at 0.7 mm press; was obtd by evaporating to dryness under reduced press a soln of N,N'-dicyclohexyl-1,2-diaminoethane, its dihydrochloride & formaldehyde in abs alc; forms a dinitrate:

1,3-Di(cyclohexyl)-imidazolidine Dinitrate,



$O_3NH{\cdot}C_6H_{11}.\overbrace{N.CH_2.CH_2.N}^{CH_2}.C_6H_{11}HNO_3$; mw 362.42, N 15.46%; wh ppt, mp 143-44° & after 2 hrs 147-48°; was obtd by chilling its parent compd in abs alc at -60° & adding cold abs HNO_3 (Ref 2); decomp in presence of moisture to the dinitrate salt of N,N'-Dicyclohexyl-1,2-diaminoethane

Its expl props were not reported

Refs: 1) Beil-not found 2) J.L. Boivin & G.F. Wright, CanJRes **28B**, 216, 220(1950)

Didi. Ger & Swiss abbr for Diethyleneglycol Dinitrate (DEGDN)

Didi(Pulver). Ger & Swiss designation of a DEGN proplnt

Di-p-diazoamidophenol or Di(4-dinitrophenyl)-triazene. See under Diphenyl-triazene and Derivatives

Di(β,γ-dihydroxypropyl)-ether Trinitrate or Dipropylol-ether Tetranitrate. See Tetranitrodiglycerol, under Diglycerol

N,N'-Di(β,γ-dihydroxypropyl)-oxamide Tetranitrate. See N,N'-Bis(2,3-dihydroxypropyl)-oxamide Tetranitrate in Vol 2, p B141-L

Di(2,6-dimethyl-pyridino)-diazido Copper Salt, $[(N_3)_2Cu(C_7H_9N)_2]$; mw 361.88, N 30.96%; explodes at 202-203° but not by impact; can be prepd by dissolving $Cu(N_3)_2$ in the appropriate amine; by addg NaN_3 to an amine soln of the appropriate Cu salt; and by rearranging of the cuprate of the appropriate amine (Ref 2)

Refs: 1) Beil-not found 2) A. Cirulis & M. Straumanis, JPraktChem **162**, 307(1943) & CA **38**, 1909(1944)

Di(3,5-dinitro-1,3,5-triazacyclohexylmethyl)-ether, called in CA **1,1'-(Oxydimethylene)-bis[hexahydro-3,5-dinitro-s-triazene.** See Bis(1,3,5-triaza-3,5-dinitro-cyclohexylmethyl)-ether in Vol 2, p B159-L

Dieckerhoff Powders. Mixtures of BkPdr with at least 15% of one/or several alkali metal picrates. The BkPdr could be substituted by a mixture of K nitrate and sulfur

Ref: Daniel (1902), 205

Diego de Alava y Viamont. A Spanish ballistician of the 16th century who published in 1590 in Madrid, a fundamental treatise on artillery. His life and achievements, as well as contents of the above treatise, are briefly discussed in the Ref

Ref: V. Montojo, MAF **13**, 764(1934)

Diego Uffano. A Spanish ballistician of the 17th century who published in 1613, in French, an important treatise entitled: "De l'Usage de l'Artillerie". His life and achievements are

briefly discussed in the Ref
Ref: V. Montojo, MAF **13**, 765-66 (1934)

Dielectric Properties of Explosives. In many experiments with expls it is necessary to rapidly heat the expl. The common technique is to introduce heat by conduction. Experiments of rapidly heating expls are difficult to treat theoretically because the temp is not known at a small fraction of a second after the start of heat conduction. As a result, interest is found in the rapid body heating of expls where large thermal gradients can be avoided. The most obvious applicable technique is "dielectric heating" (Ref 1)

For projects involving nuclear irradiation of expls and studies of the feasibility of using dielectric heating for detg induction times & related experiments, it was considered desirable that various electrical props should be known, such as resistivity, dielectric constant, dissipation factor & dielectric strength. A literature search indicated that little work of this nature has been done with expl materials. Further, such electrical measurements can be used as supplementary criteria for evaluating the purity, homogeneity and, with the exception of the dielectric strength determination, as possible non-destructive tests of expls. Hence the data listed in the following table were detd at Pic Arsn (Ref 2) for the common & readily avail expls which are believed to be of interest in research

Resistivity. Machined cylinders (3/4 x 3/4 inch) were desiccated for several days after being coated on both ends with conducting silver paint. Measurements were made with a Terra-ohmeter at both 100 & 400 volts over which range the samples appeared to have no significant voltage coefficient

Dielectric Constant. This property can be detd by measuring the capacitance of a

DIELECTRIC PROPERTIES OF COMMON HIGH EXPLOSIVES

EXPLOSIVE	RESISTIVITY[a] ohm-cm	PELLET[g] THICKNESS INCHES	DIELECTRIC STRENGTH BREAKDOWN VOLTAGE, KV	VOLTS/ mill	DIELECTRIC CONSTANT 100C	100KC
Baratol[b]	1.79×10^{11}	0.380	5.5	145	3.543	3.471
Composition B[c]	3.59×10^{10}	0.125	23	182	3.562[d]	3.297[d]
Octol (75/25-HMX/TNT)	2.99×10^{10}	0.040	5.5	139	3.121	3.013
PBX[e]	∞	0.0385	32	831	2.523	2.517
PBX-9404[f]	4.19×10^{10}	0.0385	22	560	3.679	3.414
TNT	8.97×10^{10}	0.040	3.8	94	5.167	2.849

[a] Measurements made on machined pellets 3/4 x 3/4 inch
[b] 76/24/0.06/0.1 - $Ba(NO_3)_2$/TNT/stearoxyacetic acid/NC
[c] 59.5/39.5/1 - RDX/TNT/wax
[d] Measurements made on pellets 0.125 inch
[e] 91/8.5/1.5 - RDX/dioctylphthalate/polystyrene
[f] 94/3/3 - HMX/NC/tris β-chloroethyl phosphate
[g] All pellets cast, except PBX & PBX-9404 which were pressed

fixture having typically parallel plates with the dielectric material occupying the volume between the plates, then removing the dielectric and measuring the air-spaced plates' capacitance. The ratio of the capacitances, thus measured, is a good approximation of dielectric constant

Wafers of the six types of expls were machined to 0.039 ± 0.0015" and to a diam of 2.5+0.002". Aluminum discs, machined to a diam of 5 inches and a thickness of 0.5", ground & polished to a flatness exceeding that of the wafers, acted as parallel capacitors and were connected to the measuring equipment by Cu strips of low conductance. Measurements were made on a General Radio Model 716C capacitance bridge, using a GR Model 1302A RC oscillator as a driving source for frequencies under one kilocycle, and a Hewlett Packard Model 650A test oscillator plus a HP Model decade amplifier for higher frequencies. For the Composition B samples, a Kiethley Model Electrometer and DC Amplifier were used to measure time constants of the loaded vs unloaded fixtures. All measurements were made at a RT of 75°F and at 100 cycles, 1,10 & 100 kilocycles

Dissipation Factor. This is defined as the ratio of the energy dissipation to the energy stored in the dielectric per cycle, or as the tangent of the loss angle. Dissipation factors less than 0.1 may be considered equal to the power factor of the dielectric. Values of dissipation factor and capacitances vs frequency of HE's are given in Ref 2, p 5

Dielectric Strength. Except for Composition B, this measurement was made on the same desiccated samples used in dielectric constant measurements. Aluminum electrodes, 3/4 x 3/4 inch, polished & edges rounded to 1/16 inch radius, were mounted on Lucite plates. These were connected across a 0.1 microfarad 40 kilovolt capacitor charged thru a 20 megohm resistor by a continuously variable 0-40 kilovolt power supply. The wafers were placed between these electrodes and voltage was applied at the rate of rise of 1 kilovolt per 2 secs and the voltage noted at which breakdown occurred. Two or more tests were performed on each expl, but average values are reported in the table. Tests were made on two different thicknesses of Comp B since there was some question as to the validity of the values obtd on samples 0.0405 inch thick. Hence, measurements reported were obtd on 0.125 inch wafers. See Table for data

Although the probability of detonation of these samples was small, all tests were conducted behind a barricade (Ref 2)

Dielectric props of various azides are reported in Ref 3

Refs: 1) A. Mackenzie, "Calculation of Maximum Rate of Dielectric Heating of Explosives", PATM **1115** (December 1962) 2) E.E. Walbrecht, "Dielectric Properties of Some Common High Explosives", PATM **1170** (April 1963) 3) G.C. Turrell, "Microwave Spectra and Dielectric Properties of Various Azides", Final Rept of Howard University, Washington, DC (1963) (Contract DA-44-009-Eng-4763)

Dielectrics and Dielectric Constants. A *dielectric* is an insulating material for electricity which may be characterized roughly as having an electrical conductivity less than 10^{-6} mhos/cm. Materials with conductivities in the range 10^{-6} to 10^{+3} mhos/cm are considered to be *semiconductors*. A *dielectric constant* or *specific inductive capacity* of a material is a measure of its ability to store electrostatic field energy in the presence of an electrical field and may be defined as the ratio of an electrical capacity of a condenser containing the material to the capacity of the same condenser with the material replaced by vacuum

Among dielectrical materials may be mentioned: wood, mica, asbestos, glass, porcelain, rubber, phenolic resins, polystyrene, plastics, oil & silicone fluids

See Dielectric Properties of Explosives

Refs: 1) Kirk & Othmer **5** (1950), 51-75 (Dielectrics) 2) Kirk & Othmer **11**, 2nd edit (1966); pp 776ff (Insulation, Electric)

Diels-Alder Synthesis. An additive combination of "dienes" (qv) with substances of the grouping >C=C-CO-R. For the description of this synthesis see Refs

Refs: 1) Thorpe **8**(1947), 497 (under Nitroölefins) 2) Hickinbottom (1948), 33-4 & 216-17 3) Fieser & Fieser (1950), 300-302 4) A. Wassermann, "Diels-Alder Reactions", Elsevier, NY (1965), 114pp 5) J. Hamer, "1,4-Cycloaddition Reactions. The Diels-Alder Reaction in Heterocylic Syntheses", Academic Press, NY (1967), 500pp

"dien". Designation for diethylenetriamine

Dienes. Unsaturated organic compds of general formula C_nH_{2n-2}, containg two double bonds and belonging to the group of polyenes. Typical examples are: diolefins, such as allene(propadiene), C_3H_4; divinyl(1,3-butadiene), C_4H_6; piperylene(1,4-pentadiene), C_5H_8; isoprene(2-methyl-1,3-butadiene), etc

Dienes may be subdivided into cumulative, $>C:C:C<$; conjugated, $>C:\underset{|}{C}.\underset{|}{C}:C<$; and isolated, $>C:C(CH_2)\ C:C<$

While cumulative and isolated dienes resemble in their properties olefins, the conjugated dienes show greatly enhanced chemical reactivity compared with olefins. The conjugated dienes can be oxidized by air or oxygen and sometimes the reaction may proceed with explosive violence. In cases when no expln takes place, the resulting peroxide may be isolated and examined. In many cases such peroxides proved to be explosive. For example, *Butadiene Peroxide* explodes with great violence

When treated with nitric acid, some of the dienes form expl compds, and when treated with nitrogen peroxide, the double bonds are usually broken and NO_2 group joins to each carbon

Some dienes may be polymerized to form rubber-like substances. For example divinyl and isoprene are used for the manuf of synthetic rubber

Refs: 1) Karrer (1950), 58-66 2) F.C. Whitmore, "Organic Chemistry", Van Nostrand, NY (1951), 63-4, 97 & 225 3) Kirk & Othmer **7**, 2nd edit (1965), pp 64ff (Diene Polymers)

Diénite. An explosive mixture of powdered PA 70-90 & DNT 30-10%, patented in 1880
Refs: 1) Daniel (1902), 205 2) Colver (1918), 323

Diethanolamine. See Diethylolamine in this Vol

Di(ethanolamine)-trinitrate. See Bis (β-nitroxyethyl)-amine Nitrate in this Encycl, Vol 2, p B129-L

Diethanol-aminobenzene. See Diethylol-aminobenzene in this Vol

Diethanol-ammonium-nitrate. See Diethylol-ammonium-nitrate in this Vol

Diethanol-ethylenediamine. See Diethylol-ethylenediamine in this Vol

Diethanol-oxamide. See Diethylol-oxamide in this Vol

Diethanol-peroxide. See Diethylol-peroxide in this Vol

Diethanol-piperazine. See Bis(hydroxyethyl)-piperazine in Vol 2, p B146-R

Diethanol-propyleneamine. See Diethylol-propyleneamine in this Vol

Diethanol-sulfamide. See Diethylol-sulfamide in this Vol

Diethoxy-azobenzene and Its Dinitro- and Hexanitro- Derivatives. See under Azophenetole in Vol 1, pA656-R

Diethoxyazoxybenzene and Its Trinitro- Derivative. See under Azoxyphenetole in Vol 1, p A670-R

1,9-Diethoxypentamethylene-2,4,6,8-tetranitramine; 3,13-Dioxa-5,7,9,11-tetranitro-5,7,9,11-tetraazapentadecane (CA nomenclature); or **1,9-Diethoxy-2,4,6,8-tetranitro-2,4,6,8-tetrazanonane** (called by McKay et al)(Refs 1 & 2)
$C_2H_5O.CH_2.N(NO_2).CH_2.N(NO_2).CH_2.N(NO_2).-CH_2.N(NO_2).CH_2.OC_2H_5$; mw 400.31, N 27.98%; wh crysts (fractionally crystd), mp 166-67°; was obtd by refluxing for 10 hrs 1,9-dinitroxy pentamethylene-2,4,6,8-tetranitramine (qv) with 95% alc, then cooling, filtering & fractionally crystallizing the product

Refs: 1) A.F. McKay et al, CanJRes **27B**, 467(1949) 2) W.J. Chute et al, CanJRes **27B**, 509, 519(1949) & CA **43**, 9074(1949) 3) G.F. Wright & W.J. Chute, BritP 615793 (1949), p 2 & CA **43**, 9079(1949) 4) G.F. Wright & W. J. Chute, USP 2678927(1954) & CA **49**, 7606-07(1955)

1,7-Diethoxy-2,4,6-trinitro-2,4,6-triazaheptane or 3,11-Dioxa-5,7,9-trinitro-5,7,9-triazatridecane (CA nomenclature),
$C_2H_5O.CH_2.N(NO_2).CH_2.N(NO_2).CH_2.N(NO_2).-CH_2.OC_2H_5$; mw 338.28, N 24.85%; crysts (from alc), mp 79-80.5° & 82° (Ref 2); insol in w; sol in alc & eth; obtd by refluxing 1,7-dinitroxy-2,4,6-trinitro-2,4,6-triazaheptane (qv) with 99% ethanol for 25 min and diluting with water. Its expl props were not reported

Refs: 1) W.J. Chute et al, CanJRes **27B**, 514(1949 & CA **43**, 9074(1949) 2) K.W. Dunning & W.J. Dunning, JChemSoc **1950**, 2925, 2927 & CA **45**, 6643(1951)

DIETHYLAMINE AND DERIVATIVES

Diethylamine (called Diäthylamin in Ger), $(C_2H_5)_2NH$; mw 73.14, N 19.15%; clear, col basic liq having an ammoniacal odor, fr p -50°, bp 55.4°, d 0.7056 at 20°, n_D 1.3867; Qcombstn 9995 cal/g; miscible with water; prepd either from ethyl ether & NH_3 or ethyl alcohol & NH_3, the molar ratio & catalyst used controls the type of ethylamine (mono-, di- or tri-) produced. Diethylamine forms numerous salts & addn compds. It is widely used in the prepn of rubber vulcanization accelerators, in the prepn of 2,4-D weed killer, and in org syntheses

Ref: Beil 4, 95, (345), [590] & {187}

Nitroso- and Nitroderivatives of Diethylamine

N-Nitroso-diethylamine, $(C_2H_5)_2N.NO$; mw 102.14, N 27.43%; lt-yel, aromatic smelling oil, bp 174.5°, d 0.9428 at 20° & 0.8842 at 86.7°, n_D 1.4379 at 20°; other props & methods of prepn are given in Beil

Ref: Beil 4, 129, (358), [617] & {233}

N-Nitro-diethylamine, $(C_2H_5)_2N.NO_2$; mw 118.14, N 27.31%; almost colorless, mild smelling liq, bp 203-05°, d 1.057 at 15°; can be prepd by heating at 55° diethylamine nitrate with 100% HNO_3 in acetic anhydride in the presence of $ZnCl_2$

Ref: Beil 4, 130 & {233}

Di(2,2,2-trinitroethyl)amine; Bis(2,2,2-trinitroethyl)amine or 2,2,2,2',2',2'-Hexanitrodiethylamine (HeNDEtA), $[(O_2N)_3C.CH_2]_2NH$; mw 343.14, N 28.58%; ndls (from chlf), mp 107° & 116° (Ref 3); can be prepd by treating 2 mols of trinitroethanol, dissolved in an equal vol of methanol, with 1 mol of gaseous ammonia at RT, or by treating equivalent quantities of formaldehyde & nitroform with NH_3 (Refs 2 & 3):

$2HCHO + 2(O_2N)_3CH + NH_3 = 2H_2O + [(O_2N)_3C.CH_2]_2NH$ This compd might be suitable as an ingredient of expl or proplnt compns. Ville (Ref 3) reported it to be sensitive to impact and capable of violent & spontaneous expln

Refs: 1) Beil-not found 2) F.R. Schenck & G.A. Wetterholm, SwedP 148217(1954); BritP 729469(1955); USP 2731460(1956) & CA **50**, 1893, 7125(1956) 3) J. Ville, MP **42**, 23, 26(1960) & CA **55**, 18109(1961)

Di(2,2,2-trinitroethyl)-nitramine; Bis(2,2,2-trinitroethyl)nitramine (BTNEN); or N,2,2,2,2',2',2'-Heptanitrodiethylamine, Code name **HOX,** $[(O_2N)_3C.CH_2]_2N.NO_2$; mw 388.14, N 28.85%; wh crysts (from chlf), mp 94-96°, decomposes rapidly above its mp, d 1.91 at 20°; was first synthesized by German scientists (Ref 2); can be prepd by reacting tetranitromethane with NH_3 in the presence of H_2O_2, the resulting ammonium nitroform is then treated with formaldehyde (Ref 3)

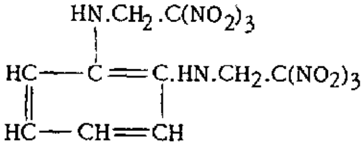
This expl has an impact sensitivity value of 12-15cm for 50% explns with 2kg wt; heat of combustion 1355cal/g; ignition temp 200° for 5 sec value; and rate detonation 7180 m/sec at d 1.5 & 8520m/sec at d 1.9g/cc (Ref 3)

BTNEN is reported to be more powerful & more brisant than TNT. It was recommended for use in mixts with Al (Ref 2)

Refs: 1) Beil-not found 2) G. Römer, "Report on Explosives", PBL Rept **85160** (1946) 3) D.L. Kouba & R.H. Saunders, "Summary Report, Task A, High Explosives", Hercules Powder Co, Navy Contract NOrd-11280 (Dec 1951) 4) J.A. Young et al, IEC **48**, 1375-78 (1956) & CA **50**, 16274 (1956) (Rept Heat of Combustion at const vol & 25°C 486kcal/mol)

1,2-Di(ethylamino)-benzene, 1,2-Bis(ethylamino)-benzene; or o-Phenylene-bis(ethylamine), $C_6H_4(HN.C_2H_5)_2$, may be considered as the parent compd of its bis(trinitro) deriv, although not used to prep it

1,2-Di(trinitroethylamino)-benzene; o-Bis (2,22 trinitroethylamino)-benzene; or o-Phenylene-bis(trinitroethylamine),



; mw 434.24, N. 25.81%; ppt, mp 70°; was prepd by condensing o-phenylenediamine in MeOH & (O_2N) C.-CH_2OH also MeOH, chilling the mixt and addg H_2O to ppt the product. The compd has an O bal of -48% and an impact sensitivity (2kg wt) of 65cm (Ref 2)

Refs: 1) Beil-not found 2) R.H. Saunders, USP 2996547 (1961) & CA **55**, 26447 (1961)

NOTE: See also Diethylaniline and Derivatives in this Vol

Di(ethylamino)-diazido Copper Salt, $[(N_3)_2Cu(C_2H_5.NH_2)_2]$, mw 237.75, N 47.13%; blue solid, burns & crepitates in a flame, explodes when placed on a hot plate but not on slow heating; does not detonate by impact; stable in water; low elec conductivity; can be prepd by dissolving $Cu(N_3)_2$ in the amine soln and addg acid, MeOH or ether to ppt the product; or by addg NaN_3 to an amine soln of the Cu^{II} salt (Ref 2)

Refs: 1) Beil-not found 2) M. Straumanis & A. Cirulis, ZAnorgChem **251**, 345-46 (1943) & CA **37**, 6574 (1943)

Diethylaminoethylol Dinitrate,

$(O_2NO)CH_2.CH_2.N(C_2H_5)_2.HNO_3$; mw 225.20, N 18.66%; crysts, mp 60°; was prepd by Barbière (Ref 2) by stirring diethylaminoethanol, $HOCH_2CH_2N(C_2H_5)_2$, into fuming HNO_3 in an ice bath, and evaporating the excess HNO_3 in vacuo at a temp less than 40°. Accdg to Fakstrop & Christiansen (Ref 3), the mixt invariably explodes when most of the HNO_3 is evapd. They proposed an alternate method: after addg the reagents during 2 hrs, the mixt is diluted with MeOH, pptd with eth, allowed to stand overnight, the solvs are decanted, and the ppt is washed with solvs

Refs: 1) Beil-not found 2) J. Barbiere, BullFr **11**, 470 (1944) & CA **40**, 2110 (1946) 3) J. Fakstrop & J. Christiansen, ActaChemScand **5**, 968-69 (1951) & CA **46**, 7522 (1952)

Diethylaminoethylguanidine, $C_5H_{18}N_4$, may be considered as the parent compd of its nitro deriv, although not used to prep it:

1,1-Diethyl-3(2-nitraminoethyl)-2-nitroguanidine or N-(β-Nitraminoethyl)-N'-diethyl-N''-nitroguanidine,

$NH.CH_2.CH_2.NH.NO_2$

$C(:N)NO_2$

$N(C_2H_5)_2$; mw 248.25, N 33.86%; crysts (from 95% alc), mp 152.5° (dec); was obtd when 1-nitro-2-nitrimino-imidazolidine was covered with diethylamine and allowed to stand at RT for 3 days, the mixt acidified and refrigerated for 2 days, and the ppt collected (Ref 2).

Its expl props were not detd

Refs: 1) Beil-not found 2) A.F. McKay et al, CanJChem **33**, 1199(1955) & CA **50**, 4130 (1956)

3-(β-Diethylaminoethyl)-α-sym-triazole Dipicrate. See Vol 1, p A209-L

Diethylammonium Diazidonitratocuprate, $(C_2H_5)_2NH_2[Cu(N_3)_2NO_3]$; mw 283.74, N 39.49%; crysts, explodes weakly at 214° expl in contact with concd H_2SO_4, but not by impact; can be best prepd by reaction of the ammonium azide & $Cu(NO_3)_2$ (Ref 2)

Refs: 1) Beil-not found 2) M. Straumanis & A. Cirulis, ZAnorgChem **252**, 9-23(1943) & CA **38**, 3564(1944) [Describe many addn complex compds of $Cu(N_3)_2$, called Cuprates]

DIETHYLANILINE AND DERIVATIVES

Diethylaniline, Diethylphenylamine or Diethylaminobenzene, $C_{10}H_{15}N$, mw 149.23, N 9.39%. The following isomers are known:

N,N-Diethylaniline (called Diäthylanilin in Ger), $C_6H_5.N(C_2H_5)_2$, col to yel oil, existing as stable form, fr p -21.3° and as unstable form, fr p -34.4°; bp 216-17°, d 0.935 at 20°, n_D 1.5421 at 20°; sl sol in alc, chlf & eth; prepd by heating aniline hydrochloride with excess ethyl alcohol at 180° under pressure, or by reaction of aniline with triethyl phosphate in the presence of a catalyst. Diethylaniline is used in org synthesis and in the prepn of dyes. It forms numerous cryst salts & addn compds (Refs 1 & 5)

C-Diethylaniline (called Amino-diethylbenzol or Diäthylanilin in Ger), $H_2N.C_6H_3(C_2H_5)_2$. Three derivs are known: 2,4-Diethylaniline, liq, volatile with steam, bp 141° at 30mm press, d 0.9545 at 25°, n_D 1.5395 at 25°; sol in alc & eth; insol in w (Ref 2); 2,5-Diethylaniline, liq, bp 140-42° at 20mm & 128-30° at 11mm press; other props & prepn are given in Ref (Ref 2); and 2,6-Diethylaniline, liq, bp 235-36°, n_D 1.5461 at 20°; obtd by alkylation of aniline with ethylene in the presence of Al anilide in an autoclave at 600-800 psi & 300° over a 7-hr period (Refs 3 & 4)

Refs: 1) Beil **12**, 164, (158) & [92] 2) Beil **12**, 1174 & [642] 3) G.C. Ecke et al, JOC **21**, 712(1956) 4) A.J. Kolka et al, USP 2814646(1957) & CA **52**, 5463(1958) 5) Kirk & Othmer, 2nd edit **2** (1963), 412, 420

Azido Derivatives of Diethylaniline

Monoazidodiethylaniline, $C_{10}H_{14}N_4$, not found in Beil or in CA thru 1961

Diazidodiethylaniline or N,N-bis(2-azidodiethyl)-aniline, $C_6H_5N(CH_2.CH_2.N_3)_2$; mw 231.26, N 42.40%; liq, bp 132-34° at 0.2mm press, n_D 1.5748 at 25°; was prepd by refluxing for 24hrs a mixt of N,N-bis(2-chloroethyl)-aniline & NaN_3 in methanol, distilling off the methanol under reduced pressure, and extracting the residue with eth (Ref 2). No expl props were reported

Refs: 1) Beil-not found 2) A.H. Sommers & J.D. Barnes, JACS **79**, 3492(1957) & CA **51**, 16328(1957)

Triazidodiethylaniline, $C_{10}H_{12}N_{10}$, not found in Beil or in CA thru 1961

Nitroso- and Nitroderivatives of Diethylaniline

4-Nitroso-N,N-diethylaniline (called N.N-Diäthyl-4-nitroso-anilin, 4-Nitroso-diäthylanilin or p-Nitroso-diäthylanilin in Ger), $ON.C_6H_4.N(C_2H_5)_2$; mw 178.23, N 15.72%; grn prisms (from eth), grn lfts (from acet), or grn prisms having a violet surface shimmer, mp 84°, d 1.24 at 15°; sol in alc & eth; sl sol in w; can be prepd by nitrosation of diethylaniline in HCl with $NaNO_2$ soln and by other methods. It forms cryst salts & addn compds (Ref 1). No expl props were reported

Refs: 1) Beil **12**, 684, (338) & [365] 2) J.I.G. Cadogan, JChemSoc **1957**, 1079 & CA **51**, 10407(1957)

NOTE: No higher Nitroso derivs of Diethylaniline were found in Beil or in CA thru 1961

Mononitro-N,N-diethylaniline, $O_2N.C_6H_4.N(C_2H_5)_2$; mw 194.23, N 14.42%. The three possible isomers are described in Beil:

2-Nitro-N,N-diethylaniline, red-colored oil, fr p -37°; forms some unstable salts, such as Hydrobromide, $C_{10}H_{14}N_2O_2 + HBr$, hygr lfts (from alc), dec 160°; Hydrochloride, $C_{10}H_{14}N_2O_2 + HCl$, prisms (from alc), dec 156° ; Hydroiodide, $C_{10}H_{14}N_2O_2 + HI$, hygro ndls (from alc), dec 112°; and Picrate, $C_{10}H_{14}N_2O_2 + C_6H_3N_3O_7$, golden lfts, mp 119-20°, deflgr on rapid heating (Ref 1)
3-Nitro-N,N-diethylaniline, dk-yel oil, bp 288-90° ; forms a Picrate, crysts (from alc), mp 138° (Ref 2)
4-Nitro-N,N-diethylaniline, lt-grn crysts, mp 59° (Ref 3)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **12**, (341) 2) Beil **12**, 702 & (346) 3) Beil **12**, 715, (351) & [386]

Dinitro-N,N-diethylaniline, $(O_2N)_2C_6H_3N(C_2H_5)_2$; mw 239.23, N 17.57%. Three isomers are found in Beil:
2,4-Dinitro-N,N-diethylaniline, orn-col prisms or plts (from acet) or yel crysts, mp 80°, d 1.374 at 15°; readily sol in hot alc; sol in acet, chlf, CS_2, benz & hot eth; sl sol in petr eth (Ref 1)
2,5-Dinitro-N,N-diethylaniline, dk-red monoclinic crysts (from alc), mp 74-76°, d 1.362 at 18° (Ref 2)
3,4-Dinitro-N,N-diethylaniline, exists as α- modification or stable form, orn-colored crysts (from acet), mp 95°; and a β-modification or labile form, lt-yel lfts (from MeOH), mp - stable in dry state at RT but goes to α- form at 90° or above; forms an addn compd with 2,4-Dinitro-N,N-diethylaniline, $C_{10}H_{13}N_3O_4 + C_{10}H_{13}N_3O_4$, mp 57-59°, which, on crystn from chlf + petr, separates into its components (Ref 3)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **12**, 750, (362) & [406] 2) Beil **12**, 758, (365) & [413] 3) Beil **12**, 759, (366) & [414]

Trinitro-N,N-diethylaniline, $(O_2N)_3C_6H_2N(C_2H_5)_2$; mw 284.23, N 19.71%. Three isomers are known:
2,3,4-Trinitro-N,N-diethylaniline, crysts (from CCl_4), mp 132-33°; decomp to 2,4-dinitrophenylene-1,3-diamine on heating with alcoholic NH_3 to 125°; and to 2,4-dinitro-N^1,N^3-dimethylphenylene-1,3-diamine on heating with methylamine in alc at 100°; was obtd with the 2,4,5-trinitro deriv on nitrating 3,4-dinitrodiethylaniline with HNO_3 (Ref 1)
2,4,5-Trinitro-N,N-diethylaniline, yel crysts, mp 158°; for prepn see above; forms an eutectic with 2,3,4-Trinitro-N,N-diethylaniline (30%) having a mp 117° (Ref 2)
2,4,6-Trinitro-N,N-diethylaniline, ruby-red monoclinic prisms (from acet or benz), mp 163-64° & 166.5-67°, d 1.476 at 16°; sol in eth acet, acet, 96% alc, abs alc, MeOH, benz, chlf, eth pyridine, CS_2, CCl_4 & toluene; decomp by heating in KOH into Picric Acid & diethylamine; can be prepd by reacting picryl chloride & diethylamine in alcoholic soln (Refs 3, 4 & 5)

Their expl props were not reported

Refs: 1) Beil **12**, [419] 2) Beil **12**, (367) & [420] 3) Beil **12**, 764, (368) & [421] 4) P. van Romburgh, BerslagAkadWetenschappen-Amsterdam **33**, 329-32 (1924) & CA **19**, 978 (1925) 5) L. Desvergnes, MonSci **16** (5), 201 (1926) & CA **21**, 740 (1927)

NOTE: See also 1,2-Di(trinitroethylamino)-benzene under 1,2-Di(ethylamino)-benzene in this Vol

N,N-Diethylaniline Pentaazidodicuprate, $(C_{10}H_{16}N)[Cu_2(N_3)_5]$, mw 685.28, N 33.47% ; brn lustrous ndls, burns in a flame with a crackling noise (1 mg), larger amts detonate (0.1 g), mp - explodes with a loud report at 198-99°; readily decompd by dissolving in water; was prepd by reaction of $CuCl_2.2H_2O$ in MeOH, diethylaniline and a hot aq soln of NaN_3

Refs: 1) Beil-not found 2) A. Cirulis & M. Straumanis, Ber **76B**, 829 (1943) & CA **38**, 1972 (1944)

DIETHYLBENZENE AND DERIVATIVES

Diethylbenzene (called Diäthyl-benzol in Ger), $C_6H_4(C_2H_5)_2$; mw 134.21, H 10.51%. The three possible isomers are known:

1,2-Diethylbenzene, liq, fr p -31.2 to -32.1°, bp 183.4 to 185°, d 0.8801-0.8805 at 20°, n_D 1.5035-1.5039 at 20° (Ref 1)
1,3-Diethylbenzene, liq, fr p -83.8 to -85°, bp 181.1 to 181.6°, d 0.8640 at 20°, n_D 1.4955 at 20° (Ref 2)
1,4-Diethylbenzene, liq, fr p -42.8°, bp 183.8°, d 0.8620 at 20°, n_D 1.4947 at 20° (Ref 3)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **5**, 426, [327] & {964} 2) Beil **5**, 426, [327] & {965} 3) Beil **5**, 426, (206), [327] & {968}

Azido, $C_{10}H_{13}N_3$, and *Diazido*, $C_{10}H_{12}N_6$, derivatives of Diethylbenzene were not found in Beil or in CA thru 1961

Mononitrodiethylbenzene, $O_2N.C_6H_3(C_2H_5)_2$; mw 179.21, N 7.82%. Three isomers are known:
4-Nitro-1,2-diethylbenzene, yel oil, bp 139-41° at 10mm press, d 1.0852 at 22°, n_D 1.5440 at 25° (Ref 3)
4-Nitro-1,3-diethylbenzene, yel liq, having a pleasant odor, bp 112-14° at 3.8mm press, d 1.0644 at 25°, n_D 1.5271 at 25° (Ref 1)
2-Nitro-1,4-diethylbenzene, liq, bp 137-40° at 12mm press (Ref 2)

Other props & methods of prepn are given in Refs

Refs: 1) Beil **5**, 426, [327] & {968} 2) Beil **5**, 427 & {970} 3) Beil **5**, {965}

Dinitrodiethylbenzene, $(O_2N)_2C_6H_2(C_2H_5)_2$; mw 224.21, N 12.50%. Only one isomer is described in the literature:
3,5-Dinitro-1,2-diethylbenzene, yel prisms (from alc), mp 89-90°; was obtd, in addn to mononitro derivs, on nitrating 1,2-diethylbenzene (Ref 2). No expl props were reported

Refs: 1) Beil-not found 2) C.G. Alberti & V. Valcavi, Gazz **87**, 332, 340 (1957) & CA **52**, 14550 (1958)

2,4,6-Trinitro-1,3-diethylbenzene, $(O_2N)_3C_6H(C_2H_5)_2$; mw 269.21, N 15.61%; prisms (from petr), lfts (from alc), mp 62-63°; was prepd by nitrating 1,3-diethylbenzene with mixed acid in the cold for 4 hrs and then heating to 135° (Refs 1, 2 & 3). No expl props of this compd were reported

Refs: 1) Beil **5**, 426 2) B.N. Dolgov & N.A. Kuchumova, ZhurObshchKhim **20**, 445 (1950) & CA **45**, 566 (1951) 3) A.B. Kuchkarov & I.P. Tsukervanik, JGenChem (Russia) **20**, 485-89 (1950) & CA **45**, 3346 (1951) (English translation of Ref 2) 4) B.N. Dolgov & S.A. Bnatov, IzvestVysshikhUchebZavedeniiKhim i KhimTekhnol **2**, 714-19 (1959) & CA **54**, 7594 (1960) (Compd listed in Formula Indexes, but not found in abstract)

2,3,6-Trinitro-1,3-di (β-nitroxyethyl)-benzene, $(O_2N)_3C_6H(CH_2.CH_2.ONO_2)_2$; mw 391.21, N 17.90%; expl oil, impact sensitive; was prepd by DuPont by condensing Trinitro-m-Xylene with HCHO and nitrating the product

Ref: Blatt, OSRD **2014** (1944)

DIETHYLBENZIDINE AND DERIVATIVES

N,N'-Diethylbenzidine or 4,4'-Diethyldiaminobiphenyl, (called N.N'-Diäthyl-benzidin in Ger), $[-C_6H_4.NH.C_2H_5]_2$; mw 240.34, N 11.66%; plts or ndls (from alc), mp 115-16°; readily sol in benz, hot alc & eth; v sl sol in cold petr eth; can be prepd by reacting ethyl iodide in alc with benzidine under press on a water bath

Ref: Beil **13**, 222

N,N'-Dinitroso-N,N'-diethylbenzidine, $[-C_6H_4.N(NO).C_2H_5]_2$; mw 298.34, N 18.78%; yel lfts, mp 162.5-163.5°; mod sol in hot glac acet ac; less sol in hot petr eth; was prepd by nitrosating N,N -diethylbenzidine with $NaNO_2$ in dil HCl (Refs 1 & 2)

Refs: 1) Beil **13**, 233 2) E. Bamberger & M. Tichvinsky, Ber **35**, 4184 (1902)

N,N'-Di(2-nitroethyl)-benzidine or N,N'-Bis (2-nitroethyl)-benzidine, $[-C_6H_4.NH.CH_2.CH_2.NO_2]_2$; mw 330.34, N 16.96%; crysts(from EtOAc, MeOH & xylene), mp 177°; was obtd by addg dropwise an excess of nitroethylene (CH_2:$CHNO_2$) in an inert solv (eth, dioxane, xylene, Nitrobenz) to benzidine in the same solv and heating at 60° for 2 hrs (Ref 2). No expl props reported

Refs: 1) Beil-not found 2) H. Hopff & M. Capaul, Helv **43**, 1902, 1905, 1909 (1960) & CA **55**, 12282 (1961)

3,5,3',5'-Tetranitro-N,N'-diethylbenzidine, $[-(O_2N)_2C_6H_2.NH.C_2H_5]_2$; mw 420.34, N 20.00%; crysts (from methyl salicylate), mp 248°; was obtd by heating in a sealed tube 3,5,3',5'-

tetranitro-4,4'-dimethoxy(or diethoxy)-biphenyl & ethylamine at 100°(Refs 1 & 2). No expl props were reported

Refs: 1) Beil **13**, [109] 2) G. van Romburgh, Rec **41**, 41(1922) & CA **16**, 1238(1922)

N,N',3,5,3',5'-Hexanitro-N,N'-diethylbenzidine, $[-(O_2N)_2C_6H_2N(NO_2).C_2H_5]_2$; mw 510.33, N 21.97%; lt-yel ndls (from HNO_3), mp 230° (dec); was prepd by treating the tetranitro deriv with concd HNO_3 (Ref 2). No expl props were detd

Refs: 1) Beil **13**, [109] 2) G. Van Romburgh, Rec **41**, 41(1922) & CA **16**, 1238(1922)

NOTE: See also Diethylbiphenyldiamine and its Tetranitro Derivatives in this Vol

Diethylbenzylamine and Derivatives

Diethylbenzylamine (called Diäthylbenzylamin in Ger), $C_6H_5.CH_2.N(C_2H_5)_2$;mw 163.25, N 8.58%; clear oil, bp 211-12°; diffc sol in w; can be prepd by reacting diethylbenzylcinnamylammonium chloride with Na amalgam in water and by other methods; forms some cryst, unstable salts (Ref)

Ref: Beil **12**, 1021, (448) & [546]

Azido, $C_{11}H_{16}N_4$, and *Diazido*, $C_{11}H_{16}N_2O_2$, derivatives were not found in Beil or in CA thru 1961

Nitroderivatives of Diethylbenzylamine

Diethylnitrobenzylamine, $O_2N.C_6H_4.CH_2.N(C_2H_5)_2$; mw 208.25, N 13.45%. Three isomers are known:

Diethyl-2-nitrobenzylamine, yel liq, having a herring-like odor, bp 144° at 13mm & 175-77° at 42mm press; readily sol in common org solvs; v sl sol in w; forms a Picrate, $C_{11}H_{16}N_2O_2 + C_3H_3N_3O_7$, mp 122°(Ref 1)

Diethyl-3-nitrobenzylamine, yel liq, having a herring-like odor, bp 158° at 13mm & 206-208° at 42mm press; readily sol in common org solvs; v sl sol in w; forms a Picrate, mp 161° (Ref 2)

Diethyl-4-nitrobenzylamine, yel liq, having a herring-like odor, bp 160° at 13mm & 219-21° at 42mm press; readily sol in common org solvs; v sl sol in w; forms a Picrate, mp 131° (Ref 3)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **12**, 466 & [577] 2) Beil **12**, 466 & [578] 3) Beil **12**, 466 & [581]

N,N-Di(2-nitroethyl)-benzylamine, $C_6H_5.CH_2.N(CH_2.CH_2.NO_2)_2$; mw 253.25, N 16.59%; crysts (from petr eth + eth, below 10°, 7 times), mp 66°; was obtd by adding dropwise an excess of nitroethylene ($CH_2{:}CHNO_2$) in an inert solv to benzylamine at -20° for 30 mins, isolating by treating the yel oil in ether with HCl, converting the resulting hydrochloride to base with aq NaOAc and recrystallizing as above (Ref 2)

Refs: 1) Beil-not found 2) H. Hopff & M. Capaul, Helv **43**, 1905, 1908(1960) & CA **55**, 12282(1961)

NOTE: No higher nitrated derivs of Diethylbenzylamine were found in Beil or in CA thru 1961

Diethylbiphenyldiamine or 3,3'-Diethyldiaminobiphenyl, $C_{16}H_{20}N_2$, may be considered as the parent compd of its tetranitro deriv although not used to prep it:

4,6,4',6'-Tetranitro-N,N'-diethyl-3,3'-biphenyldiamine (called 4,6,4',6'-Tetranitro-3,3'-di(ethylamino)-diphenyl by van Alphen), $[-(O_2N)_2C_6H_2NH.C_2H_5]_2$; mw 420.34, N 20.00%; mp yel powd which turns quickly red on heating at 270-80°, mp 315-20° (dec); was prepd by heating 4,6,4',6'-tetranitro-3,3'-dichlorobiphenyl with ethylamine in alc for 5 hrs at 100°(Ref 2)

Refs: 1) Beil-not found 2) J. van Alphen, Rec **51**, 365-66(1932) & CA **26**, 2447(1932)

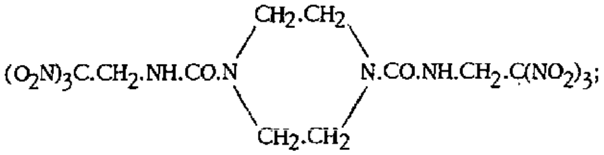
NOTE: No higher nitrated derivs of Diethylbiphenyldiamine were found in Beil or in CA thru 1961. See also N,N'-Diethylbenzidine and Derivatives in this Vol

N,N'-Diethylcarbanilide. See Centralite 1 in Vol 2, p C127-L

Diethyl Carbonate. See Ethyl Carbonate in Vol 6

Di(ethylcarboxamide)-piperazine, $C_{10}H_{20}N_4O_2$, may be considered as the parent compd of its hexanitro deriv although not used to prep it:

1,4-Di[N(2',2',2'-trinitroethyl)-carboxamide]-piperazine or N,N'-Bis(2,2,2-trinitroethyl)-piperazine-1,4-dicarboxamide,



mw 498.29, N 28.12%; crysts (from acet), mp - explodes at 188°. It was prepd as follows: Piperazine hexahydrate ($QH_2.6H_2O$, where QH_2 represents 1,4-piperazine & Q the 1,4-piperazinyl group) in water was neutralized with concd HCl, heated 1 hr at 50° with KCNO in w, and chilled to give $Q(CONH_2)_2$, mp 288-90° (dec). Formalin was added to $Q(CONH_2)_2$ suspended in saturated aq $Ba(OH)_2$. CO_2 was passed into the soln, $BaCO_3$ removed, the filtrate concd at RT, and acetone added to ppt $Q(CONHCH_2OH)_2$, mp - sintering 190-210°. Solns of the latter compd & $CH(NO_3)_2$, both in water, were mixed for 1 hr at 45°, filtered and the product collected. This compd was patented as an expl & propInt (Ref 2)
Refs: 1) Beil-not found 2) P.O. Tawney, USP 3038902 (1962) & CA **58**, 9098 (1963)

NOTE: This compd is listed in Vol 2 of this Encycl, p B160-R but not described because of its Confidential classification at that time in US

3,3'-Diethyldiaminobiphenyl. See Diethylbiphenyldiamine in this Vol

4,4'-Diethyldiaminobiphenyl. See N,N'-Diethylbenzidine in this Vol

Diethyldiborane, $B_2H_4(C_2H_5)_2$; mw 83.79; exists as *sym-*, vapor press 36mm at 0°, which does not decomp in 24 hrs at 25° & *unsym-*, vapor press 46mm at 0°, which 4.5% decompd in 0.5 hr, and 29.4% decompd in 16 hrs at 25°. *Sym*-diethyldiborane was separated by fractionating from the equilibrium mixt obtd by heating ethyldiborane. *Unsym*-diethyldiborane was prepd by allowing a mixt of diethyldiboranes to react with diborane (Ref 4). IR spectra are reported by Solomon et al (Ref 4), Wilson Jr & Shapiro (Ref 5), and Lehmann et al (Ref 6). When Diethyldiborane reacts with B_5H_9 at 25-65°, there is produced $B_5H_7(C_2H_5)_2$, mp 101°, n_D 1.4004 at 20°, useful as a high-energy fuel in bipropellant systems (Ref 8)

See also Diboranes in this Vol, and Boranes in Vol 2 of this Encycl under Boron Hydrides, p B253-Rff

Refs: 1) Beil-not found 2) H.I. Schlesinger, ZAnorgChem **225**, 407-09 (1935) & CA **30**, 4421 (1936) (Prepn from B_2H_6 & $B(C_2H_5)_3$ in 5:1 molar proportions) 3) T. Wartik & R.K. Pearson, JInorg & NuclearChem **5**, 250-51 (1958) & CA **52**, 10868 (1958) [Prepn from $NaBH_4$ in tetraethylene glycol dimethyl ether, $(-CH_2.CH_2OCH_2CH_2OCH_3)_2$, on standing with vinyl bromide, $CH_2{:}CHBr$] 4) I.J. Solomon et al, JACS **80**, 4520-23 (1958) & CA **53**, 4114 (1959) 5) C.O. Wilson Jr & I. Shapiro, AnalChem **32**, 78-81 (1960) & CA **54**, 14924 (1960) 6) W.J. Lehmann et al, JChemPhys **33**, 590-97 (1960); **34**, 476-83 (1961) & CA **55**, 4149, 26666 (1961) 7) H.G. Weiss, USP 2977390 (1961) & CA **55**, 18039 (1961) (Prepn) 8) M. Lichtenwalter & K.E. Harwell, USP 2979530 (1961) & CA **55**, 20960 (1961)

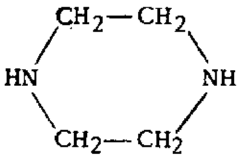
Diethyldihydroxyamine. See Diethylolamine in this Vol

N,N'-Diethyl-N,N'-diphenylurea. See Centralite 1 in Vol 2, p C127-L

N,N'-Diethyl-N,N'-diphenylurea-Nitroguanidine Complex (Centralite 1-NGu Complex), $H_2N.C({:}NH).NH.NO_2 + [H_5C_2.N.C_6H_5]_2CO$; lt-yel solid, mp softens at 160° & at 210° dec (Maquenne bloc); and shows liq phase at 160° & at 170° dec (capillary tube); was obtd when aq alc homogeneous soln saturated with both components in equiv concns at RT for 30 mins (Ref 2)
Refs: 1) Beil-not found 2) M. Parpaillon, MP **38**, 233-41 (1956) & CA **51**, 11717 (1957)

DIETHYLENEDIAMINE AND DERIVATIVES

Diethylenediamine, Piperazine or Hexahydropyrazine (called Piperazin, Hexahydropyrazin or Diäthylendiamin in Ger),



; mw 86.14, N 32.53%; col, deliq, transparent ndls (from hot alc), mp 104°, bp 140-46°; readily sol in w; mod sol in alc; insol in eth; can be prepd from N,N'-diphenyl piperazine by treating with $NaNO_2$ in cold HCl, warming the reaction products with $NaHSO_4$ at 80°, and heating with NaOH; forms numerous cryst salts, many of which are unstable on heating, for example: *Chlorate*, $C_4H_{10}N_2 + 2HClO_3$, lfts, dec 98-100°; *Perchlorate*, $C_4H_{10}N_2 + 2HClO_4 + H_2O$, mp 80°, explodes at 290° (Ref 1). Its *Dibromo-*, $BrNC_4H_8NBr$, yel prisms (from chlf + petr eth), mp - explodes violently at 79-80°; and *Dichloro Salt*, explodes at 80-85° (Ref 2)

Other props & methods of prepn are given in Beil

Refs: 1) Beil **23**, 4, (4) & [3] 2) Beil **23**, 14 3) Kirk & Othmer, 2nd edit **15** (1968), pp 638-46

Nitroso- and Nitroderivatives and Piperazine

N-Nitroso-piperazine, $HNC_4H_8N.NO$; mw 115.14, N 36.50%; known as its Nitrite, $C_4H_9N_3O + HNO_2$, grn crysts; v sol in water

Ref: Beil **23**, (7)

N,N'-Dinitroso-piperazine, $ON.NC_4H_8N.NO$; mw 144.14, N 38.87%; lt-yel lfts (from w), mp 156-58°, dec 260°; readily sol in hot dil alc; less sol in cold w & eth

Ref: Beil **23**, 14 & (7)

N,N'-Dinitro-piperazine, $O_2N.NC_4H_8N.NO_2$; mw 176.14, N 31.81%; ndls (from glac acet ac or eth acet), mp 215°, dec on prolonged heating at its mp; readily sol in hot xylol; mod sol in acet; sl sol in w, benz & chlf; can be prepd by treating piperazine hydrochloride with aq $AgNO_2$ soln, or by reaction of N,N'-dibenzoyl-sulfonyl-piperazine with anhyd HNO_3 (Ref 1). It is an expl compd not fully studied (Ref 2)

Refs: 1) Beil **23**, 15 & (7) 2) R.C. Elderfield, "Explosives from Hydroxy and Amino Compounds, OSRD **907** (Sept 1942), pp 18-19

Diethylenediamine Complexes

Diethylenediamine Complexes of Cobalt. Six complexes are listed in Vol 3, p C384

Di(ethylenediamino)-cadmium (II) Azide, $\{Cd[C_2H_4(NH_2)_2]_2\}(N_3)_2$; mw 325.37, N 26.55%; wh ppt, mp - melts & deflgr with a bright flame when heated on a Ni spatula; readily sol in cold w; decomp by dissolving in acids; was prepared by reacting Cd triethylene-diamine-sulfate with Barium Azide (Ref 2)

Refs: 1) Beil-not found 2) W. Strecker & E. Schwinn, JPraktChem **152**, 213-14(1939) & CA **33**, 5314(1939)

Di(ethylenediamino)-copper (II) Azide, $\{Cu[C_2H_4(NH_2)_2]_2\}(N_3)_2$; mw 267.79, N 31.39%; violet crysts, mp 180-82°, melts in a flame followed by expln, explodes in a preheated block at 212-16°; sol in w; sl sol in alc; insol in eth; was prepd from aq triethylenediamine-copper sulfate & Ba Azide (Refs 2 & 3)

Refs: 1) Beil-not found 2) W. Strecker & E. Schwinn, JPraktChem **152**, 211-12(1939) & CA **33**, 5314(1939) 3) M. Straumanis & A. Cirulis, ZAnorgChem **251**, 339(1943) & CA **37**, 6573(1943)

Dinitro-bis(ethylenediamine)cobalt (III) nitrates, cis- and trans- forms. Prepn and properties are given in Ref 2.

1) *cis-Dinitro-bis(ethylenediamine)cobalt (III) nitrate* cis-$[Co\ en_2(NO_2)_2]NO_3$, N 29.44%. Brown-yell cryst, may be prepd by treating potassium hexanitrocobaltate (III) with dil aq soln of ethylenediamine (abbr en), $C_2H_4(NH_2)_2$, followed by heating and cooling. The resulting nitrite cis-$[Co\ en_2(NO_2)_2]NO_2$ was oxidized to nitrate by treatment with concd HNO_3

2) *trans-Dinitro-bis(ethylenediamine)cobalt (III)- nitrate*, trans-$[Co\ en_2(NO_2)_2]NO_3$, N 29.44%. Yell cryst; may be prepd by adding ethylenediamine (70% aq soln), partially neutralized by HNO_3, to aq soln of $Co(NO_3)_2 6H_2O$ and $NaNO_2$. After passing a vigorous stream of air thru the soln for about 20 min, the reaction is complete and the nitrate precipitates out

Refs: 1) Beil-not found 2) H.F. Holtzclaw Jr et al, InorgSynth **4**(1953), 177-79

Diethyleneglycol and Derivatives

Diethyleneglycol [called Diäthylenglykol;

2.2'-Dihydroxy-diäthyläther; Bis(β-oxy-äthyl)-äther; β,β'-Dioxy-diäthyläther; or Glykol (oxy-äthyl)-äther in Ger], O(CH$_2$.CH$_2$.OH)$_2$; mw 106.12, O 45.23%; clear, col, practically odorless, syrupy, hygr liq, fr p -10.1 to -11.5°, bp 245°, flash p 275°F (OC), d 1.1122 at 25°, n_D 1.4469 at 20°; can be obtd as a co-product with ethylene glycol by the hydration of ethylene oxide, and also by reaction of ethylene glycol & ethylene oxide (Refs 1, 2 & 7)

Diethyleneglycol is used in plasticizers, humectants, solvs, brake fluids, antifreezes & softening agents. Its derivs are useful in many other applications (Refs 5 & 7)

The conditions of acceptance & examination of Diethyleneglycol intended for nitration are reported by Aubertein (Ref 3). Its determination in binary & ternary mixts with other glycols, by means of a refractometer is described by Lang (Ref 4)

Refs: 1) Beil 1, 468, [520]& {2090} 2) H. Moureau et al, MSCE **33**, 409(1947)(Prepn) 3) P. Aubertein, MP **32**, 65-71(1950) 4) F.M. Lang, MP **33**, 143-53(1951) 5) CondChemDict (1961), 375 6) Sax (1963), 709 7) Kirk & Othmer, 2nd edit **10**(1966), 646

Diethyleneglycol Dinitrate (DEGDN) (called Diäthylenglykol-dinitrat in Ger), O(CH$_2$.CH$_2$.ONO$_2$)$_2$; mw 196.12, N 14,29%; clear, colorless liq, having no odor, fr p -11.2 to -11.4°, bp 160-61°, volatile betwn 15 & 55°, d 1.3909 at 15°, $Q^v_{combstn}$ 548.7 kcal/mol; readily sol in chlf, MeOH, eth, acet, benz, toluene & glac acetic acid; almost insol in alc, CCl$_4$ & CS$_2$; prepd in 85% yield by nitrating pure diethyleneglycol (50 gms) with mixed acid (HNO$_3$ 50, H$_2$SO$_4$ 45 & H$_2$O 5% - 183.4 gms) at a temp of 10-15° (Refs 1, 2, 5 & 9). It was made in Germany by a continuous process (Refs 6, 7, 20 & 25)

DEGDN is relatively stable in storage at RT. It is definitely toxic when absorbed thru the skin, or its vapors are inhaled. The symptoms are similar to those produced by NG, affecting the heart & blood vessels (Ref 5)

DEGDN is itself expl in nature, but is so insensitive, it must be mixed with other nitrated compds to be useful as an expl. Its props are tabulated in Ref 26 and described in more detail by Urbański (Ref 25)

Hough (Ref 3) patented its prepn and of proplnts "based" on NC gelatinized with 20-25% DEGDN (Ref 4). Other uses in proplnts are described in patents by Achilles (Ref 18), Recherches Chimique SA (Ref 21) & Scharf (Ref 22) and in books by Fedoroff (Ref 20) & Urbanski (Ref 25). The advantages of using DEGDN in proplnts are due to its props of being a good gelatinizer for NC, and its high chem stability resulting in less hazards during processsing of proplnts than is possible with NG proplnts

Refs: 1) Beil 1, [521]& {2112} 2) W.H. Rinkenbach, IEC **19**, 925-27(1927) 3) A. Hough, BritP 341456(1929) & CA **25**, 5033(1931) 4) A. Hough, BritP 343107 (1929) & CA **27**, 2303(1933) 5) W.H. Rinkenbach & H.A. Aaronson, IEC **23**, 160-63(1931) & CA **25**, 1998(1931) 6) O.W. Stickland et al, "General Summary of Explosive Plants", PB Rept **925**(1945), p 57 (Prepn as practiced at German Krümmel Fabrik of DA-G) 7) R.A. Cooley, ChemInd **59**, 645(1946) & CA **41**, 863(1947) 8) A. Bresser, IndChemist **25**, No 289, 92(1949) & CA **43**, 4013(1949) (Industrial scale prepn) 9) P. Aubertein, MP **30**, 7-42(1948) & CA **45**, 353(1951) (Prepn & props of DEGDN and of several other glycol nitrates) 10) L. Médard, MP **31**, 131(1949) & CA **46**, 11685 (1952) (Impact sensitivity of DEGDN) 11) S. Pinchas, AnalChem **23**, 201(1951) & CA **45**, 3763(1951)(Detn by IR spectroscopy of DEGDN content in mixts with NG) 12) J. Boileau & M. Thomas, MP **33**, 155(1951) & CA **47**, 5200(1953)(Viscosity, density & refractive index of DEGDN) 13) F. Pristera, AnalChem **25**, 844(1953) & CA **47**, 9207(1953)(Analysis by IR spectroscopy of proplnts contg DEGDN) 14) J. Chosson, MP **33**, 357(1951) & CA **47**, 10315(1953) (Diffusion of DEGDN in solventless proplnts due to humidity) 15) H. Liogier, MP **35**, 199 (1953) & CA **49**, 12831(1955)(Hygroscopicity of solventless proplnts contg DEGDN or NG) 16) P. Tavernier, MP **35**, 233(1953) & CA **49**, 12832-33(1955)(Heat of formation of DEGDN and calorimetric potential of proplnts contg it) 17) N.A.P. Nilsson, USP 2737522(1956)

& CA **50**, 6796(1956)(Prepn of DEGDN by an improved method) 18) F.R. Achilles, GerP 963674(1957) & CA **54**, 2746(1960); USP 2852359(1958) & CA **53**, 1717-18(1959)[Shaped proplnts contg NC (12.1%N) 2.8, DEGDN 1.8 & TNT 5.3 parts] 19) Gorst (1957), 95 20) Fedoroff et al, PATR 2510(1958), Ger 36 21) RecherchesChimiques SA, BelgP 566271(1958) & CA **53**, 10766(1959); BritP 832137(1960) & CA **54**, 14690(1960)(Surface treatment with 4-24% by wt of DEGDN gives proplnts having low temp coefficents) 22) W. Scharf, USP 2995430(1961) & CA **55**, 26450(1961)(Composite propnt, contg DEGDN, having reduced flash, smoke & corrosion props) 23) CondChemDict(1961), 376 24) Sax(1963), 711 25) Urbański **2**(1965), 149-54 26) Tomlinson & Sheffield, AMCP **706-177**(1967), 103-06

Diethyleneglycol Perchlorate,
$HO.H_2C.H_2C.O.CH_2.CH_2.ClO_4$; mw 204.53, O 46.94%; col oil, very hygro, explodes violently on heating rapidly; was obtd from ethylene oxide & $HClO_4$ in the presence of ether at 0°(Refs **1** & 2)
Refs: 1) Beil **1**, 469 2) K.A. Hofmann et al, Ber **42**, 4394(1909) & CA **4**, 584(1910)

Diethylenetriamine (DETA) or Triazaheptane.
See Bis(aminoethyl)-amine in Vol 2, p 128-L

Diethylenetriamine Complexes of Cobalt.
See Cobalt (III) bis(diethylenetriamine) Perchlorate in Vol 3, p C383-R

Diethylether and Derivatives

Diethylether, Ethyl Ether, commonly known as **Ether** (called Diäthyläther, Äthyläther or Äther in Ger)' $O(C_2H_5)_2$; mw 74.12, O 21.59%; col, very volatile, highly flammable liq, having a sweet pungent odor & burning taste, fr p -116.3°, bp 34.5°, flash p (cc) -49°, autoignition temp 180-90°, expl range in air 1.85-48% by vol; d 0.7145 at 20° & 0.7019 at 30°, n_D 1.3527 at 20°, $Q_{combstn}$ 651.7 kcal/mol at 20°; miscible with alc, benz, chlf, petr eth, oils & many fat solvs; very sol in concd HCl, but may explode in contact with anhyd HNO_3; prepd commercially by the dehydration of ethyl alcohol by sulfuric acid (Refs 1,5 & 7)

The toxicity of Ether is low. A concn of 35000ppm in air produces unconsciousness in 30 mins. Recovery from exposure to sublethal concns of Ether is rapid & generally complete if one is promptly removed from the area. The greatest hazard in industry is fire, resulting in possible expln of Ether. If ether fires occur, CO_2, CCl_4 & dry chemical fire extinguishers may be used. Automatic sprinklers, deluge systems & high-pressure water spray are also effective in extinguishing Ether fires (Refs 1, 6 & 7)

Uses. Diethylether is probably the most important member of the ether family. It has a wide range of uses in the chem industry: a good solv for fats, waxes, oils, resins, dyes, gums & alkaloids; mixed with alc, it is an excellent solv for Guncotton, Collodion Cotton & Pyroxylin; a denaturant for alcohols; a starting fuel for diesel engines; a general anesthetic in surgery; and as an inert reaction medium in Grignard & Würtz-Fittig reactions (Refs 5 & 7)

Exposure of Ether to air & light leads to the formation of nonvolatile & expl peroxides. Under vigorous oxidation or by action of inorg acid, Ether undergoes cleavage. It can be oxidized to acetic acid. With halides, Ether forms a variety of cryst addn compds. Ether can be nitrated safely in the vapor phase to give methyl nitrate, ethyl nitrate & 2-nitroethyl ethyl ether

For addnl info, see in Vol 6 of Encycl under Ether

Refs: 1) Beil **1**, 314, (158), [311] & {1289} 2) P.W. Danckwortt, ZAngChem **40**, 1317(1927) & CA **22**, 687(1928)(Spontaneous expln of a mixt of liq air & Ether) 3) J. van Alphen, Rec **49**, 492-500(1930) & CA **24**, 2980(1930) (Explosive reactions with ethers & esters) 4) A. Rieche, AngChem **44**, 896-99(1931) & CA **26**, 1124(1932)(Auto-oxidation of Ether) 5) CondChemDict(1961), 452 6) Sax(1963), 809-10 7) Kirk & Othmer, 2nd edit **8**(1965), 477-86

Diethylether, Peroxide Formation in. It has been known since the last century that partial oxidation of Diethyl Ether and of some other ethers, such as Di-isopropyl Ether, produces compds which are very expl & dangerous to handle because of their instability. Several explns reported in the literature are attributed to formation, or presence of Peroxides of Ether

In storing Ether it should be remembered that sunlight and the absence of moisture are conducive to peroxide formation, while the presence of impurities (such as water & alcohol) tend to inhibit it. The presence of as little as 0.2% Peroxide is dangerous and such an amt can be formed sometimes after a few weeks storage. Ethers contg more than 0.2% Peroxide should never be distilled. In general, the Peroxides have higher bp's than Ether, and, hence, concentrate in the distillation vessel until an expln takes place. In the case of Ethers boiling above 100°, it is presumed that the Peroxides decomp before they can accumulate, and no explns have been reported in handling them

Before distilling a low-boiling Ether, the presence of Peroxide should be detd. If Ether has been stored for some time, the presence of Peroxide should be assumed. In order to destroy Peroxide, a small quantity of a reducing agent, such as a sulfite, should be added & the mixt shaken. Another method is to add one vol of 25% aq KOH to each 200 vols of Ether before proceeding with distillation. Brandt (Ref 2) reported that Peroxides may be removed by shaking Ether with concd $FeSO_4$ soln, acidified with H_2SO_4, followed by distilling over NaOH. Bailey & Roy (Ref 5) found that Na amalgam is the best inhibitor to prevent formation of Peroxides. Storage in amber colored glass also delays Peroxide formation

It must be remembered that purified Ether again forms Peroxides on standing in contact with air. No inhibitor is completely successful, and the presence of Peroxides should always be detd before distilling Ether

Refs: 1) Beil **1**, 320, [321] 2) L. Brandt, ChemZtg **51**, 981-83(1927) & CA **22**, 869(1928) (Expln of Ether contg Peroxide; detection & removal of Peroxide) 3) R. Robertson, Chem & Ind **52**, 274 (1933) (A lab expln attributed to Peroxide in Isopropyl Ether) 4) G.T. Morgan & R.H. Pickard, Chem & Ind **55**, 421 (1936) (Explns arising from Di-isopropyl ether Peroxide) 5) W.G. Bailey & A. Roy, JSCI **65**, 421-22(1946) & CA **41**, 2899(1947) 6) CondChemDict(1961), 453

NOTE: See Diethyl Peroxide in this vol

Diethylether Analytical Procedures. US specifications for tech grade Diethylether are set forth in MIL-E-199A(1), dated Dec 1966. Ref 7 reports the requirements for Anesthesia grade, USP grade, and Absolute ACS grade Diethylether. Marqueyrol & Loriette (Ref 2) suggest comparing the behavior of a test sample with that of pure Ether, prepd by washing ordinary Ether with aq H_2SO_4 and drying it by shaking with metallic Na, and passing the vapors thru a column of lime. The following tests are recommended:

a) A very dil soln of chromic acid (dil K_2CrO_4 + dil H_2SO_4) should turn blue when agitated with Ether sample. Examine for the presence of Diethyl Peroxide

b) A sample of 10ml of Ether is vigorously shaken in a test tube with 1ml of 10% KI soln. Place tube in a dark place and examine after 1 hr. Coloration in the Ether or KI layer shows the presence of Peroxide or Ozone

c) Impure Ether reddens litmus and decolorizes an appreciable amt of $KMnO_4$ soln

Ploux & Parpaillon (Ref 5) discuss various methods for the prepn of pure Ether meeting the specifications of the Service de Santé de l'Armee. Their method consists of treating Ether used for the manuf of proplnts with $KMnO_4$ in alk soln, followed by distilling into a receiver contg dry calcium chloride

In Bofors Analytical Methods (Ref 6), Ether is examined for appearance, specific gravity, aldehydes, peroxides, sulfur compds & acidity. The presence of Peroxides is detd by adding a few drops of distilled water to 10mm of Ether on a watch glass and allowing to evaporate at RT. To the remaining water a small cryst of KI is added. In the presence of Peroxides the water will be colored yellow from free iodine

Refs: 1) Beil **1**, 321, (163), [322] &{1305} 2) M. Marqueyrol & P. Loriette, MP **21**, 277-80(1924) 3) L. Brandt, ChemZtg **51**, 981-83 (1927) & CA **22**, 869 (1928) (Peroxide detection by KI - starch & Ti^{++++} solns) 4) A. Lalande, MP **25**, 421-35 (1932-33) (Freezing points & densities of binary mixts of Ether-Alcohol) 5) (?). Ploux & (?). Parpaillon, MP **30**, 255-61 (1948) 6) AB Bofors, Nobelkrut, "Analytical Methods for Powders and Explosives", Bofors, Sweden (1960), 86 7) CondChemDict (1961), 453

1-Azidodiethylether (called β-Azido-diäthyläther in Ger), $N_3.H_2C.H_2C.O.C_2H_5$; mw 115.14, N 36.50%; col liq, having an odor resembling that of chloroether, and in steam is pungent & sweet, bp 49° at 25mm press, d 0.9744 at 25°; does not explode when thrown on a hot plate, the vapor burning with a luminous, wh flame; was prepd by reaction of 1-azidoethanol & ethyl iodide with dry Ag oxide in the dark for 2 days (Refs 1 & 2)

Refs: 1) Beil **1**, (171) 2) M.O. Forster & S.H. Newman, JCS **97II**, 2579 (1910) 3) K.A. Kornev & S.B. Serebryaniĭ, ZapiskiInstKhim-AkadNauk, URSR, InstKhim **6**, Nos 3 & 4, 343-50 (in Russian), 350-51 (in German) (1940) & CA **35**, 2469 (1941) (Prepn in 10-15% yield from ethyl iodide, allyl bromide & alcolate of azidoethanol) 4) Y.A. Sinnema & J.F. Arens, Rec **75**, 1423-28 (1956) & CA **51**, 8683 (1957) (Describe prepn of 1-azido-1-ethoxyethane [$H_3C.CH(N_3)O.C_2H_5$] and 1,1-diazido-1-ethoxyethane [$H_3C.C(N_3)_2OC_2H_5$])

Mononitrodiethylether [called Äthyl-(2-nitro-äthyl)-äther or 2-Nitro-diäthyläther in Ger], $O_2N.H_2C.H_2C.O.C_2H_5$; mw 119.12, N 11.76%; col liq, having a sharp odor & bitter taste, bp 178° & 72° at 12mm press & d 1.079 at 25° (Ref 4); n_D 1.4160 at 25° (Ref 4); was first prepd by heating β-iododiethylether with Ag nitrate, and later by heating either 36 parts of 1,2-dinitroethane or 8 parts 2-nitroethylnitrate with ethanol (Ref 1); and by other methods (Refs 2, 3 & 4)

Refs: 1) Beil **1**, 339 & {1365} 2) A.E. Wilder-Smith et al, USP 2416650 (1947) & CA **41**, 3478 (1947) (Prepn of 2-Nitroethyl ethers) 3) A. Lambert et al, JCS **1947**, 1475 & CA **42**, 4907 (1948) 4) H.B. Hass & D.E. Hudgin, JACS **76**, 2692 (1954) & CA **49**, 8786 (1955)

Di(2-nitroethyl)ether or Bis(2-nitroethyl) ether (called β.β-Dinitro-diäthyläther or Äthyl-β.β-dinitroäthyl-äther in Ger), $(O_2N)_2HC.H_2C.O.C_2H_5$; mw 164.12, N 17.07%; colorless oil, bp 100° at 11mm press; sol in org solvs; v sl sol in w; was obtd on acidification if its *Potassium salt*, $K.C_4H_7N_2O_5$, lt-yel ndls (from alc), prepd by reacting trinitroethane with K ethoxide; forms a *Bromine deriv*, $C_4H_7N_2O_5Br$, col oil, bp 103-04° at 13mm press; and a *Silver salt*, $Ag.C_4H_7N_2O_5$, which is probably expl (See Refs)

Refs: 1) Beil **1**, 340 2) A. Hantzsch & A. Rinckenberger, Ber **32**, 637 (1899) & JCS **76I**, 404 (1899) 3) J. Meisenheimer, Ber **36**, 434 (1903) & JCS **84I**, 223 (1903) 4) J. Meisenheimer & M. Schwartz, Ber **39**, 2546 (1906) & JCS **90I**, 618-19 (1906) 5) J.A. Chenicek, USP 2818453 (1957) & CA **52**, 8539 (1958) (Solv useful to separate aromatic hydrocarbons from their mixts)

Diethyl-ethylenediamine and Derivatives

N,N'-Diethyl-ethylenediamine [called N.N'-Diäthyl-äthylendiamin or α.β-Bis(äthylamino)-äthan in Ger], $H_5C_2.HN.CH_2.CH_2.NH.C_2H_5$; mw 116.20, N 24.11%; liq, bp 151-52° & 65.5° at 35mm press; was prepd by reaction of ethylene dichloride & ethylamine; forms some salts which are unstable on heating: $[Cu(C_6H_{16}N_2)_2](ClO_4)_2$, blue-violet crysts, deflgr on heating; and $[Ni(C_6H_{16}N_2)_2(H_2O)_2]-(ClO_4)_2$, reddish-blue plts (from MeOH), deflgr on heating (Refs 1 & 2)

May be considered the parent compd of its hexanitro deriv, but was not used to prep it

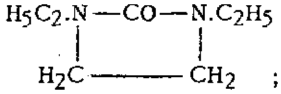
Refs: 1) Beil **4**, 251, [690] & {513} 2) R.A. Donia et al, JOC **14**, 948 (1949)

N,N'-Di(2,2,2-trinitroethyl)-ethylenediamine or N,N'-Bis(2,2,2-trinitroethyl)-ethylenediamine, $(O_2N)_3C.H_2C.HN.CH_2.CH_2.NH.CH_2.C(NO_2)_3$; mw 386.20, N 29.02%; yel solid, mp 97°; was obtd by treating 1 mol of ethylenediamine, $(H_2N.CH_2.CH_2.NH_2)$, with 2 moles of trinitroethanol [$(O_2N)_3C.CH_2OH$], at RT to give a 75-80% yield (Ref 2). Its expl props were not investigated, but this compd may find use as an ingredient of explosives or proplnts

Refs: 1) Beil-not found 2) F.R. Schenck

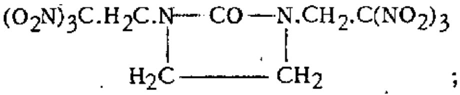
& G.A. Wetterholm, SwedP 148217(1954); BritP 729462(1955); USP 2731460, Example 4(1956) & CA **50**, 1893, 7125(1956)

N,N'-Diethyl-ethyleneurea or 1,3-Diethyl-2-imidazolidinone,



; may be considered as the parent compd of its hexanitro deriv, although not used to prep it:

N,N'-Di(2,2,2-trinitroethyl)-ethyleneurea or 1,3-Bis(2,2,2-trinitroethyl)-2-imidazolidinone (CA Nomenclature),



; mw 412.19, N 27.19%; col ndls (from MeOH), mp 177-78° (dec); was prepd by heating for 20 min on a steam bath bis(hydroxymethyl)-2-imidazolidinone, MeOH & $CH(NO_3)_3$. This compd can be detonated by impact and was patented as an expl (Ref 3)

Refs: 1) Beil-not found 2) I.J. Schaffner & P.O. Tawney, US Rubber Co February Summary Rept (Feb 1949), p 2 and Progress Rept **No 5**, p 31 (Contract NORD 10129) 3) H.A. Hageman, USP 3035062(1962) & CA **57**, 11203(1962)

Diethyl-ethylmalonate (called Äthylmalonsäure-diäthylester or Diäthyl-äthylmalonat in Ger), $C_2H_5.CH(COOC_2H_5)_2$, liq, bp 211° at 748mm press (dec) & 121° at 40mm press, d 1.0012 at 25°, n_D 1.4157 at 25° (Ref 1). May be considered as the parent compd of its trinitro deriv, although not used to prep it:

Diethyl-trinitroethylmalonate, $(O_2N)_3C.CH_2.CH(COOC_2H_5)_2$; mw 323.11, N 13.01%; liq, bp 110-11° at 1mm press, n_D 1.4538 at 20°; was prepd by reacting methylenemalonic acid with nitroform (Ref 2). It is probably an expl compd (Ref 3)

Refs: 1) Beil **2**, 644, (275), [570] & {1698} 2) US Rubber Co Progress Rept **No 7** (1 May - 1 Aug 1949), p 13 (Contract NORD 10129) 3) The trinitro compd was not found in Beil or in CA thru 1966

Diethylguanidine and Derivatives

Diethylguanidine (called Diäthyl-guanidin in Ger), $C_5H_{13}N_3$; mw 115.18, N 36.49%. Two isomers are known:

N,N-Diethylguanidine, $(C_2H_5)_2N.C(:NH).NH_2$; hygr ndls (from eth), mp 88-89°; readily sol in w, alc & benz; sl sol in eth; forms cryst salts with acids (Ref 2)

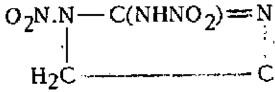
N,N'-Diethylguanidine, $C_2H_5HN.C(:NH).NH.C_2H_5$; lt-yel oil; forms cryst salts (Ref 1)

Other props & methods of prepn are found in Beil

Refs: 1) Beil 4. 116 & {219} 2) Beil **4**, 121, [611] & {223}

N,N-Diethyl-3-nitroguanidine or 1,1-Diethyl-3-nitroguanidine, $(C_2H_5)_2N.C(:NH).NH.NO_2$; mw 160.18, N 34.98%, crysts (from alc), mp 92-94°; was prepd by reacting 2-methyl-1 (or 3)-nitro-2-thiopseudourea $[CH_3SC(:NH).NH.NO_2]$, with diethylamine at RT (Ref 3)

N,N'-Diethyl-2-nitroguanidine or 1,3-Diethyl-2-nitroguanidine, $C_2H_5HN.C(:N.NO_2).NH.C_2H_5$; crysts (from 95% alc), mp 115-16°; was obtd by reacting 1-nitroso-2-nitramino-2-imidazoline,



, with ethylamine in water below 30° (Ref 2)

Their expl props were not reported

Refs: 1) Beil-not found 2) A.F. McKay, JOC **16**, 1849(1951) & CA **46**, 9097(1952) 3) L. Fishbein & J.A. Gallaghan, JACS **76**, 1878(1954) & CA **49**, 6838(1955)

NOTE: No higher nitrated derivs of Diethylguanidine were found in Beil or in CA thru 1961

Di(2-ethylhexyl) adipate or Dioctyl Adipate (DOA) $C_4H_8[COOCH_2CH(C_2H_5)C_4H_9]_2$; mw 370.56, O 17.27%; lt-colored oil, fr p -70°, bp 417°, 214° at 5mm press, flash p 405°F, d 0.9268 at 20°, n_D 1.4472; insol in water (Ref 3). DOC is a solv for NC & resins, and can be used as a substitute for Di(2-ethylhexyl) sebacate in the binder or plasticizer for Composition C-4 (See Vol 3, p C485-L). US military requirements & tests are described in Ref 2

Refs: 1) Beil-not found 2) US Specification MIL-D-13383 (April 1954) 3) CondChemDict(1961), 377 4) Sax(1963), 714

Di(2-ethylhexyl) azelate or Dioctyl Azelate (DOZ) $(CH_2)_7[COOCH_2CH(C_2H_5)C_4H_9]_2$; mw 412.63, O 15.51%; odorless liq; fr p -67.8°, bp 376°, flash p (COC) 430°F, d 0.919 at 20°, n_D 1.4472 (Refs 2 & 3). DOZ is used as a plasticizer in some composite rocket proplnts (Ref 3)
Refs: 1) Beil-not found 2) CondChemDict (1961), 378 3) Conf "Propellant Manual" SPIA/M2(1962), Unit Nos 591, 650 & 651 4) Sax(1963), 714

Di(ethylhexyl)-phthalate. See Bis(ethylhexyl)-phthalate in Vol 2, p B142-R and also Dioctyl Phthalate in this Vol

Di(2-ethylhexyl) sebacate or Dioctolyl Sebacate (DOS), $(CH_2)_8(COOC_8H_{17})_2$; mw 426.66, O 15.00%; pale, straw-colored liq, fr p -55°, bp 248° at 9mm press, flash p 213°(COC)(Ref 3) & 410°F (Ref 4), d 0.913 at 25°, n_D 1.447 at 28°; insol in water (Refs 3 & 4)

DOS is used in the binder for Composition C-4 (See Vol 3, p C485-L)
Refs: 1) Beil-not found 2) US Specification MIL-D-10692 (Nov 1950) 3) CondChemDict(1961), 379 4) Sax(1963), 715

Diethylhydrazine and Derivatives

Diethylhydrazine (called Diäthyl-hydrazin in Ger), $C_4H_{12}N_2$; mw 88.15, N 31.78%. Two isomers are known:
N,N-Diethylhydrazine, $(C_2H_5)_2N.NH_2$; hygr liq, having an etherish ammonia-like odor, bp 96-99°; readily sol in w, alc, eth, chlf & benz (Ref 1)
N,N'-Diethylhydrazine, $C_2H_5.HN.NH.C_2H_5$; liq, bp 84-86° at 758mm press; forms a Hydrochloride salt, $C_4H_{12}N_2 + HCl$, plts (from aq HCl), mp 160°(dec)(Ref 2)

Other props & methods of prepn are given in Beil
Refs: 1) Beil **4**, 550, (561) & [959] 2) Beil **4**, 550 & {1730} 3) G. Desseigne & (?). Cohen, FrP 1186902(1959) & CA **56**, 320 (1962)(Prepn of N,N-Diethylhydrazine); MP **41**, 487-89(1959)

N,N'-Di(2,2,2-trinitroethyl) hydrazine or 1,2-Bis(2,2,2-trinitroethyl) hydrazine, $(O_2N)_3C.CH_2.HN.NH.CH_2.C(NO_2)_3$; mw 358.15, N 31.29%; prepn from $(O_2N)_3C.CH_2OH$ & aq $N_2H_4.H_2O$ described in patents (Ref 2), but no info is given in abstract
Refs: 1) Beil-not found 2) G.A. Wetterholm, SwedP 148217(1954); BritP 729469(1955); USP 2731460(1956) & CA **50**, 1893, 7125 (1956)

Diethylhydroxylamine and Derivatives

Diethylhydroxylamine (called N.N-Diäthyl-hydroxylamin in Ger)
$H_3C.H_2C.N(OH).CH_2.CH_3$; mw 89.14, N 15.71%; foul-smelling liq, fr p -10°, bp 130-35°(dec), d 0.8612 at 25°, n_D 1.4173 at 25°; readily sol in w, alc, eth, chlf & benz; can be prepd by oxidizing diethylamine with H_2O_2, or by other methods (Refs)
Refs: 1) Beil **4**, 536, [954] & {1717} 2) CondChemDict(1961), not listed 3) Sax (1963), not found 4) Kirk & Othmer, 2nd edit **11**(1966), 503

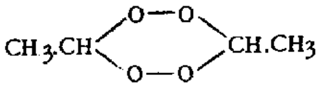
N-Nitroso-O,N-Diethylhydroxylamine, $C_2H_5.N(NO).O.C_2H_5$; mw 118.14, N 23.71%; yel oil, bp 56-57° at 15mm press, dec & explodes on heating above 200°; was obtd by reacting O,N-diethylhydroxylamine hydrochloride with $NaNO_2$ in cold water
Ref: Beil **4**, {1717}

Di(2,2,2-trinitroethyl)hydroxylamine or N,N-Bis(2,2,2-trinitroethyl)hydroxylamine, $(O_2N)_3C.CH_2.N(OH).CH_2.C(NO_2)_3$; mw 359.14, N 27.30%; yel solid; was prepd by treating 2 mols of 2,2,2-trinitroethanol with 1 mol hydrazine hydrate in aq soln (Ref 2). This compd may be suitable as an ingredient of expl or proplnt compns
Refs: 1) Beil-not found 2) F.R. Schenck & G.A. Wetterholm, SwedP 148217(1954); BritP 729469(1955); USP 2731460, example 5 (1956) & CA **50**, 1893, 7125 (1956)

Diethylhyponitrite or Ethylhyponitrite (called Diäthylhyponitrit, Äthylhyponitrit or Diazoäthoxan in Ger), $C_2H_5.ON:NO.C_2H_5$; mw 118.14, N 23.71%; oil, bp - cannot be distilled even under reduced press, explodes on heating at 80-82°, on impact, or by friction; d 1.0326 at 20°, n_r 1.4047 at 20°; readily sol in alc, eth & benz; insol in w, HCl & NaOH; was prepd by treating ethyliodide in ether with Ag hyponitrite in a freezing mixt (Refs)

Refs: 1) Beil. **1**, 328 & {1321} 2) J.R. Partington & C.C. Shah, JCS **1932**, 2595 3) L. Seed, BritP 795824(1958) & CA **53**, 219(1959)(Prepn of Diethylhyponitrite & other esters of hyponitrous acid by oxidation or dehydrogenation, below 50°, of O-ethers of NH_2OH having general formula NH_2OR, where R is an org radical. Oxidizers may be HgO, halogens, peroxides, or azo compds) 4) G. Scott, BritP 823103(1959) & CA **54**, 6549(1960)(Prepn of Ethylhyponitrite & other hyponitrous esters by treating Ag hyponitrite with an org halide in the presence of a hydrogen halide catalyst) 5) CondChemDict(1961), not listed 6) Sax (1963), not listed

Diethylidene Diperoxide; Diacetaldehyde Peroxide; Acetaldehyde Superoxide; or Ethylidene Peroxide (called 3.6-Dimethyl-1.2.4.5-tetroxan or Diäthyliden-diperoxyd in Ger),



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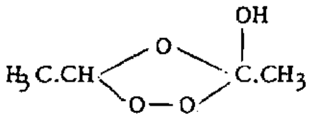


;

mw 120 10, O 53.29%; resin which explodes when touched by a glass rod; insol in w; was obtd by keeping bis-(hydroxyethyl)-peroxide under vacuum for several days (Refs 1 & 2)

The *dimeric* peroxide, $(CH_3CHOO\text{-})$, crysts, mp 22.5-23°, n_D 1.4160, a highly expl compd, has been prepd by several methods: a) allowing an ethereal soln of α-hydroxyalkylhydroperoxide to stand over P_2O_5 b) vacuum treatment of 1-hydroperoxyethanol at 70-75° c) dehydration of di-(1-hydroxyethyl) peroxide with P_2O_5 and d) from butene ozonide

Refs: 1) Beil **19**, [449] 2) H Wieland & A. Wingler, Ann **431**, 315(1923) & CA **17**, 2558(1923) 3) A. Rieche & R. Meister, Ber **64**, 2335(1931) & CA **26**, 82(1932) 4) A. Rieche & R. Meister, Ber **72**, 1933, 1938(1939) & CA **34**, 712(1940) 5) J. Malicki, PrzmyslChem **32**, 7-9(1953) & CA **48**, 4428(1954) 6) J.d'Ans et al, AngChem **66**, 633-35(1954) & CA **49**, 10832(1955) 7) E.G.E. Hawkins, "Organic Peroxides", Van Nostrand, Princeton, NJ(1961), pp 138 & 148

Diethylketene Peroxide (called Diäthylketen Peroxyd in Ger), $C_6H_{10}O_3$; mw 130.14, O 36.88%; oil; was prepd by treating an ethereal soln of diethylketene, $(C_2H_5)_2C:C:O$, with H_2O_2 at −20°; an extremely expl compd; decomp into CO_2 & diethylketone if allowed to stand in ether for several days (Refs 1 & 2)

Refs: 1) Beil. **1**, (384) 2) H. Staudinger & J. Maier, Ann **401**, 297(1913) & CA **8**, 496 (1914)

Diethyl Lead Diazide (called Diäthylbleidiazid in Ger), $(C_2H_5)_2Pb(N_3)_2$; mw 349.39, N 24.10%; cryst, mp - detonates with a bright flame when heated; was prepd by adding dropwise aq Na azide to an aq soln of diethyl lead chloride at 50°(Refs 1 & 4). Diethyl lead chloride, $(C_2H_5)_2PbCl_2$, can be prepd by the action of tetraethyl lead on chlorine, followed by crystn from alc (Refs 1, 2 & 3)

Diethyl Lead Diazide is easily detonated by an elec current. It req a *firing current* of 0.35-0.42 amp vs 0.40-0.47 for LA. It was patented by Burrows et al (Ref 4) as a component of ignition compositions for use in elec caps. Its *bridge-wire lag* is 0.00052 sec, *induction period* is 0.00032 sec and *total lag* is 0.0009 sec vs 0.00020,

and 0.0002 sec respectively for LA
Refs: 1) Beil 4, (599), [1021] & {1952} 2) G. Grüttner & E. Krause, Ber **49**, 1426 (1916) 3) H. Gilman & J. Robinson, JACS **52**, 1977(1930) 4) L.A. Burrows et al, USP 2105635(1935) & CA **32**, 2357(1938)

Diethyl Lead Dibenzoate, $(C_2H_5)_2Pb(OOC.C_6H_5)_2$; mw 507.55, O 12.61%; col ndls (from benz), mp 168°, explodes mildly when heated rapidly; sol in hot alc & benz; insol in w; can be prepd by reaction of benzoic acid with tetraethyl lead & a small piece of silica gel on a water bath for ½ hr, or by refluxing for 1½ hrs an excess of benzoic acid in alc with diethyl lead sulfite (Ref 2)
Refs: 1) Beil-not found 2) R. Heap et al, JCS **1951**, 661, 662 & CA **45**, 8972(1951)

Diethyl Mercury (called Diäthylquecksilber in Ger), $(C_2H_5)_2Hg$; mw 258.73; mobile liq having a penetrating odor, bp 97-99° at 25mm press & 159° at 760mm press, d 2.4583 at 24°, Qvaporzn 1012kcal/mol, n_D 1.5487 at 20°; almost insol in w; sl sol in alc; sol in eth; was prepd by the method of Gilman & Brown (Ref 2) from ethyl magnesium iodide & $HgCl_2$ and by other methods (Ref 1). Konton (Ref 3) found that heating Diethyl Mercury with equal wts of various phenols in sealed tubes and opening the tubes resulted in explns in most cases
Refs: 1) Beil **4**, 679, (612), [1048] & {2004} 2) H. Gilman & R.E. Brown, JACS **52**, 3314-17(1930) & CA **24**, 4758(1930) 3) M.M. Koton, ZhObshchKhim **19**, 730-33(1949) & CA **44**, 1044(1950) 4) Sax(1963), 717(Compd is highly toxic)

Diethyl-methylenediamine and Derivatives

N,N'-Diethyl-methylenediamine, $H_5C_2.HN.CH_2NH.C_2H_5$, may be considered as the parent compd of it dinitro deriv, although not used to prep it:

N,N'-Dinitro-N,N'-diethyl-methylenediamine; N,N'-Bis(ethylnitramine)-methane; or 3,5-Dinitro-3,5-diazaheptane, $H_5C_2.N(NO_2).CH_2.N(NO_2).C_2H_5$; mw 192.18, N 29.16%; crysts (from hexane), mp 75.7-77.2°; was prepd by addg in small portions ethylenitramine to a chilled soln of paraformaldehyde in 90% H_2SO_4 and drowning the reaction mixt (Ref 2). Its expl props were not detd
Refs: 1) Beil-not found 2) L. Goodman, JACS **75**, 3019-20(1953) & CA **49**, 4630(1955)

Diethylnitramines. See under Diethylamine and Derivatives in this Vol

1,1-Diethyl-3(2-nitraminoethyl)-2-nitroguanidine. See under Diethylaminoethylguanidine in this Vol

Di(ethylnitrosamino)-azoxybenzene. See Bis(ethylnitrosamino)-azoxybenzene in Vol 2, p B142-R

DIETHYLOLAMINE AND DERIVATIVES

Diethylolamine; Diethanolamine; 2-Aminobutane-1,4-diol; or Diethyl-2,2'-dihydroxyamine; or Bis (2-hydroxyethyl)-amine [called Bis-(2-hydroxy-äthyl)-amin; 2.2'-Dihydroxy-diäthylamin or Diäthanolamin in Ger], $HN(CH_2.CH_2.OH)_2$; mw 105.14, N 13.32%; col prisms, becoming a viscous liq in humid atmosphere; active base, mp 28°, bp 271°(dec), flash p (OC) 280°F, d 1.101 at 20°, n_D 1.4776 at 20°; sol in w, alc & chlf; v sl sol in petr eth, benz & eth; forms numerous cryst salts & addn compds, some of which are unstable on heating. Other props & methods of prepn are found in Ref 1
Refs: 1) Beil **4**, 283, [729] & {689} 2) CondChemDict (1961), 372 3) Sax (1963), 704

Diethylol-nitrosamine or Di(2-hydroxyethyl)-nitrosamine, $ON.N(CH_2.CH_2.OH)_2$; mw 134.14, N 20.89%; liq, bp 100-120° at $2.6x10^{-5}$ mm press, n_D 1.4849 at 20°; was obtd when $NaNO_2$ in water was slowly added to diethylolamine in w, previously neutralized with 2N HCl (Refs 1 & 2)

Refs: 1) Beil **4**, {721} 2) E.R.H. Jones & W. Wilson, JChemSoc **1949**, 550

Diethylolamine Dinitrate; Di(nitroxyethyl)-amine or 2,2'-Iminodiethanol Dinitrate (CA nomenclature), $HN(CH_2.CH_2.ONO_2)_2$; known in the form of its salts: *Hydrochloride,* $C_4H_9N_3O_6.HCl$, plts (from acet ac), mp 162.5-63° (dec); *Nitrate* [called Di(nitroxyethyl) ammonium nitrate] $C_4H_9N_3O_6.HNO_3$, wh hygr crysts (from acet ac or acet), mp 120.5 & 127°, can be ignited and burns leaving a deposit of C; can be detonated by impact; readily sol in acet, MeOH, hot alc, w & nitromethane; insol in benz, chlf, eth & petr eth; was obtd by reaction of concd HNO_3 at low temp on diethylolamine; *Picrate,* $C_4H_9N_3O_6 + C_6H_3N_3O_7$, crysts (from toluene + eth), mp 136-37°

Refs: 1) Beil **4**, {692} 2) W.J. Chute et al, CanJRes **26B**, 96 (1948) & CA **42**, 4918 (1948) 3) R.N. Jones & G.D. Thorn, CanJRes **27B**, 854 (1949 (UV absorption spectrum of Nitrate) 4) G.F. Wright & W.J. Chute, CanP 479929 (1952) & CA **50**, 8747 (1956) 5) G. Desseigne, MP **35**, 117-20 (1953) & CA **49**, 14639 (1955) (Prepn & props of Dinitratodiethylammonium Nitrate) 6) L. Médard & M. Thomas, MP **36**, 97 (1954) & CA **50**, 3763 (1956) (Heat of combustion of Nitrate salt) 7) L. Médard, MP **36**, 93-96 (1954) & CA **50**, 6795 (1956) (Expl props of Bis(2-nitroxyethyl) ammonium nitrate) 8) G.F. Wright & W.J. Chute, CanP 479929 (1952) & CA **50**, 8747 (1956)

Di(nitroxyethyl)-nitrosamine or 2,2'-(Nitrosimine)-diethanol Dinitrate (CA nomenclature), $ON.N(CH_2.CH_2.ONO_2)_2$; mw 224.14, N 25.00%; crysts (from eth + petr eth), mp 46-47°, slowly dec spontaneously at 25°; readily ignites and burns, leaving a brn residue; its sensitivity to impact is 2.6xTNT; power by BM is 1.5xTNT; sol in most org solvs; insol in petr eth & w, the latter in which it is decompd rapidly on boiling; was prepd by the action of $NaNO_2$ in w & dil HNO_3 on Di(nitroxyethyl)-ammonium Nitrate (qv) at 0°

Refs: 1) Beil **4**, {721} 2) W.J. Chute et al, CanJRes **26B**, 96 (1948) & CA **42**, 4918 (1948) 3) G.F. Wright & W.J. Chute, CanP 479929 (1952) & CA **50**, 8747 (1956)

Di(2-nitroxyethyl)-nitramine, N,N-Bis(β-nitroxyethyl)-nitramine; Diethanolnitramine Dinitrate (DINA) or 2,2'-(Nitroimino)-diethanol Dinitrate (CA nomenclature) [called Bis-(2-nitryloxyathyl)-nitramin or Disalpetersäureester des Bis-(2-hydroxy-äthyl)-nitramins in Ger], $O_2N.N(CH_2.CH_2.ONO_2)_2$; mw 240.14, N 23.33%; crysts (from aq acet), mp 52.5°, dec at 165°, d 1.67 at 25°; heat capacity 0.38 cal/g, heat of fusion 23.5 cal/g, heat of combstn 1260-1350 cal/g; sol in glac AcOH, MeOH, alc, benz & eth; insol in w, CCl_4 & petr eth; can be prepd from diethylolamine by nitrating with 100% HNO_3 & acetic anhydride in the presence of HCl at 5-40° (Ref 1), and by other methods (Refs 3, 4, 5, 8, 10, 11, 13, 15, 18 & 20)

The toxicity of DINA was studied by Sunderman (Ref 7). Incomplete observations on humans exposed to DINA did not show abnormalities in high blood press, urinalysis or blood count. The subjects complained of headaches similar to those caused by NG

DINA is a very powerful expl, similar to NG in this respect. It is ca 150% as powerful as TNT, but much more sensitive to impact, resembling Tetryl in this property. Explosive & other props of recrystd DINA were detd and/or tabulated by Livingston & Rinkenbach (Ref 2):

Brisance Index, 1.24 vs 1.23 for Tetryl. See Sand Test in Vol 1, pp XXI–XXII

Density, cast chge 1.658 & pressed at 3000 psi 1.488 g/cc

Explosion Temp, °C 322, a low of 235° is reported by Henkin & McGill (Ref 17)

Friction Sensitivity, unaffected by steel shoe

Heat Test at 100°C, 0.69% loss 1st 48 hrs vs 0.17 for Tetryl, 0.62% loss 2nd 48 hrs vs 0.00 for Tetryl; no expln in 100 hrs

Hygroscopicity at 30°C & 90% RH, 0.03%

Impact Sensitivity, PA App, 2 kg wt 7-12 inches vs 9 for Tetryl; BM App, 31 cm vs 26 for Tetryl

International Heat Test at 75°, 0.4% loss in 48 hrs reported by British; no loss reported in tests conducted at Picatinny Arsenal
Power by Trauzl Pb Block, 146 vs 100 for Picric Acid
Rate of Detonation, 7730 meters/sec at d 1.60 g/cc vs 7400 for Tetryl; and 7580 m/sec at d 1.55 g/cc vs 7300 for Tetryl at same d
Rifle Bullet Impact Sensitivity, 2 explns in 5 trials vs no explns for Tetryl
Sand Test, 55.3 g sand crushed vs 54.7 for Tetryl
Sensitivity to Initiation, 0.26 g LA reqd vs 0.10 g for Tetryl; 0.23 g MF reqd vs 0.19 g for Tetryl
Surveillance Test at 65° C, 6 days for DINA vs 730+ for Tetryl
Vacuum Stability Test at 100° C, 11+ cc gas evolved in 16 hrs by DINA vs 0.5 cc in 40 hrs by Tetryl
Volatility, 0.01% at 21°; 0.02% at 40°

Thus, DINA is seen from the above data to be markedly less stable to heat than Tetryl. This lack of stability was indicated to be due to the presence of an impurity, Di(nitroxylethyl)-nitrosamine, which might be removed by heating
Uses. DINA has been proposed as a nonvolatile plasticizer for NC, and its use in place of NG has been suggested. Kincaid & McGill (Ref 21) obtd a patent for a flashless, nonhygroscopic proplnt contg NC, Nitroguanidine & DINA. It has been used in a proplnt called **Albanite** (See Vol 1 of this Encycl, p A119-R)
Refs: 1) Beil **4** {722} 2) S. Livingston & W.H. Rinkenbach, PATR **1510** (March 1945) 3) A.T. Blomquist, "DINA and Related Compounds", OSRD **5155** (June 1945) 4) M. Carmack, "DINA and Related Compounds", OSRD **5157** (June 1945) 5) R.L. Shriner, "DINA and Related Compounds", OSRD **5156** (Aug 1945) 6) L. Pauling, OSRD **5953** (Oct 1945) 7) F.W. Sunderman, OSRD **5547** (Jan 1946) 8) OSRD Summary Rept of Div 8, NDRC, Vol 1, Washington, DC (1946), pp 14-16 (Prepn), 39 (Props), 43 (Impact), 45 (Performance), 47 (Qc) 9) A.M. Soldate & R.M. Noyes, AnalChem **19**, 442 (1947) & CA **41**, 6105 (1947) (X-ray diffraction data) 10) W.J. Chute et al, CanJRes **26B**, 89 (1948) & CA **42**, 4918 (1948) (Prepn & props) 11) G.S. Meyers & G.F. Wright, CanJRes **26B**, 257 (1948) & CA **42**, 5843 (1948) (Prepn) 12) M. Kirsh et al, CanJRes **26B**, 435-40 (1948) & CA **42**, 6538 (1948) (Describe an increase in vel of deton for axially cavitated cylinders of DINA) 13) G.F. Wright & W.J. Chute, USP 2461582 (1949) & CA **43**, 4286 (1949) (Prepn) 14) R.N. Jones & G.D. Thorn, CanJRes **27B**, 830, 834 (1949) & CA **44**, 2848 (1950) (UV absorption spectrum of DINA called 1,5-Dinitroxy-3-nitro-3-azapentane) 15) G. Desseigne, MP **32**, 117-20 (1950) & CA **47**, 9013 (1953) (Prepn & props) 16) W.A. Schroeder et al, AnalChem **23**, 1740 (1951) & CA **46**, 5434 (1952) (UV & visible absorption spectra) 17) H. Henkin & R. McGill, IEC **44**, 1391-95 (1952) & CA **46**, 8857 (1952) 18) G.F. Wright & W.J. Chute, CanP 479929 (1952) & CA **50**, 8747 (1956) (Prepn & props of DINA) 19) A.T. Blomquist et al, USP 2669576 (1954) & CA **48**, 6700 (1954); USP 2678946 (1954) & CA **49**, 4704 (1955), pp 5-8 (Improved method of purifying & stabilizing DINA) 20) J. Cason Jr, USP 2686804 (1954) & CA **49**, 2075 (1955) (Prepn) 21) J.F. Kincaid & R.C. McGill, USP 2698228 (1954) & CA **49**, 5846 (1955) 22) L. Médard & M. Thomas, MP **36**, 97 (1954) & CA **50**, 3763 (1956) (Report $Q^v_{combstn}$ 577 kcal/mol at 18° and Q^v_{form} 70.5 kcal/mol at 18°) 23) P. Tavernier & M. Lamouroux, MP **37**, 201, 206 (1955) & CA **51**, 716-17 (1957) (Report heat capacity 2415 cal/g at const vol) 24) P. Tavernier, MP **38**, 309, 329 (1956) & CA **51**, 15952 (1957) (Reports Q^v_{form} 290 & Q^p_{form} 314 cal/g) 25) A.G. Merzhanov & F.I. Dubovitskiĭ, DoklAkadN **124**, No 2, 362-65 (1959) (Investigated DINA mixts with liq Tetryl and reported thermal expln) (Cited by Pvt E-2 M.A. Neiverth, FSTC Literature Search on "Soviet Research on Liquid Explosives in the Last Decade", 10 March 1964) 26) F.I. Dubovitskiĭ, IzvestAkadNauk, OtdelKhimNauk, No 6, 124-26 (June 1960); OTIA Translation 166618, MCL 846 846/182 (20 March 1961)

(Thermal expln of DINA under purely convective heat transfer conditions) (See also CA **56**, 11873 (1962) 27) Urbański **3** (1967), 36

Diethylolamine Trinitrate; Diethanolamine Trinitrate, Di(β-nitroxylethyl)-ammonium Nitrate; Bis(2-nitroxyethyl)-ammonium Nitrate; or Dinitroxyaminodiethane Nitrate [called in Fr Nitrate de Dinitroxy-diéthylammonium or Nitrate de Bis(nitroxyéthyl)-ammonium], $O_3NH.NH(CH_2.CH_2.ONO_2)_2$; mw 258.16, N 21.71%; wh crysts, mp 120.5-23.8°, d 0.80 (bulk) 1.595 (pressed at 2800 kg/sq cm; $Q_{combstn}$ 2260 cal/g; sol in acet, MeOH, hot alc & w; less sol in cold alc & cold w; was prepd by treating diethylolamine with either 98% HNO_3 or mixed acid (Refs 2, 3 & 4)

Its expl props were detd by Chute et al (Ref 3) and by Médard (Refs 5 & 6). It can be detonated by impact; power by Pb block test 131 (PA=100), a value close to that of DEGDN; and velocity of deton 7000 m/sec at d 1.55. The compd is relatively stable at RT, but decomp at higher temp (70°). It was patented for use alone or in expl mixts melting below 100° (Ref 2), but Chute et al (Ref 3, p 90) and Médard (Ref 5) do not recommend its use

Refs: 1) Beil - not found 2) P Naoum & H. Ulrich, GerP 500407 (1929) & CA **24**, 4397 (1930); BritP 350293 (1929) & CA **26**, 5423 (1932) 3) W.J. Chute et al, CanJRes **26B**, 89, 96 (1948) & CA **42**, 4918 (1948) 4) G. Desseigne, MP **35**, 117-20 (1953) & CA **47**, 9013 (1953) 5) L. Médard, MP **36**, 93 (1954) & CA **50**, 6795 (1956) 6) L. Médard & M. Thomas MP **37**, 101, 124 (1954) & CA **50**, 3763 (1956) 7) Urbański **2** (1965), 472-73

Di(2-nitroxyethyl)-chloramine; 2,2'-(Chloroimino)-diethanol Dinitrate (CA nomenclature) or **β-β'-Dinitroxydiethyl-chloramine** (called by Meyers & Wright) [called Bis(2-nitryloxyäthyl)-chloramin or Disalpetersäureester des Bis(2-hydroxy-äthyl)-chloramins in Ger], $ClN(CH_2.CH_2ONO_2)_2$; mw 229.58; N 18.30%; grn-yel liq, extremely unstable on standing even at 0°; should not be stored as it dec violently and bursts into flame; d 1.443 at 25°, n_D 1.4840 at 25°; can be prepd by adding aq Na hypochlorite & 12% HCl to a soln of Dinitroxydiethylammonium Nitrate in equal volumes of water & ether at 25°, or by nitrating dinitroxydiethylammonium chloride with HNO_3 & acetic anhydride at 10° (Refs 1 & 2). This compd can be nitrated to give an 80% of DINA (qv)

Refs: 1) Beil **4**, {721} 2) G.S. Meyers & G.F. Wright, CanJRes **26B**, 263 (1948) & CA **42**, 5843 (1948)

Di(ethylolamino)-benzene and Derivatives

1,3-Di(ethylolamino)-benzene; 1,3-Bis(β-ethanolamino)-benzene; or 1,3-Bis(β-hydroxyethylamino)-benzene, $C_6H_4(NH.CH_2.CH_2OH)_2$; may be considered as the parent compd of its nitrated derivs although not used to prep them:

Dinitro-1,3-di(ethylolamino)-benzene, $(O_2N)_2C_6H_2(NH.CH_2.CH_2OH)_2$; mw 286.24, N 19.58%:

2,4-Dinitro-1,3-di(ethylolamino)-benzene or 2,4-Dinitro-di(hydroxyethyl)-m-phenylenediamine, yel crysts (from ethyl acet), mp 140-41°; was prepd by reaction of an excess of hydroxyethylamine & 2,3,4-trinitrodimethylaniline at 100° (Ref 2)

4,6-Dinitro-1,3-di(ethylolamino)-benzene or 4,6-Dinitro-1,3-bis(β-hydroxyethylamino)-benzene; orn-yel ndls (from alc), having a bitter taste, mp 211°; practically insol in eth, petr eth, chlf, benz & toluene; sl sol in w, was prepd by treating an alcoholic soln of 1,3-dichloro-4,6-dinitrobenzene with 4 equivs of β-hydroxyethylamine (Ref 3). It gives, on nitration, an expl compd.

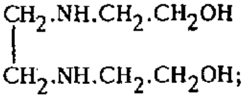
Refs: 1) Beil - not found 2) P. VanRomburgh & C.W. Zahn, Rec **57**, 441-42 (1938) & CA **32**, 5708-09 (1938) 3) K.F. Waldkötter, Rec **57**, 1305-07 (1938) & CA **33**, 1287 (1939)

2,4,6-Trinitro-1,3-di(N-nitro-β-ethanolamino)-benzene or 2,4,6-Trinitro-1,3-bis[N-Nitro-N-(β-hydroxyethyl)-amino]-benzene, $(O_2N)_3C_6H[N(NO_2).CH_2.CH_2OH]_2$; mw 421.27, N 23.28%; thick, sticky mass (no cryst product obtd), mp - softens at 30°, decomp violently at 98° & ignited at ca 230°; prepn attempted by nitrating the 4,6-dinitro deriv, but no cryst product could be isolated

It is an expl compd, but was not investigated further (Ref 2)
Refs: 1) Beil - not found 2) K.F. Waldkötter, Rec **57**, 1307 (1938) & CA **33**, 1287 (1939)

Di(ethylol)-ethylenediimine and Derivatives

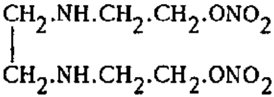
N,N'-Di(β-ethylol)-ethylenediimine; N,N'-Bis(2-hydroxyethyl)-ethylenediimine; or 2,2'-(Ethylenediimino)-diethanol (CA nomenclature) [called 1.2-Bis(2-hydroxy-äthyl-amino)-äthan or N.N'-Bis(2-hydroxy-äthyl)-äthylendiamin in Ger],



$$\begin{array}{l} CH_2.NH.CH_2.CH_2OH \\ | \\ CH_2.NH.CH_2.CH_2OH \end{array}$$

; mw 148.20, N 18.90%; crysts (from alc), mp 102-03°; was prepd by heating 2 mols 2-aminoethanol with 1 mol 1,2-dichloroethane at 130-50° (Refs 1 & 2) and by other methods. It is the parent compd of its nitrated derivs
Refs: 1) Beil **4**, {718} 2) H. Ulrich et al, FrP 801121 (1936); GerP 635904 (1936) & CA **31**, 111, 703 (1937) 3) J.R. Johnson, OSRD **915** (Oct 1942), p 2 4) Sax (1963), 726 (N,N'-Dihydroxyethylethylene Diamine)

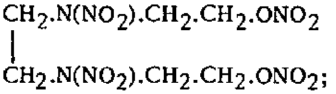
N,N'-Di(2-ethylol)-ethylene diimine Dinitrate; 2,2'-(Ethylenediimino)-diethanol Dinitrate (CA nomenclature) **or N,N'-Bis (2-nitroxyethyl)-ethylenediimine** [called N.N'-Bis-(2-nitryloxyäthyl)-äthylendiamin or Disalpetersäureester des 1.2-Bis-(2-hydroxy-äthylamino)-äthans in Ger],



$$\begin{array}{l} CH_2.NH.CH_2.CH_2.ONO_2 \\ | \\ CH_2.NH.CH_2.CH_2.ONO_2 \end{array}$$

; mw 238.20; N 23.52%; crysts, mp 170-71° dec; ignites instantaneously when dropped on a hot plate; was prepd by nitrating the parent compd with 98% HNO_3 as described in Refs 2, 3, 5, 6 & 7
Refs: 1) Beil **4**, {718} 2) J.R. Johnson, OSRD **915** (Oct 1942), 23-24 3) R.N. Jones & G.D. Thorn, CanJRes **27B**, 855 (1949) & CA **44**, 2848 (1950) (UV absorption spectrum of the Dinitrate) 5) A.T. Blomquist, OSRD **4134** (Nov 1944), 48, 73 6) A.T. Blomquist & F.T. Fiedorek, USP 2481283 (1949) & CA **44**, 4925-26 (1950) 7) J.R. Johnson et al, USP 2683165 (1954), CA **49**, 7590 (1955)

N,N'-Di(2-nitroxyethyl)-ethylenedinitramine; N,N'-Bis(β-nitroxyethyl)-ethylenedinitramine; 2,2'-[Ethylene-bis(nitroimino)]-diethanol Dinitrate (CA nomenclature); **and 1,8-Dinitroxy-3,6-diazaoctane Dinitrate** (Jones & Thorne) [called N.N'-Dinitro-N.N'-bis-(2-nitryloxy-äthyl)-äthylendiamin; or Disalpetersäureester des N.N'-Dinitro-N.N'-bis(2-hydroxy-äthyl)-athylendiamins in Ger],



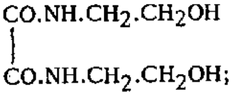
$$\begin{array}{l} CH_2.N(NO_2).CH_2.CH_2.ONO_2 \\ | \\ CH_2.N(NO_2).CH_2.CH_2.ONO_2 \end{array}$$

; mw 328.11, N 25.62%; wh crysts (from MeOH), mp 60-62° & 66.5-67.5°; was prepd from the tetranitrate deriv by heating it with acetic anhydride & a small amt of a chloride catalyst ($ZnCl_2$) (Refs 2, 3, 4 & 5), or by a two-stage process from ethylene oxide & ethylenedinitramine, $(CH_2.NHNO_2)_2$, in NaOH, and treating the product, $[CH_2N(NO_2)CH_2.CH_2OH]_2$, with HNO_3 at 10-15° (Ref 6)

This compd has expl props which are reported by Blomquist (Ref 3). It was patented as a nonvolatile plasticizer for NC used in proplnts
Refs: 1) Beil **4**, {722} 2) A.T. Blomquist, OSRD **4134** (Nov 1944), 48, 73, 125 3) A.T. Bolmquist, OSRD **5155** (June 1945), 160-72 4) R.N. Jones & G.D. Thorne, CanJRes **27B**, 834, 858 (1949) & CA **44**, 2848 (1950) (UV absorption spectrum) 5) A.T. Blomquist & F.T. Fiedorek, USP 2481288 (1949), pp 3-6 & CA **44**, 4925 (1950) 6) J.R. Johnson et al, USP 2683165 (1954) & CA **49**, 7590 (1955) 7) Sax (1963), 726 (Dihydroxyethyl Nitramine Dinitrate)

Diethylol-oxamide and Derivatives

N,N'-Diethylol-oxamide, Diethanol-oxamide, Dihydroxy-diethyl-oxamide, or Bis(hydroxyethyl)-oxamide [called N.N'-Bis(2-hydroxy-äthyl)-oxamid or Oxalsäure-bis(2-hydroxy-äthylamid) in Ger],



$$\begin{array}{l} CO.NH.CH_2.CH_2OH \\ | \\ CO.NH.CH_2.CH_2OH \end{array}$$

; mw 176.17, N 15.90%;

crysts (from alc), mp 167-69.2°; readily sol in w, glac acet ac & hot alc; less sol in chlf, benz & petr eth; insol in eth; was prepd by addg 1 equiv of dibutyl oxalate to a well stirred soln of 2 equivs of monoethylanolamine in dry butanol below 37°, and then heating the reaction mixt to 98° for 2 hrs (Refs 1 & 2)

Refs: 1) Beil 4, 285 & {708} 2) R.S. Stuart & G.F. Wright, CanJRes **26B**, 402, 408 (1948) & CA **42**, 6753 (1948)

N,N'-Di(nitroxyethyl)-oxamide; N,N'-Di(ethylol)-oximide Dinitrate; N,N'-Bis(2-nitroxyethyl)-oxamide; or N,N'-Bis(2-hydroxyethyl)-oxamide Dinitrate (CA nomenclature),

$CO.NH.CH_2.CH_2.ONO_2$
|
$CO.NH.CH_2.CH_2.ONO_2$; mw 266.17, N 21.05%; crysts (from HNO_3), mp 148.2°; was prepd by nitrating the parent compd with abs HNO_3 for 3 hrs at 0°, or by nitroxylating monoethanolamine, converting the nitroxyethylammonium nitrate to free base by cautiously addg KOH, and treating with diamyl oxalate (Refs 1, 2 & 3). This compd on nitration yields the powerful expl NENO

Refs: 1) Beil 4, {709} 2) R.S. Stuart & G.F. Wright, CanJRes **26B**, 409 (1948) & CA **42**, 6753 (1948) 3) G. Desseigne, MP **30**, 103-06 (1948) & CA **44**, 7242 (1950)

N,N'-Dinitro-N,N'-di(2-nitroxyethyl)-oxamide; N,N'-Dinitro-N,N'-di(2-ethylol)-oxamide Dinitrate; N,N'-Dinitro-N,N'-bis(2-hydroxyethyl)-oxamide Dinitrate (CA nomenclature); **or Bis-(nitroxyethylnitro)-oxamide,** abbreviated as **NENO** [called N.N'-Dinitro-N.N'-bis(2-nitryloxy-äthyl)-oxamid or Disalpetersäureester des N.N'-Dinitro-N.N'-bis(2-hydroxy-äthyl)-oxamids in Ger; and Dinitrate du Dioxyéthyldinitrooxamide in Fr],

$CO.N(NO_2).CH_2.CH_2.ONO_2$
|
$CO.N(NO_2).CH_2.CH_2.ONO_2$; mw 356.17, N 23.60%; exists in two polymorphic forms: *alpha*, ndls, stable at RT, obtd by slow crystn from chlf below 38°; and *beta*, massive monoclinic, obtd by crystn from chlf above 38° & filtering to prevent formation of alpha form; mp 90-92°, dec 105°, d 1.706 at 22° (α-form), d 1.686 at 22° & 1.562 at 92.6° (β-form); $Q^{p}_{combstn}$ 712 kcal/mol; sol in acet, hot alc, eth acet & cis-dichloroethylene; sl sol in benz, chlf & alc; insol in w (Refs 1, 17 & 18). This compd was first prepd by Von Herz (Ref 2) by condensing ethanolamine with oxalic acid, and nitrating the product. Stuart & Wright (Ref 17) prepd it by nitrating N,N-diethylol-oxamide with mixed acid at 40°, and described a batch process as well as a continuous method

Purified NENO is nonhygro & has a bulk density of 0.85-0.95 g/cc, and a cast d of 1.60-.64 g/cc entirely free of cavitation. It does not stain the hands or cause dermatitis as does Tetryl. No health hazards have been encountered in its use after 4 yrs (Ref 17)

NENO is an HE comparable in power & brisance to Tetryl and 1.3 times more powerful than TNT. It forms an eutectic with TNT (52% by wt of NENO), melting at 61° & d 1.678 at 22°, and with 1,3-DNB (62% by wt of NENO), melting at 61.8° (Ref 16)

The expl & other props of NENO recorded in various Refs below are as follows:

Explosion Temp, °C, 260-70° deflgr; 210° for 25 mg sample in 5 sec (Ref 20)

Heat of Combustion, 2024 cal/g at C_v (Refs 7, 10 & 14)

Heat of Explosion at 25°, 432 kcal/mol

Heat of Fusion, ca 20 cal/g

Hygroscopicity, v slight

Impact Sensitivity, less than that of PETN, approx the same as RDX & Tetryl, and much more sensitive than PA or TNT

Power by Ballistic Mortar 131-35% of TNT at d 1.65, and by Trauzl Pb block 143% of TNT

Thermal Stability, by Abel Test at 100° 25-30 mins (Ref 17)

Vacuum Stability Test at 100° 4 cc gas evolved in 6 hrs

Velocity of detonation, 7800-7860 at d 1.60-1.65 for an unconfined chge, and 5400 m/sec at d 1.0 g/cc

Uses. Although NENO can be melted & cast below 100°, Naoum does not consider it desirable to use it alone, due to formation of acidity, and recommends the addn of nitro compds, particularly nitramines. He patented (Ref 3) for example, a castable chge consisting of NENO 85, dinitrodimethyloxamide 10 & dimethyloxalate 5%, which freezes at 72-73° to a homogeneous, hard, nonhygro, powerful expl fairly insensitive to shock & friction. VonHerz (Ref 2) patented several expl mixts contg NENO. A filler for munitions, proposed by Stuart & Wright (Ref 17), consists of NENO 79, TNT 19.5 & beeswax 1.5%, is castable at 78° to a d 1.60-64 g/cc and 125% more powerful than TNT. Mixts of NENO & Al may be useful since a mixt of NENO 82 & Al 18% is 150% of TNT in power, of the same order of impact sens as NENO but more sensitive to friction

Other uses of NENO have included its substitution for Tetryl in boosters & detonators, and as a bursting chge for ammo when mixed with other ingredients

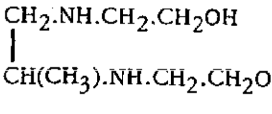
Refs: 1) Beil **4**, {722} 2) E. vonHerz, GerP 543174(1930) & CA **26**, 2598(1932) 3) P. Naoúm, GerP 56800(1931) & CA **27**, 2814(1933) 4) A. Foulon, SS **27**, 399 (1932); MAF **14**, 462(1935) & CA **27**, 844 (1932) (Prepn, props & uses of NENO) 5) M.S. Fishbein, VoyennayaKhimiya **1933**, No 6, 3-8; ChemZtr **1934** II, 1074-75 & CA **29**, 7077(1935) (Prepn & props of NENO) 6) Stettbacher (1933), 364 (Props) 7) A. Schmidt, SS **29**, 262(1934) (Props) 8) R. Adams & C.S. Marvel, OSRD **86** (Aug 1941), pp 13 & 42-45 (Prepn & props) 9) D.P. MacDougall, OSRD **560** (May 1942) (Phys & chem props) 10) G.B. Kistiakowsky, OSRD **702** (July 1942) (Heat of combustion) 11) R.C. Elderfield, OSRD **907** (Sept 1942) (Rates of hydrolysis) 12) A.T. Blomquist, OSRD **3014** (Aug 1944) (Microscopic examination) 13) ERL, Bruceton, Pa, OSRD **4758** (Feb 1945) (Adiabatic ignition time for NENO) 14) G. Stegeman, OSRD **5306** (July 1945) (Heat of combstn) 15) OSRD Summary Rept of Div 8, NDRC, Vol 1 (1946), pp 37, 43, 45 16) A.N. Campbell & H.A. Kushnarov, CanJRes **25B**, 216-27(1947) & CA **41**, 7218(1947) 17) R.S. Stuart & G.F. Wright, CanJRes **26B**, 401-14(1948) & CA **42**, 6752(1948) 18) G. Desseigne, MP **30**, 106-10(1948) & CA **44**, 7242(1950) (Prepn & props of NENO) 19) J. Vaughan, JCS **1950**, 748 & CA **44**, 6818(1950) (Hydrolysis of NENO) 20) H. Henkin & R. McGill, IEC **44**, 1391-95(1952) & CA **46**, 8857(1952) 21) Gilman **4** (1953), 979-80 22) A.R. Martin & H.J. Yallop, TrFaradSoc **54**, 257, 264(1958) & CA **52**, 17714-15(1958) (Calcn of deton vel as a function of structure, O balance & heat of formation) 23) Urbański **3**(1967), 37

Diethylol Peroxide, Diethanol Peroxide or Bis(1-hydroxyethyl)-peroxide [called *α.α'*-Dioxy-diäthylperoxyd or Bis-(1-hydroxyäthyl)-peroxyd in Ger], $CH_3.CH(OH).OO.CH(OH).CH_3$; mw 122.12, O 52.41%; col, easily flowing oil having a pungent odor, bp - explodes on heating in a tube; can be prepd by treating freshly distilled acetaldehyde with a 3% dry ethereal soln of H_2O_2 and by other methods (Ref 1). This compd is reported to be highly toxic (Ref 4)

Refs: 1) Beil **1**, [674] & {2653} 2) H. Wieland & A. Wingler, Ann **431**, 314, 317 (1923) & CA **17**, 2558(1923) 3) H. Wieland & H. Rau, Ann **436**, 259, 261(1924) & CA **18**, 2130(1924) 4) J. Mita, ArchExptlPath-Pharm **104**, 276-88(1924) & CA **19**, 1163-64 (1925) 5) J. Stamm, Pharmacia (Estonia) **18**, 71, 103(1938) (in German) & CA **33**, 7958(1939) (Detection as an impurity in ether) 6) A.C. Egerton, TrFaradSoc **44**, 745(1948) & CA **43**, 3724(1949) (UV absorption spectrum) 7) Tobolsky & Mesrobian (1954), 171

Diethylol-piperazine. See Bis(hydroxyethyl)-piperazine in Vol 2, p B146-R

Diethylol-propylenediamine and Derivatives
N,N'-Diethylol-propylenediamine; N,N'-Diethanol-propylenediamine; N,N'-Bis (2-hydroxyethyl)-propylenediamine or 2,2'-(Propylenediimino)-diethanol (CA nomenclature),

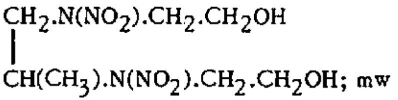


$$\begin{array}{l} CH_2.NH.CH_2.CH_2OH \\ | \\ CH(CH_3).NH.CH_2.CH_2OH \end{array}$$

; mw

162.23, N 17.27%; liq, bp 202-04° at 20 mm, d 1.0463 at 20°, n_D 1.4895 at 20°; prepn described by Tkaczyński (Ref 2). It may be considered as the parent compd of its dinitramines although not used to prep them
Refs: 1) Beil - not found 2) T. Tkaczyński, ActaPolonPharm **17**, 367 (1960) (in Polish) & CA **55**, 12417 (1961)

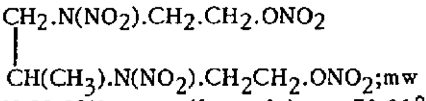
N,N'-Diethylol-propylenedinitramine or N,N'-Bis(2-hydroxyethyl)-propylenedinitramine,



$$\begin{array}{l} CH_2.N(NO_2).CH_2.CH_2OH \\ | \\ CH(CH_3).N(NO_2).CH_2.CH_2OH \end{array}$$

; mw 252.23, N 22.21%; oil - no props given; was obtd by passing ethylene oxide gas thru stirred propylenedinitramine in water & a small amt of NaOH (Ref 2)
Refs: 1) Beil - not found 2) A.T. Blomquist, "DINA and Related Compounds", OSRD **5155** (June 1945), pp 173-74 3) No later Refs were found thru 1966

N,N'-Bis(2-nitroxyethyl)-propylenedinitramine,

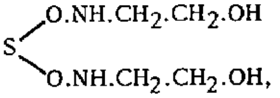


$$\begin{array}{l} CH_2.N(NO_2).CH_2.CH_2.ONO_2 \\ | \\ CH(CH_3).N(NO_2).CH_2CH_2.ONO_2 \end{array}$$

;mw 342.23, N 30.92%; cryst (from alc), mp 78-81°; obtd by nitrating the above compd with 98% HNO_3 at 5-10° (Ref 2). No expl props were detd
Refs: 1) Beil - not found 2) A.T. Blomquist, OSRD **5155** (June 1945), 174 3) No later Refs were found thru 1966

Diethylol-sulfamide and Derivatives

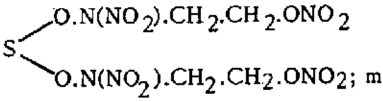
Diethylol-sulfamide, Diethanol-sulfamide, Bis(β-hydroxyethyl)-sulfamide,



$$S\begin{array}{l} \diagup O.NH.CH_2.CH_2.OH \\ \diagdown O.NH.CH_2.CH_2.OH \end{array}$$

, may be considered as the parent compd of its nitrated deriv, although not used to prep it:

N,N'-Dinitro-N,N'-di(2-nitroxyethyl)-sulfamide or N,N'-Bis(β-ethylolnitrate)-N,N'-dinitrosulfamide,



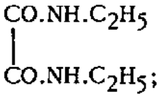
$$S\begin{array}{l} \diagup O.N(NO_2).CH_2.CH_2.ONO_2 \\ \diagdown O.N(NO_2).CH_2.CH_2.ONO_2 \end{array}$$

; mw 364.17, N 23.08%; crysts, expl; prepd by condensing sulfuryl chloride, (SO_2Cl_2), with ethanol amine, $HO.C_2H_4.NH$, and nitrating the product. This expl was claimed to be comparable with the corresponding oxamide deriv called NENO (See under Diethylol-oximide and Derivatives, above)
Refs: 1) Beil - not found 2) E. von Herz, GerP 543174 (1930) & CA **26**, 2598 (1932)

Diethyloxalate. See Ethyl Oxalate in Vol 6

Diethyloxamide and Derivatives

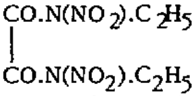
N,N'-Diethyloxamide [called Oxalsäure-bis-äthylamid or N.N'-Diäthyl-oxamid in Ger],



$$\begin{array}{l} CO.NH.C_2H_5 \\ | \\ CO.NH.C_2H_5 \end{array}$$

; mw 144.17, N 19.43%; ndls (from alc), mp 175-80°, d 1.1685 at 4°; can be prepd by treating a cold strong aq soln of ethylamine with ethyl oxalate (Ref 1). It forms on nitration an expl deriv
Ref: 1) Beil **4**, 112, (352), [605] & {213}

N,N'-Dinitro-N,N'-diethyloxamide,



$$\begin{array}{l} CO.N(NO_2).C_2H_5 \\ | \\ CO.N(NO_2).C_2H_5 \end{array}$$

; mw 234.17, N 23.93%; wh crysts (from alc), mp 34-35°, bp - explodes; readily sol in eth; diffc sol in alc; was prepd by nitrating the parent compd with mixed acid at a temp below 15° (Refs 1 & 2). It is a fairly powerful but very insensitive expl. Was recommended in Germany for use in blasting & military expls (Ref 2)

Other Dinitrodialkyloxamides have been prepd as indicated in Refs 1 & 2 and claimed to be powerful & insensitive expls
Refs: 1) Beil **4**, 130 2) WASAG, GerP 203190 (1907) & CA **3**, 717 (1909) 3) No later Refs were found in CA thru 1961
NOTE: No higher nitrated derivs of Diethyloxamide were found in Beil or in CA thru 1961

Diethyl Peroxide (called Diäthylperoxyd in Ger), $C_2H_5 - OO - C_2H_5$; mw 122.12, O 52.41%; col liq, fr p -70°, bp 62-64° at 740 mm press, d 0.8273 at 15° & 0.8240 at 19.4°, n_D 1.3715 at 16.5°; miscible with alc & eth; diffc sol in w; was prepd by Baeyer & Villiger (Ref 2) from diethyl sulfate, H_2O_2 & aq KOH soln (Ref 1)

Diethyl Peroxide easily inflames when touched with an object preheated to 250°; explodes in an atmosphere of CO_2 when touched with a hot Cu wire; forms expl mixts with air; could not be exploded by impact. Its thermal decompn was reported by Harris (Ref 4) and by Moriya (Ref 6). See also other Refs listed

Refs: 1) Beil **1**, 324, [325] & {1313} 2) A. Baeyer & V. Villiger, Ber **33**, 3388 (1900) 3) A. Rieche & F. Hitz, Ber **62**, 218(1929) 4) E.J. Harris, ProcRoySoc **175A**, 254(1940) & CA **34**, 4908)1940) 5) R.H. Wiley, USP 2357298(1944) & CA **39**, 312(1945) (Prepn) 6) K. Moriya, RevPhysChem, Japan, ShinkichiHoribaCommem Vol **1946**, 143-52 & CA **44**, 1312(1950) 7) A.C. Egerton et al, Discussions FaradaySoc **1951**, No 10, 278-82 & CA **46**, 3358(1952) (Props) 8) Tobolsky & Mesrobian (1954), 18, 164 & 178 9) D.H. Fine et al, "Experimental Measurements of Self-Heating in the Explosive Decomposition of Diethyl Peroxide", 12thSympCombstn (1968), 100-02 (Abstracts, Paper 54)

Diethyl Peroxydicarbonate,
$H_5C_2.OCO-OO-OCO.C_2H_5$; mw 178.14, O 53.89%; expl oil or solid possessing the sharp ozone-like odor of volatile acyl peroxides, mp 28-35°(dec), decompg spontaneously at RT, deflgr on contact with a flame; n_D 1.4017 at 20°; sol in a wide range of org solvs; sl sol in w; obtd in an impure form by Wieland et al (Ref 2) by reaction of ethyl chloroformate in chlf soln with powdered Na peroxide. Strain et al (Ref 3) prepd the compd by the same procedure but pointed out the special care that was reqd in temp control (0-10°) in all operations of prepn & purification because of its high degree of thermal instability. When subjected to heat, friction or shock, Diethyl Peroxydicarbonate appears to be more sensitive than Benzoyl Peroxyde (qv)

Refs: 1) Beil - not found 2) H. Wieland et al, Ann **446**, 46(1925) 3) F. Strain et al, JACS **72**, 1254(1950) 4) Tobolsky & Mesrobian (1954), 170

Diethylphenylenediamine and Derivatives

Diethylphenylenediamine, $C_{10}H_{16}N_2$; mw 164.24, N 17.06%. Four isomers are found in Beil:

N,N-Diethyl-o-phenylenediamine or 2-Amino-diethylaniline, $H_2N.C_6H_4.N(C_2H_5)_2$; strong smelling oil, bp 312.5° at 744 mm press (Ref 2)

N,N-Diethyl-m-phenylenediamine, liq, bp 276-78° (Ref 3)

N,N-Diethyl-p-phenylenediamine, liq rapidly turning brown on exposure to light, bp 260-62° (Ref 4)

N,N -Diethyl-1,3-phenylenediamine, $H_5C_2.HN.C_6H_4.NH.C_2H_5$; no description given (Ref 1)

Other props & methods of prepn are found in the Refs

Refs: 1) Beil **13**, 41 2) Beil **13**, (6) 3) Beil **13**, 41, (12) & [26] 4) Beil **13**, 75, (22) & [40]

Nitroderivatives of Diethylphenylenediamine

4-Nitro-N',N'-diethyl-1,3-phenylenediamine, $H_2N.C_6H_3(NO_2).N(C_2H_5)_2$; mw 209.24, N 20.08%; lt-yel crysts, mp 139°; was prepd by heating 3,4-dinitrodiethylaniline with alcoholic NH_3 in a tube at 120° (Ref)

Ref: Beil **13**, [30]

Dinitrodiethylphenylenediamine, $C_{10}H_{14}N_4O_4$, not found in Beil or in CA

2,4,6-Trinitro-N,N'-diethyl-1,3-phenylenediamine, $(O_2N)_3C_6H(NH.C_2H_5)_2$; mw 299.24, N 23.41%; yel crysts; mp 142-44°; was prepd by heating N-nitro-N-ethyl-2,3,4,6-tetranitroaniline with ethylamine in a tube at 50-60°, or from 3-chloro-2,4,6-trinitrophenol & ethylamine in alc. Its expl props were not reported

Refs: 1) Beil **13**, 61 & (17) 2) J.J. Blanksma, Rec **21**, 325(1902)

Tetranitrodiethylphenylenediamine, $C_{10}H_{12}N_6O_8$, not found in Beil or in CA

N,N',2,4,6-Pentanitro-N,N'-diethyl-1,3-phenylenediamine, $(O_2N)_3C_6H[N(NO_2).C_2H_5]_2$; mw 389.24, N 25.19%; col crysts, mp 165°; was obtd by nitrating the above trinitro deriv (Refs 1 & 2). Its expl props were not reported

Refs: 1) Beil **13**, 61 2) J.J. Blanksma, Rec **21**, 326(1902) & JCS **84 I**, 158-59(1903)

N,N'-bis(2,2,2-Trinitroethyl)-o-phenylenediamine; bis(Trinitroethylamine-o-phenylene or o-bis (2,2,2-Trinitroethylamino) benzene, $C_6H_4[NH.CH_2.C(NO_2)_3]_2$; mw 434.24, N 25.81%; expl compd, mp 70°; was prepd by condensing o-phenylenediamine & 2,2,2-trinitroethanol, $(O_2N)_3C.CH_2.OH$, both in methanol, chilling the mixt and pptg the product by dropwise addn of H_2O (Ref 2). The expl has an O balance of -48 and an impact sensitivity value (2 kg wt) of 65 cm

Refs: 1) Beil - not found 2) R.H. Saunders, USP 2996547 (Aug 1961) & CA **55**, 26447 (1961)

o-Diethylphthalate (DEP or DEtPh) or Ethyl Phthalate (called Diäthylphthalat or Phthalsäure-diäthylester in Ger), $C_6H_4(COOC_2H_5)_2$; mw 222.23, O 28.80%; clear, colorless liq, fr p -40.5°, bp 295-98°, d 1.1268 at 15°, d 1.0872 at 55.7°, n_D 1.5049 at 16°, viscosity 0.1006 g/cm sec at 25°; flash p 243° F (CC); $Q^v_{combstn}$ 6404 cal/g or 1423 kcal/mol; Q^v_{form} 829 cal/g or 184 kcal/mol (Refs 1 & 4). Diethylphthalate can be prepd by the action of alcohol on phthalic anhydride in the presence of anhyd HCl, $CaCl_2$ or $CuSO_4$

It is miscible with alc, ether, ketones, esters & aromatic hydrocarbons; partly miscible with aliphatic solvs; and insol in w. Diethylphthalate gelatinizes NC and is used as a gelatinizing & flash-reducing ingredient in proplnts

The requirements of the US Armed Forces, as covered by specification (Ref 2), include the following:

Color - shall not be darker than a soln contg 0.5 ml of 0.1N iodine in 100 ml of distilled water

Specific Gravity - 1.12±0.01 at 15°

Ester Content as diethylphthalate - 99.0% min

Acidity as phthalic acid - 0.03% min

Ash - 0.01% max

Its detn in proplnts and some other props of Diethylphthalate are found in the following Refs and under Analytical Procedures

Refs: 1) Beil **9**, 798, (351) & [584] 1a) US Specification JAN-D-242 (July 1945) (DEPh for use in expls) 2) P.G. Butts et al, AnalChem **20**, 1066-67 (1948) & CA **43**, 1568 (1949) (Detn of DEPh in proplnts) 3) S. Wachtell, "Investigation of Chromatographic and Spectrophotometric Methods of Powder Analysis. Development of Chromatographic-Spectrophotometric Method for Determining Diethylphthalate in Type I, Casting Powder", PATR **1731** (May 1949) 4) L. Médard & M. Thomas, MP **34**, 426-28 & 434-37 (1952) 5) F. Pristera & A. Gonzales - Villafane, "Simple, Rapid Infrared Method for the Determination of Diethylphthalate in N4 Propellant", PATR **1886** (July 1952) 6) C. Ribaudo, "A Polarographic Method for the Determination of Diethylphthalate in M-8 Type Propellant Powder", PATR **1918**, (Jan 1953) 7) L. Marvillet, MP **40**, 273ff (1958) & CA **54**, 25825 (1960) (Chromatographic analysis of powders contg DEPh) 8) G. Norwitz, "Determination of Diethyl and Dibutyl Phthalates in Propellants", AnalChemActa **19**, 216-23 (1958) 9) L. Marvillet, MP **42**, 248, 255, 257 (1960) & CA **55**, 14917 (1961) (Quantitative analysis by chromatographic methods of phthalic esters in proplnts) 10) ProplntManual SP1A/M2 (1962) (Unit Nos 46, 260, 385 & 389) (Conf) 11) Sax (1963), 718 (See also Refs under Dibutylphthalate)

Diethylphthalate, Analytical Procedures. A general method, using gravimetric or titrimetric procedures, suitable for detn of dimethyl-, diethyl-, dibutyl-, or dioctyl-phthalate content in proplnts contg a phthalate ester and NG, NC, DNT, DPhA, 2-Nitro-DPhA, Ethyl Centralite & Triacetin, is described by Pierson in StdMethodsChemAnalysis (Ref 4, p 1396). The procedures are based on methods previously reported by Stalcup et al (Ref 2)

A. *Gravimetric Procedure.* a) Place an accurately weighed 2 g sample of proplnt in a small paper thimble, cover with glass wool & extract with methylene chloride for 3 hrs in a Soxhlet app with a 250-mm refluxing flask. Remove the methylene chloride by air jet evapn and add 25 ml of 65-75% acetic acid. Heat the soln in reflux flask to 70°, add a few drops of satd $CuSO_4$, stopper the flask and swirl occasionally while addg 5 g of Zn dust in small portions during a 15 min period

b) Filter the hot soln thru a Büchner funnel into a 500-ml separatory funnel contg 250-ml of water. Rinse the filter & flask with two 10-ml portions of hot acetic acid and 75 ml of methylene chloride. After shaking the funnel and allowing the mixt to separate, transfer the lower layer to a 500-ml Squibb-type separatory funnel. Add 5 ml addnl methylene chloride to the first funnel and, without shaking, transfer the lower layer to the second funnel. Extract the acetic acid in first funnel by shaking twice with 25-ml portions of methylene chloride, add these extracts to second funnel, and discard the acid in first funnel

c) Now add 50 ml of 10% K_2CO_3 soln to the second separatory funnel, shake, test with indicator paper to assure an excess of K_2CO_3, and transfer the methylene chloride to a 250-ml iodine flask. Wash the K_2CO_3 soln several times with 10-ml portions of methylene chloride

d) Evaporate the methylene chloride in the iodine flask until no odor is detectable, add 25 ml of 0.5N KOH soln in anhyd alc, and heat on a water bath for 1 hr

e) Cool the flask to RT, add 25 ml of dry ether, and filter the contents thru a fritted-glass crucible previously weighed. Wash the crucible with 1:1 eth-alc, and test with a drop of phenolphthalein indicator for complete removal of KOH

f) Heat the crucible for 1 hr at 210° C in an oven, converting the dipotassium phthalate alcoholate to dipotassium phthalate. Cool the crucible in a desiccator for at least one hr and weigh

g) Correct for the small amt of K_2CO_3, which may be present as an impurity, by dissolving the crucible contents in warm neutral water, and titrating with 0.05N HCl to the phenolphthalein end point. Milliliters of std acid times its normality times 0.1382 = grams K_2CO_3

h) Calculate the phthalate ester content using the following formula:

$$\%\ \text{Phthalate Ester} = \frac{F \times P \times 100}{W}$$

where: F = factor for converting dipotassium phthalate to the phthalate ester of the proplnt: Dimethyl=0.85, Diethyl=0.917, Dibutyl=1.148 & Dioctylphthalate=1.610

P = wt of dipotassium phthalate after correcting for carbonate content in grams and

W = wt of sample in grams

B. *Titrimetric Procedure.* a) Follow above procedure thru step e

b) Wash the solvent-wet ppt into a 250-ml Erlenmeyer flask with 30 ml of glacial acetic acid

c) Add 5 drops of crystal violet indicator (0.1% in glac acet ac) and titrate the soln with std 0.1N perchloric acid in glacial acetic acid

d) The end point color change is from violet to blue.

e) If sample titration is at a temp diff from that of the standardization of the perchloric acid soln, use the factor 0.0011 ml/ml/° C for buret-reading temp correction

f) Calculate the phthalate ester content using the following formula:

$$\%\ \text{Phthalate Ester} = \frac{V \times N \times M}{20W}$$

where: V = std perchloric acid soln

N = normality of std perchloric acid soln

M = molecular wt of the phthalate ester

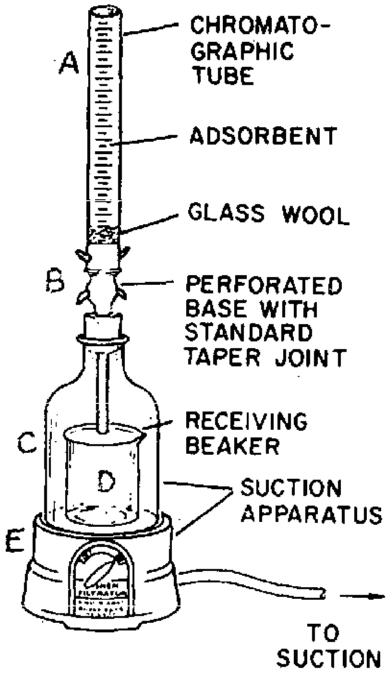
W = wt of sample taken for analysis in grams

C. *Chromatographic-Spectrophotometric Procedure* (Ref 3, Method 203.2.2).

It is used for detg relatively small percentages of DEtPh (such as ca 3%) that occur in mortar proplnts. It contains a procedure for removing Ethyl Centralite, which interferes with the spectrophotometric detn of DEtPh

For prepn of proplnt sample, use Method 509.3 of Ref, which is also described in Vol 2 of Encycl, p C131-L, under CENTRALITES *Procedure:* a) Weigh a 5 g sample to within 0.2 mg and transfer it to the flask of extraction apparatus (Soxhlet, Roweg or equivalent) to be extracted by the Method 104.1.3 of Ref, which is also described in Vol 2 of Encycl, p C131-L

b) While extraction proceeds, assemble the chromatographic apparatus similar to that shown in Fig on p C290-R of Vol 3. The apparatus is also shown here for convenience. The adsorbent tube (A) is 230 mm long and 28 mm ID



CHROMATOGRAPHIC APPARATUS

c) After assembling the apparatus, cover the bottom of the tube (A) with a thin mat of glass wool and apply sufficient suction to achieve, inside suction apparatus (CE), a pressure of ca 1/3 atm

d) Using a spatula, slowly add the " adsorbent" into tube (A) to a height of ca 5 inches, without tamping. The adsorbent is a 2:1 mixt of silicic acid (Malinckrodt's or equivalent) and Celite (Johns-Manville's Hydro-Supercel, or equivalent)

e) Using a flat-ended glass rod, 27 mm diam, level off the adsorbent in (A) to a height of ca 4.5 inches

f) Determine the volume (V) of ethyl ether (anhydrous, Spec JAN-E-199, Grade B) that will just wet the column of adsorbent. Dry the column by suction

g) Prepare 1.5V of a trial " developer", consisting of 2% soln (by vol) of ethyl ether in methylene chloride (Spec MIL-D-6998)

h) Pour thru the column in (A) the solns listed below in such a manner that a continuous flow of liquids thru the column is assured. For this add each new soln just before the one previously added is completely drawn into the adsorbent. In other words, the top of the column must always be covered with liquid until all the ingredients have been added

The liquids to be poured in succession are: 1 V of ethyl ether, 1 V of petroleum ether (bp 35-45°), "standard test solution" consisting of 2.0 mg EtCentr in 15 ml of the 1:2 - petr ether-methylene chloride, 3V of 1:2 petr ether-methylene chloride and 1.5V of the above "developer"

i) Test the adsorbent column for EtCentr as follows:

j) Disconnect the tube (A) from (B) and, using the above flat-ended rod, extrude the adsorbent column onto a piece of white paper

k) Place a contiguous series of drops of 1% ceric sulfate in 85% sulfuric acid along the column starting from the bottom, and observe the point at which a strawberry-red coloration begins. Disregard a light pink coloration. This indicates the lower edge of the EtCentr band. It should lie betw 1 & 3 cm from the bottom of column and if it does, consider both the adsorbent and the developer as satisfactory

l) If the lower end of red band lies below 1 cm or above 3 cm from the bottom of column, increase or decrease the concn of ether in developer within the range 1 to 5% and use another freshly prepared column

m) Remove the flask with extraction of propellant from Soxhlet, etc [See procedure (a)], add 10 ml of petr ether and 20 ml methylene chloride and transfer the resulting soln to a 250-ml volumetric flask. Rinse the extraction flask with 1:2 - petr eth-methylene chloride, and pour the rinsings into the flask. Fill to the mark with above mixture and pipet from it exactly 15 ml into a small beaker. This will be the soln to test

n) Prepare a fresh column of adsorbent material as described in procedures (c), (d) & (e) and add in the manner described in procedure (h), 1 V ethyl ether, 1 V petr ether, and 15 ml of test soln from small beaker (See procedure (m). Rinse the small beaker with portion of 1:2 - petr eth-methylene chloride soln (3 V), which will go next, pour the rinsings on the column and then add the remaining portion of above soln and, before its complete disappearance from the top of the column, add 20 ml of the developer

o) As soon as 15 ml of developer will enter the column, rapidly disconnect the suction

p) Remove the original 400-ml beaker (D) and replace it with a clean 400-ml beaker; reconnect the suction and pour the rest of developer thru (A), so that in all 1.5 V will have been used. Discard the liquid in original 400-ml beaker

q) When no more liquid will be dripping into new 400-ml beaker, disconnect the suction

r) Extrude the column, as described in procedure (j) and test it with 1% ceric sulfate soln in 85% sulfuric acid as described in procedure (k)

s) If the lower line of EtCentr band lies within 1-3 cm from the bottom of the column, test for channeling by cutting a slice ca 0.5 cm thick from the bottom of column and touching its center with a drop of ceric sulfate soln. The presence of red color indicates that channeling occurred and the new column should be used. The same should be done if the lower part of red band is not betw 1 & 3 cm from the bottom of column

t) If the tests indicate that the adsorbent column has removed all the EtCentr from the soln of proplnt, evaporate the solvent in 400-ml beaker by means of dry air current and pour enough 95% ethyl alcohol (Spec MIL-E-463, Grade 1) into the beaker to dissolve the residue

u) Transfer the soln into a 100-ml volumetric flask. Rinse the beaker with alcohol and transfer the rinsings into the flask. Fill the flask to 100-ml mark with alc and save it

v) Fill one silica cell, 1.0 cm wide (light path length) with the above soln and another matched cell with 95% ethyl alcohol to serve as a reference

w) Using the Quartz Ultraviolet Spectrophotometer (Beckman Instruments Inc, Model DU, or equivalent), determine the adsorbence of the soln at a wave length of 275 millimicrons

x) Calculate the percentage of DEtPh in the propellant using the following formula:

$$\% \text{ DEtPh} = (29950A) / CW$$

where: A = Corrected adsorbance of proplnt soln
C = Width of cell in cm, and
W = Weight of original sample in mg corrected for total volatiles

D. *Zinc Reduction-Volumetric Procedure* (Ref 3, Method 203.5). It is used for detg DEtPh or DBuPh content of proplnts that do not contain interfering esters such as other phthalates, dimethyl sebacate, triacetin, or sucrose octaacetate

The method is the same as described under "Dibutylphthalate, Analytical Procedures", except that the formula for calcn is different, namely:

$$\% \text{ DEtPh} = [11.1(A-B)N] / W$$

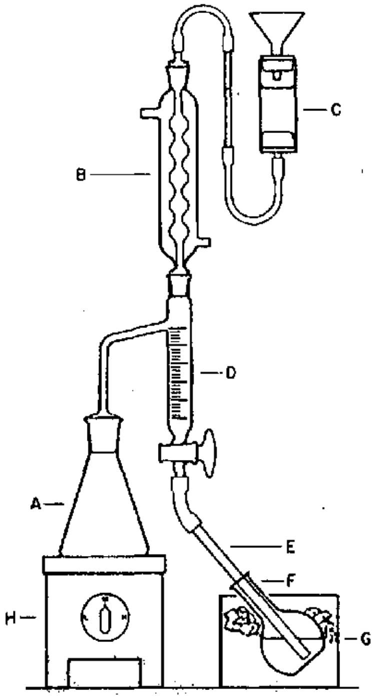
where: A = Ml of standard hydrochloric acid soln for blank titration
B = Ml of hydrochloric acid for titration of sample
N = Normality of standard hydrochloric acid soln
W = Weight of sample in grams, corrected for total volatiles

E. *Azeotropic Distillation Procedure* (Ref 3, Method 203.4.1)

It is used for detg the DEtPh content by saponification with KOH soln; the alcohol liberated in the reaction is separated by azeotropic distillation and measured by means of iodometric analysis after dichromate oxidation

The sample should consist of a portion of proplnt contg ca 0.3 g of DEtPh (as detd from the detailed specification of proplnt being tested)

Procedure: a) Weigh the sample (previously prepd as described in Method 509.3, which is also given in Vol 2 of Encycl, p C131-L, under CENTRALITES) to within 0.2 mg and transfer it to Erlenmeyer flask (A) of apparatus (See Fig). Add small pieces of porcelain, glass or carborundum (to prevent bumping during boiling) and 50 to 100 ml of 30% KOH soln. Such a large amt is required because NC & NG are also saponified. Swirl the flask in order to thoroughly wet all grains of proplnt and to prevent them from clumping and adhering to the walls of the flask



Azeotropic distillation apparatus

A	Erlenmeyer flask, 500-ml
B	Condenser, Allihn type, 400-ml
C	Water trap
D	Distillation receiving tube
E	Extension tube
F	Volumetric flask, 250-ml
G	Ice water bath
H	Hotplate (3-heat)

b) Cover the lower outer joint of the condenser (B) with a thin coat of high mp stopcock grease and attach it directly to (A), leaving out (D, E, F & G) of Fig. Grease also other glass joints

c) Attach the water trap (C) to the condenser, and pour 10 to 15 ml of distd w into it

d) Place the hot plate (H) under (A) with the control set at LOW. As soon as refluxing begins, turn to MEDIUM, and reflux for 30 mins. Remove the hot plate and allow the soln to cool for 10 mins without disconnecting condenser

e) Detach the water trap (C) and pour 25 ml of 3% hydrogen peroxide soln into (B). Reattach the trap and raise (C) high enough to cause its w to flow thru (B), flushing the peroxide down into (A)

f) Refill (C) with a fresh 10-15 ml of distd w, place the hot plate, set at LOW, under (A) and reflux for 40 mins. Remove the plate and without disconnecting (B), cool (A) by placing it in cold w bath

g) When the contents of (A) reach RT, pour 50 ml distd w thru the trap (C) and condenser (B) into flask (A)

h) Detach (B) from (A) and rearrange the apparatus by incorporating (D, E, F & G), as shown in Fig

i) Detach the trap and, with the stopcock of (D) closed, pour thru (B) 50 ml of benzene (ACS grade, thiophene-free) into (D) and (A)

j) Add thru (B) just enough distd w into (D) to displace all benzene and cause it to flow to (A)

k) Open the stopcock of (D), and drain all but 2.0 ml of w into volumetric flask (F); then close the stopcock and replace the water trap (C), contg 10-15 ml distd w

l) With the current turned off, place the hot plate under flask (A) and then turn control to LOW. Distill at the beginning very slowly to avoid bumping or foaming and then the control can be turned to MEDIUM and distn continued until the water-alcohol layer that forms at the bottom of receiving tube (D) reaches ca 20 ml. Open stopcock and drain off 10-15 ml of bottom layer leaving at least 5 ml in tube (D). No benzene must enter the flask (F)

m) Close the stopcock and continue distn. As it proceeds, draw off periodically 10-15 ml of aliquots, until a total of 85 ml has been collected in (F)

n) Remove the hot plate and disconnect the trap (C). Carefully drain the last of the water layer from (D) to (F), closing the stopcock when the bottom of benzene layer in (D) reaches the 5-ml mark

o) Wash the benzene layer in (D) by pouring 5-ml portions of distd w thru (B) and then drawing each rinsing thru stopcock into (F). Disconnect the extension tube (E), and pour a small amt of distd w thru it into (F)

p) Transfer the w from trap (C) into (F) and rinse the trap twice with small portions of distd w, adding the rinsings to contents of (F)

q) Remove from ice bath the flask (F), warm it to ca 20° and dilute contents to the 250-ml mark with distd w. Then determine the percentage of alcohol in the soln by iodometric analysis as described below

r) Pipet a 50-ml portion from (F) to a 250-ml iodine titration flask and add 25 ml of 0.2N standard soln prepd from K dichromate (BurStdsSample No 136) in 40-percent (by wt) H_2SO_4

s) Cover the flask loosely with a moistened glass stopper and heat it on a steam bath for 75 mins, to allow the dichromate to completely oxidize the alcohol

t) Remove the flask from the bath and cool it to RT

u) Add 15 ml of 15% KI (free from iodate) soln and titrate liberated iodine with 0.1N Na thiosulfate soln, using a starch indicator

v) Conduct a blank detn using a synthetic proplnt mixt of all ingredients (except DEtPh) that approximates the proplnt compn described in the detailed Spec

w) Calculate the percentage of DEtPh in the proplnt on a volatile-free basis as follows:

$$\% \text{ DEtPh} = [13.89(A-B)N] / W-C,$$

where: A = Ml of Na thiosulfate soln required to titrate iodine liberated from KI by 25 ml of K dichromate soln

B = Ml of Na thiosulfate soln required to titrate iodine liberated from KI by K dichromate remaining after oxidation of alcohol in the 50-ml aliquot of the distillate

C = Ml of Na thiosulfate soln required for ingredient blank

$$\text{Ml} = [13.89(V-v)N] / W'$$

N = Normality of Na thiosulfate soln

W = Weight of sample corrected for total volatiles, grams

W' = Weight of synthetic proplnt used for blank, grams

V = Ml of Na thiosulfate soln required for the blank run concurrently with the synthetic proplnt mixture

v = Ml of Na thiosulfate soln required for the synthetic proplnt sample

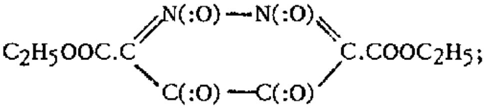
This procedure, also described by Pierson (Ref 4, p 1382), is a modification of that given by Butts et al (Ref 1)

Refs for DEPh Analytical Procedures: 1) P.G. Butts, G.B. Prine, D.L. Kouba & W.W. Becker, AnalChem **20**, 1066 (1948) 2) H. Stalcup, F. McCollum & C.L. Whitman, AnalChem **29**, 1066, 1479 (1957) 3) US Military Standard **MIL-STD-286A** (Aug 1961) 4) R.H. Pierson, "Explosives and Propellants", Chapter 32 in StdMethodsChemAnalysis **2B** (1963), 1382-83 & 1396-98 5) See also Refs under Dibutylphthalate

N,N'-Diethyl-1,4-piperazine-dicarboxamide. See Di(ethylcarboxamide)-piperazine in this Vol

1,1-Diethyl-2-propynylhydroperoxide or 3-Methyl-3-hydroperoxy-1-pentyne. See under Acetylene Hydroperoxides and Peroxides

3,6-Diethyl-1,2,4,5-tetraketo-4,5-dihydro-pyridazine-3,6-dicarboxylate or 4,5-Dihydro-4,5-dioxo-3,6-pyridazine-dicarboxylic acid-1,2-dioxide diethylester,

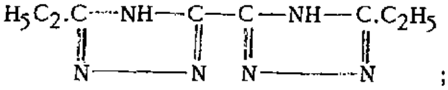


mw 286.20, N 9.79%; crysts, mp 70°, dec on long standing at RT, explodes when heated with Pt black; readily sol in acet & eth acet; diffc sol in alc, eth, acet acid & benz; was obtd by reacting diethyl ketipinate in eth with N_2O_4 at -15° (Ref 2)

Refs: 1) Beil - not found 2) E.J. Virgin, Diss, UnivUppsala, **1914**, 63 pp & CA **14**, 1321 (1920)

Di(ethyltriazeno)-benzene. See Bis(ethyltriazeno)-benzene in Vol 2, p B142-R

Di(ethyl-s-triazole) or 5,5'-Diethyl-3,3'-bis-s-triazole,



$$\begin{array}{l}H_5C_2.C{-}NH{-}C{-}C{-}NH{-}C.C_2H_5\\ \quad \| \qquad\qquad\quad \| \quad \| \qquad\qquad\quad \|\\ \quad N{-}{-}{-}{-}N \quad N{-}{-}{-}{-}N\end{array}$$;

mw 192.22, N 43.72%; crysts (from alc); sol in acids; sl sol in hot w & NH_3; was prepd by heating dihydrazone oxamide or oxalhydraazidine, ($C_2H_8N_6.H_2O$), with propanoic anhydride, $(C_2H_5CO)_2O$, to yield the intermediate propionyl diethylbitriazole (wh crysts, mp 192°) which on heating in HCl and addg NH_3 yields the product. This compd with $AgNO_3$ or $HgCl_2$ in alc forms wh insol salts, the Ag salt of which is sensitive to light (Ref 2)

No expl props of the compd or its salts were detd

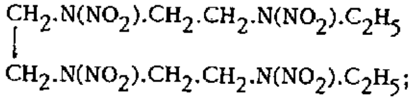
Refs: 1) Beil - not found 2) G. Dedichen, AnhandlNorskeVidenskaps-AkadOslo, I, Mat-NaturvKlasse **1936**, No 5, 42 pp & CA **31**, 4987 (1937)

Diethyltriazolyl-formamidine. See N,N'-Bis(5-ethyl-s-triazol-3-yl)-formamidine in Vol 2, p B142-R

Diethyltribromobenzene-azohydrazine-dicarboxylate [called 1-(2.4.6-Tribrom-phenyl)-tetrazen-(1)-dicarbonsäure-(3.4)-diäthylester or (2.4.6-Tribrom-benzoldiazo)-hydrazin-N.N'-dicarbonsäurediäthylester in Ger], $Br_3C_6H_2.N{:}N.N(COO.C_2H_5)NH.COO.C_2H_5$; mw 517.00, N 10.84%; ndls (from eth acetate + petr eth), mp explodes 111-15°; turns brn on exposure to light; sol in alc, eth & benz; insol in water & petr eth; decomp in aq NaOH; was obtd by reaction of 2,4,6-tribromobenzenediazonium salt & diethyl hydrazinedicarboxylate in the presence of aq Na acetate & Na carbonate (Refs 1 & 2)

Refs: 1) Beil **16**, (417) 2) O. Dimroth & G. deMontmollin, Ber **43**, 2910 (1910) & CA **5**, 494 (1911)

N',N⁴-Diethytriethylene Tetranitramine or 1,10-Diethyl-1,4,7,10-tetranitrotriethylenetetramine,



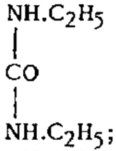
$$\begin{array}{l}CH_2.N(NO_2).CH_2.CH_2.N(NO_2).C_2H_5\\ |\\ CH_2.N(NO_2).CH_2.CH_2.N(NO_2).C_2H_5\end{array}$$;

mw 382.34, N 29.51%; crysts (from alc), mp 96.5-96.8°; sol in alc; insol in alkalies; gives a positive Franchimont test for nitramine; was prepd from a suspension of triethylenetetranitramine in eth soln contg diazoethane, which reaction evolved gas rapidly, and on evaporation of the ether left a sirup which gradually solidified during four days (Ref 2). This is an expl compd

Refs: 1) Beil - not found 2) G.N.R. Smart & G.F. Wright, JACS **70**, 3142 (1948) & CA **43**, 570 (1949)

Diethylurea and Derivatives

1,3-Diethylurea or N,N'-Bis-ethylurea (called N.N'-Diäthyl-harnstoff or Kohlensäure-bis-äthylamid in Ger),



$$\begin{array}{l}NH.C_2H_5\\ |\\ CO\\ |\\ NH.C_2H_5\end{array}$$;

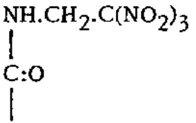
mw 116.16, N 24.12%; anisotropic lfts (from petr eth), ndls (from alc), mp 112°, bp 263°, d 1.0415; readily sol in w, alc & eth; can be prepd from ethylamine & CO_2 at 180-200°, and by many other methods (Ref). It may be considered as the parent compd of its hexanitro deriv

Ref: Beil **4**, 115, (353), [608] & {218}

Azido, $C_5H_{11}N_5O$, and *Diazido*, $C_5H_{10}N_8O$, derivs were not found in Beil or in CA thru 1961

Mononitro, $C_5H_{11}N_3O_3$, *Dinitro*, $C_5H_{10}N_4O_5$, *Trinitro*, $C_5H_9N_5O_7$, *Tetranitro*, $C_5H_8N_6O_9$, and *Pentanitro*, $C_5H_7N_7O_{11}$, derivs were not found in Beil or in CA thru 1961

1,3-Di(2,2,2-trinitroethyl) urea; Hexanitrodiethyl urea; or N,N'-Bis(β,β,β-trinitroethyl) urea, Code name **BTNEU,**



$$\begin{array}{l}NH.CH_2.C(NO_2)_3\\ |\\ C{:}O\\ |\\ NH.CH_2.C(NO_2)_3\end{array}$$;

mw 386.16, N 29.09%; crysts, mp 187-91° (dec); d 0.5-0.6 g/cc,

$Q_{combstn}$ 1645 cal/g; Q_{form} 70 kcal/mol (Ref 5); was first reported in the open literature by Schenck & Wetterholm (Ref 2) who prepd the compd by stirring 2 mols of trinitroethanol with 1 mol of urea in aq soln for 2 hrs at 80°. This compd was also prepd by addg dimethylolurea in water to trinitromethane in aq soln and stirring the mixt for 5 mins at 40° (Ref 3)

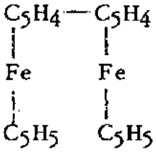
Its rate of deton is 7400 m/sec; compression 26 mm, Trauzl value 460 ml, and stability 1% loss at 80° in 700 hrs and 1% loss at 100° in 53 hrs (Ref 3)

This expl compd may be used in expl mixts as rocket proplnts, in pyrotechnic compns, as Diesel oil additives, and as synthesis intermediates. See also Ref 1a

A new type of gelatinized O-balanced safety expl, known as *"Securit"*, was patented by Wetterholm (Ref 4). Thus a mixt which contd BTNEU 34, starch 0.8, water 6.5, and a mixt of AN & $Ca(NO_3)_2$ 58.7% was prepd (called A) and compared with a 35% NG Ammonia Dynamite (called B). Heat of expln, d, gas vol and confined rate of deton (6000 m/sec) were about the same values for both expls. Small Pb block, gap (25 mm cartridge at 20°), ballistic mortar and rock power tests for A & B were 25, 13-14; 40-5, 180-200; 77.5, 89; and 82, 88, respectively. Expl A may be fired with a No 4 cap and its rate remains almost constant, regardless of cartridge size, while the rate of "B" increases from about 2000 m/sec in 12-mm cartridges to about 5000 m/sec in 45-mm cartridges. After 100 days storage, the rate of A was unchanged while that of B had dropped about 50%. A also showed greater superiority in impact, drilling, friction, splinter initiation, and heat stability tests. A gave no expln at 280°, with evolution of red gas starting at 195°; B exploded at 195° and evolved red gas starting at 155°

Refs: 1) Beil - not found 1a) S. Helf, PATR **1750**(1949) (Some tests of BTNEU) 2) F.R. Schenck & G.A. Wetterholm, SwedP 148217 (1954); BritP 729469 (1955); USP 2731460 (Example 3) (1956) & CA **50**, 1893, 7125 (1956) 3) G.A. Wetterholm; GerP 934694 (1955); BritP 813477 (1959) & CA **53**, 17513, 19884 (1959) 4) G.A. Wetterholm, Compt-RendCongrInternChimInd, 31°, Liège, 1958 (Pub as IndChimBelge,Suppl) **2**, 226-30 (Pub 1959 in English) & CA **54**, 23330 (1960) 5) G.A. Wetterholm, SvenskKemTidskr **76** (11), 628-34 (1964) (Swed) & CA **62**, 11684 (1965)

Diferrocenyl, Biferrocene or Bicyclopentadienylene-bis[cyclopentadienyl iron]



$$\begin{array}{ll} C_5H_4 & -C_5H_4 \\ | & | \\ Fe & Fe \\ | & | \\ C_5H_5 & C_5H_5 \end{array}$$

; mw 370.14, Fe 30.18; orn crysts (from alc), mp - darkens at 205° & melts with decompn at 230°; readily sol in benz; mod sol in petr eth, toluene, dioxane, tetrahydrofuran & alc; was formed by the decompn of diferrocenyl mercury, $(C_5H_5FeC_5H_4)_2Hg$, in the presence of Pd black (Refs)

Refs: 1) Beil - not found 2) O.A. Nesmeyanova et al, DoklAkadN **126**, 1004-06 (1954) & CA **54**, 6673 (1954) 3) O.A. Nesmeyanova & E.G. Perevalova, DoklAkadN **126**, 1007-08 (1959) & CA **54**, 1478 (1960) (Engl translation by J.W. Palmer, Ministry of Aviation, London RAE Trans **853** (Nov 1959) 4) E.G. Perevolova & O.A. Nesmeyanova, DoklAkadN **132**, 1093-94 (1960) & CA **54**, 21027 (1960) (Prepn by Ullmann reaction) 5) H. Shechter & J.F. Helling, JOC **26**, 1034-37 (1961) & CA **55**, 18691-92 (1961) (Prepn from ferrocenyl Grignard reagents) 6) M.D. Rausch, JOC **26**, 1802-05 (1961) & CA **55**, 23473 (1961) (Prepn by Ullmann reaction and IR & UV spectra)

Differential Pressure Measuring for Examination of Propellants. This subject is discussed in a paper by H. Rumpff, Explosivst **5**, 43-45 (1957)

Differential Thermal Analysis of Explosives, Propellants & Pyrotechnics. Differential Thermal Analysis (DTA) involves measuring the temp difference between an inert ref compd *(ignited alumina)* and the material under study as they are both heated to elevated temps at a const rate. Since a ref material is selected which will undergo no thermal reactions over the temp range under investigation, any endo- or exothermal changes of the test sample will cause its temp to be lower or higher, respectively, than that of the ref material, resulting in endo- or exothermal differential temps which are recorded as a function of the sample or furnace temp. DTA provides a continuous record, over the whole temp range under consideration, and of the thermal effects accompanying melting, boiling, crystalline transition, dehydration, decomposition, oxidation & reduction. DTA is therefore worthy of consideration as an adjunct to the multiple of techniques available for characterizing substances, in this case by their relative thermal stabilities and unique behavior at elevated temps

The following Refs give some of the more important studies made with DTA apparatus

Refs: 1) S. Gordon & C. Campbell, "Differential Thermal Analysis of Inorganic Compounds. Nitrates and Perchlorates of the Alkali and Alkaline Earth Groups and their Subgroups", AnalChem **27**, 1102-09 (1955) [Based on PATR **2079** (Nov 1954) & PATR **2200** (July 1955)] 2) S. Gordon & C. Campbell "Preignition and Ignition Reactions of the Pyrotechnic Smoke Composition Zinc-Hexachlorobenzene-Potassium Perchlorate", PATR **2123** (March 1955) 3) V.D. Hogan, S. Gordon & C. Campbell, "Differential Thermal Analysis and Thermogravimetry Applied to Potassium Perchlorate-Aluminum-Barium Nitrate Mixtures", AnalChem **29**, 306-10 (1957) [See also PATR **2373** (Feb 1957) 4) W.J. Smothers & Y. Chiang, "Differential Thermal Analysis", ChemPublCo, NY (1958) 5) S. Lopatin, "Thermal Analysis of D-16 Delay Powders", PA FRL Pyro Rept **PL-C-TN5** (Aug 1957)] 6) E.E. Mason & H.A. Davis, "The Application of Differential Thermal and Thermogravimetric Analyses to Military High Explosives", NAVORD Rept **5802** (Jan 1960) 7) E.E. Mason, "Application of Derivative Differential Thermal Analysis to Military High Explosives". NAVWEPS Rept **6996** (June 1960) 8) R.H. Bohon, "Differential Thermal Analysis of Explosives and Propellants under Controlled Atmospheres", AnalChem **33**, 1451-53 (1961) 9) R.N. Rogers, "The Simple Microscale Differential Thermal Analysis of Explosives", MicrochemJ **5**, 91-99 (1961) 10) E.E. Mason & D.H. Zehmer, "The Feasibility of Using Differential Thermal Analysis to Determine the Characteristics of Selected Primary Explosives", NAVWEPS Rept **6997** (June 1961) 11) P.G. Rivette & E.D. Besser, "Differential Thermal Analysis as a Research Tool in Characterizing New Propulsion Systems", NAVWEPS Rept **7769** (Oct 1961) 12) M.E. Baicar, "An Apparatus for the Differential Analysis of Explosives", NAVWEPS Rept **7107** (Dec 1961) 13) C.D. Lind, "The Thermal Decomposition Characteristics of Explosives", NAVWEPS Rept **7798** (Feb 1962) 14) G.O. Piloyan, I.D. Ryabchikov & O.S. Novikova, "Determination of Activation Energies of Chemical Reactions by Differential Thermal Analysis", Nature **212**, 1229 (1966) 15) J.D. Burnett et al, "The Characterization of Solid Rocket Propellants by Differential Thermal Analysis", US Army Missile Command Rept **RK-TR-63-21** (Sept 1963) 16) K.G. Scrogham, "Application of DTA to the Thermal Decomposition of Propellant and Propellant Ingredients", Rohm & Haas Rept **S-61** (June 1965) [Contract DA-01-021-ORD-11909(2)] 17) G.M. Lukaszewski, "Differential Thermal Analysis, Part II. The Present Status of Thermal Analysis", LabPractice **14**, 1399-1405 (1965); Part V."The Physical and Mathematical Models", LabPractice **15**, 187-89 (1966); and Part XI. "A Central Consideration of Thermoanalytical Systems", LabPractice **15**, 861-62, 869 (1966) 18) D.R. Satriana et al, "Differential Thermal Analysis of Nitramines and Guanidine Derivatives", PATR **3373** (May 1966)

Diffusion (Diffusivity). It is a spontaneous process of equalization of physical states: a) diffusion of temperature by heat conduction, b) diffusion of concentration in a solution by

which the particles in soln become uniformly distributed, and
c) diffusion of gases when one gas is liberated into another

The term diffusion is also applied to the process of reflection of light by a rough reflecting surface, or of transmission of light thru a translucent material

The phenomenon of diffusion may be used to separate various materials. Benedict and Halle et al in Kirk & Othmer (Refs 12 & 27) describe several devices for diffusion separations, such as mass diffusion, thermal diffusion & gas diffusion apparatuses

For addnl info on this subject, see the following Refs on Diffusion:

Refs: 1) J. Duclaux, "Diffusion dans les Liquids. Diffusion dans les Gels et les Solids", Hermann, Paris (1936) 2) A.P. Colburn, IEC **33**, 459-67 (1941) (Simplified calculation of diffusional processes) 3) R.M. Barrer, "Diffusion In and Through Solids," CambridgeUnivPress,London (1941) 4) Symposium on the Diffusion of Electrolytes and Macromolecules in Solution, AnnNYAcadSci **46**, 211-39 (1945) 5) F.H. Holmes, "Reviews of Literature on Diffusion in Solution and the Estimation of Particle Size from Diffusion Measurements", ShirleyInst, Didsbury, Manchester (1945) 6) A. Weissberger, ed, "Physical Methods of Organic Chemistry", Interscience, NY, Vol 1 (1945), pp 227-310 & 2nd edit, Vol 1, Part 1 (1949), 551-619 (by A.L. Geddes, "Determination of Diffusivity") 7) R. Höber, "The Physical Chemistry of Cells and Tissues", Churchill, London (1946), Chapter 1 8) H.S. Harned, ChemRevs **40**, 461-522 (1947) (Quantitative aspect of diffusion in electrolytic solutions) 9) R.B. Dean, ChemRevs **41**, 503-23 (1947) (Effects produced by diffusion in aqueous systems containing membranes) 10) D.A. Hougen & K.M. Watson, "Chemical Process Principles", Part 3, "Kinetics & Catalysts", Wiley, NY (1947), Chap 20 11) Perry (1950), pp 522-59 (by A.P. Colburn & R.L. Pigford, "General Theory of Diffusional Operations", includes sections on equilibrium relationships, rate of mass transfer, countercurrent apparatus, and simultaneous heat & mass transfer) 12) Kirk & Othmer, Vol **5** (1950), pp 76-133 (by M. Benedict, "Diffusion") 13) W. Jost, "Diffusion in Solids, Liquids, Gases", Academic Press, NY (1951), 550 pp 14) T.K. Sherwood & R.L. Pigford, "Absorption and Extraction", McGraw-Hill, NY (1952), pp 1-28 15) R.M. Barber, "Diffusion in and through Solids", Cambridge Univ Press, NY (1952), 477 pp 16) K.E. Grew & T.L. Ibbs, "Thermal Diffusion in Gases", Cambridge Univ Press, NY (1952), 154 pp 17) D.A. Frank-Kamenetskii, "Diffusion and Heat Exchange in Chemical Kinetics", Princeton Univ Press, NJ (1955), 370 pp (Translated from Russian by N. Thon) 18) J. Crank, "The Mathematics of Diffusion", Clarendon Press, Oxford (1956), 347 pp 19) W. Jost, "Diffusion. Methoden der Messung und Auswertung", Steinkopff, Darmstadt, Germany (1957), 177 pp 20) P. Chang & Y Shen, "An Introduction to Diffusion for Chemical Engineers", Commercial Press, Taiwan, China (1958), 80 pp 21) B. Ottar, "Self-Diffusion and Fluidity in Liquids", Oslo Univ Press, Oslo (1958), 140 pp 22) V. Vitagliano, "Introduzione allo Studio della Diffusione nei Liquidi", D'Agostino, Napoli (1959), 109 pp 23) E.J. Conway, "Microdiffusion Analysis and Volumetric Error", Crosby Lockwood & Son, London, 5th edit (1962), 454 pp 24) K.E. Grew & T.L. Ibbs, "Thermodiffusion in Gasen", Bd **7**. Physikalische-Chemische Tren- und Messmethoden, Deut Verlag Wissenschaften, Berlin (1962), 170 pp 25) P.G. Shewmon, "Diffusion in Solids", McGraw-Hill, NY (1963), 203 pp 26) C.N. Satterfield & T.K. Sherwood, "The Role of Diffusion in Catalysis", Addison-Wesley Publ Co, Reading, Mass (1963), 118 pp 27) Kirk & Othmer, 2nd edit **7** (1965), pp 79-175 (E. VonHalle, R.L. Huglund & J. Shacter, "Diffusion" and "Diffusion Separation Methods") 28) E.D. Oliver, "Diffusional Separation Processes: Theory, Design, and Evaluation", Wiley, NY (1966), 444 pp

Diffusion Coefficients of Solventless Propellants is determined by J. Chosson, MP **32**, 255-78 (1950) & **33**, 357-69 (1951)

(Equations are presented and their application shown for "Poudres SD" contg NC, DEGDN, Centralite, petrolatum and K_2SO_4)

Diffusion of Nitroglycerin in Double-Base Propellants is discussed in the following repts:
1) ABL, "Diffusion of Nitroglycerin in Wrapped Powder Grains", OSRD **4963** (PB Rept 50864) (1945)
2) S.S. Penner & S. Sherman, JACS **70**, 869-70 (1948) (Diffusion of NG in rocket propln and in cellulose acetate)
3) R.G. Wetton, "Diffusion of Nitroglycerin and Other Materials between Double-Base Propellant and Cellulose Acetate Inhibitor", PATR **1993** (Feb 1954)

Di-flamyr (Welsh for Flameless). A proplnt manufd for some time, beginning 1889, by the Smokeless Explosives Co, Ltd, Barwick, Herts. It consisted of Guncotton and inorganic nitrates, except those of Ba or Ammonium
Ref: Daniel (1902), 205

Difluoramino Compounds. Some compds of interest in the field of expls are described in the following classified repts:
Refs: 1) Thiokol Chemical Corp, Reaction Motors Div, "Difluoramine Chemistry", RMD Rept **076-S-62** (1 Jan to 31 Dec 1962) [Contract NOnr 1878(00)] (Conf) 2) B.J. Levy, "A Study of the Explosion Limits of Simple Difluoramino Compounds", Atlantic-ResCorp Quarterly Tech Summary Rept **4** (1 Jan to 31 March 1964); and Quarterly Tech Summary Rept **9** (1 April to 30 June 1965) [Contract NOnr 4065(00)] (Conf)

Difluorenyl-diperoxide. See 9,9'-Bis(fluorenyl)-diperoxide in Vol 2 of Encycl, p B143-L

Di(fluorenylidenehydro)-tetrazine. See 3,6-Bis(fluorenylidenehydro)-sym-tetrazine in Vol 2, p B143-L

Difluorobenzene and Derivatives

Difluorobenzene (called Difluor-benzol in Ger), $C_6H_4F_2$; mw 114.09. Three possible isomers are known:
1,2-Difluorobenzene, aromatic smelling liq, fr p -34°, bp 93° at 759 mm press, d 1.1496 at 25°, n_D 1.4451 at 18°; decomp in a stream of air (Ref 1)
1,3-Difluorobenzene, liq, fr p -59°, bp 83.1°, d 1.1572 at 20°, n_D 1.4404 at 18° (Ref 2)
1,4-Difluorobenzene, liq, fr p -23.7 to -13°, bp 82-83° at 752 mm & 88.95° at 763 mm press, d 1.1722 at 18.9°, n_D 1.4422 at 18.9°; $Q^v_{combstn}$ 704.3 kcal/mol (Ref 3)

Other props & methods of prepn are given in Beil

Refs: 1) Beil **5**, [147] & {523} 2) Beil **5**, (108), [147] & {523} 3) Beil **5**, 199, (108), [147] & {523}

Azido, $C_6H_3F_2N_3$, and *Diazido*, $C_6H_2F_2N_6$, derivs of Difluorobenzene were not found in Beil or in CA thru 1961

Mononitrodifluorobenzene, $C_6H_3F_2.NO_2$; mw 159.09, N 8.81%. Three isomers are found in Beil:
4-Nitro-1,2-difluorobenzene (called 3.4-Difluor-1-nitro-benzol in Ger), liq, bp 80-81° at 14mm press (Ref 2); bp 76-80° at 11 mm press, n_D 1.5055 at 20° (Ref 5)
4-Nitro-1,3-difluorobenzene (called 2.4-Difluor-1-nitro-benzol in Ger), liq, fr p 9.75°, bp 206.9°, d 1.4571 at 13.9°, n_D 1.5149 at 13.9° (Ref 1) & n_D 1.5150 at 20° (Ref 5)
2-Nitro-1,4-difluorobenzene (called 2.5-Difluor-1-nitro-benzol in Ger), yel liq, fr p 11.7°, bp 206.5°, d 1.4671 at 17.2° & 1.4571 at 13.9°, n_D 1.5149 at 13.9° (Ref 1)

Other props & methods of prepn are found in Beil

Refs: 1) Beil **5**, (129) & [180] 2) Beil **5**, {609} 3) F. Weygand et al, ChemBer **84**, 101 (1951) & CA **45**, 5164-65 (1951) (Prepn of 4-Nitro-1,2- and 2-Nitro-1,4-difluoro derivs) 4) G.C. Finger et al, JACS **73**, 153 (1951) & CA **45**, 7035 (1951) (Prepn & props of 5-Nitro-1,3-difluorobenzene, fr p 17.5°, bp 78-79° at 20 mm & 176-77° at 760 mm press) 5) G.C. Finger & C.W. Kruse, JACS **78**, 6034 (1956) & CA **51**, 4975 (1957) 6) N.M. Mel'nikov et al, RussP 113276 (1958) & CA **53**, 2530 (1959) (Use of Mononitrodifluorobenzene & other fluoro derivs in insecticides for treating seed) 7) T.L. Fletcher et al, JOC **25**, 1346(1960) & CA **54**, 24591(1960) (Prepn of 4-Nitro-1,3-difluoro deriv)

Dinitrodifluorobenzene, $C_6H_2F_2(NO_2)_2$; mw 204.10, N 13.42%. Only one isomer is described in the literature:

4,6-Dinitro-1,3-difluorobenzene (DNDFB) or 2,4-Dinitro-1,5-difluorobenzene (DNFFB) (called 4,6-Difluor-1,3-dinitro-benzol in Ger), prisms (from alc), mp 73-75°; readily sol in hot alc; less sol in cold alc; can be prepd by nitrating either 1,3-difluorobenzene or 1-nitro-2,4-difluorobenzene with mixed acids (Ref 1) or by other methods (See Refs)

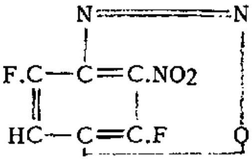
Refs: 1) Beil **5**, (136) 2) H. Zahn, Melliand-Textilber **31**, 762(1950); Kolloid-Z **121**, 39-45 (1951) & CA **45**, 5934, 7357(1951) (Use is stabilizing wool & silk against phenols & acids) 3) H. Rosenberg & J.C. Mosteller, IEC **45**, 2286(1953) & CA **48**, 307(1954) (Used to protect US Air Force fabrics against fungus attack) 4) H.Zahn & A. Würz, BiochemZ **325**, 182(1954) & CA **49**, 3864(1955) (Reactions of amine, imine, phenol, thiol & imidazole with DNFFB) 5) H. Zahn & A. Würz, TextileResJ **25**, 118(1955) & CA **49**, 5848(1955) (Protection of wool against fungus attack) 6) M.J. Newlands & F. Wild, JCS **1956**, 386 & CA **51**, 4293(1957) (Jánovsky reaction) 7) Farbwerke Hoechst A-G,vorm Meister Lucius & Brüning, BritP 75568(1956) & CA **51**, 9686(1957) (Use of DNFFB as wood preservative, pesticide & dye intermediate) 8) N.N. Vorozhtsov & G.G. Yakobson, NauchDoklVyssheiiShkoly, Khim i Khim-Teckhnol **1958**, No 1, 122 & CA **53**, 3110 (1959) (Prepn from corresponding dichloro deriv) 9) K. Fukui et al, NipponKagaky-Zasshi **79**, 1120(1958) & CA **54**, 5518(1968) (Prepn in 88% yield) 10) T.L. Fletcher et al,JOC **25**, 1346(1960) & CA **54**, 24591(1960) (Prepn, mp 73-74°)

2,4,6-Trinitro-1,3-difluorobenzene (TNDFB) or 1,3-Difluoro-2,4,6-trinitrobenzene (DFTNB), $C_6HF_2(NO_2)_3$; mw 249.09, N 16.87%; wh ndls (from boiling CCl_4), mp 147°, bp - dec at 240°; was prepd in 28% yield by nitrating the above dinitro deriv with an acid mixt of KNO_3 & fuming H_2SO_4 for 12 hrs at 180° (Ref 3). The expl & thermal props of DFTNB are given in a classified report by Siele & Warman (Ref 2)

Refs: 1) Beil - not found 2) V.I. Siele & M.Warman, Picatinny Arsenal **FRL-TN-124** (Jan 1962) 3) V.I. Siele & M Warman, "Preparation of 1,3-Difluoro-2,4,6-trinitrobenzene", JOC **27**, 1910(1962)

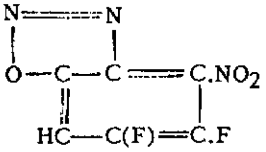
Difluorobenzene Diazooxide, $C_6H_2F_2NO_0$, a compd not found in Beil or in CA thru 1961, may be considered the parent compd of its nitro derivs, although not used to prep them:

3,6-Difluoro-2-nitro-1-diazo-4-oxide (?), $C_6HF_2N_3O_3$; mw 201.09, N 20.90%;



; bright orn-colored crysts (from MeOH), mp - darkens on heating w/o melting up to 350°; explodes violently on impact; was obtd by diazotizing 2-nitro-3,4,6-trifluoroaniline in concd HCl with aq $NaNO_2$. The ppt melted near 80°, burned explosively in a flame, and on crystn from MeO gave the above product (Ref 2)

3,4-Difluoro-2-nitro-1-diazo-6-oxide (?),



; bright orn crysts (from MeOH), mp 119.3-120° dec; explodes violently on impact; was prepd by similar diazotization as above compd, except 48% aq HF was used (Ref)

No structural assignments were made to the diazo oxides because of the uncertain positions of the labilized fluorine atoms

Refs: 1) Beil - not found 2) G.C. Finger et al, JACS **73**, 148(1951) & CA **45**, 7034 (1951)

1,3-Diformadinoguanidine,

NH.NH.CHO
|
C=NH
|
NH.NH.CHO; mw 145.13, N 51.02%; was isolated in the form of its *Nitrate salt*, $C_3H_8N_6O_5$, silky wh ndls (from alc), mp 155° (5% yield), in addn to 3,4-diamino-1,2,4-triazole, $C_2H_6N_6O_3$, wh ndls (from alc), mp 198-99°, when diaminoguanidine nitrate & formic acid in dioxane were refluxed on a steam bath for 2 hrs (Ref 2)

No expl props of the compds were reported

Refs: 1) Beil - not found 2) E. Lieber et al, JOC **18**, 227 (1953) & CA **48**, 1344 (1954)

sym-Diformylhydrazine or 1,2-Diformylhydrazine (DFH) (called N.N'-(or Symm)-Diformylhydrazin in Ger),

NH.CHO
|
NH.CHO; mw 88.07, N 31.81%; prisms, mp 159-60°; can be prepd by heating, for several hrs at 100-30°, hydrazine hydrate with an excess of the ethyl ester of formic acid, or by heating at 100° hydrazine sulfate & formylhydrazide in abs alc; readily sol in w; diffc sol in alc; insol in eth (Refs 1, 4 & 5)

It forms salts, some of which explode on rapid heating:

Lead salt, $PbC_2H_2N_2O_2$, amor ppt; prepd by treating an ice-cold aq soln of disodium diformylhydrazine with neutral lead acetate (Ref 3)

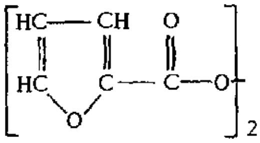
Mercurous salt, $HgC_2H_2N_2O_2$, fine wh ppt; obtd from diformylhydrazine, Na methylate & mercurous chloride in aq-alcoholic soln (Ref 6)

Silver salt, $Ag_2C_2H_2N_2O_2$, almost wh crysts, explodes violently when heated rapidly leaving a Ag residue; was prepd from diformylhydrazine, NH_3 & $AgNO_3$ in cold aq soln (Ref 6)

Sodium salt, $Na_2C_2H_2N_2O_2$, ndls; very sol in w; insol in abs alc; was prepd by mixing an ice-cold aq soln of diformylhydrazine with Na ethylate (Ref 2)

Refs: 1) Beil **2**, 93, (38) & {127} 2) C.D. Harries, Ber **27**, 2276 (1894) 3) C.D. Harries & E. Klamt, Ber **28**, 503 (1895) 4) T. Curtius et al, JPraktChem **51**, 182 (1895) 5) G. Pellizzari, GazzChimItal **39** I, 529 (1909) 6) R. Stollé, Ber **45**, 283 (1912) & CA **6**, 1004 (1912) 7) J.P. Picard & J.L. Boivin, CanJChem **29**, 224 (1951) & CA **45**, 9469 (1951) (Attempted nitration of DFH yielded gaseous products) 8) R.L. Hinman, JACS **78**, 1645 (1956) & CA **50**, 14512 (1956) (Reaction with $LiAlH_4$) 9) K. Suzuki et al, BullChemSocJapan **29**, 127-31 (1956) (in Engl) & CA **51**, 790 (1957) (Vapor pressure & H-bond energy)

Di-2-furoyl Peroxide or Furoyl Peroxide (called Difurfuroylperoxyd or Furfuroylperoxyd in Ger),

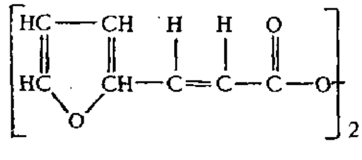


2; mw 178.14, O 53.89%; lt-yel ndls (from alc), mp 86-87° dec, darkens on exposure to light; explodes violently on heating in a flame, or on impact or friction; sol in eth, acet methanol, chlf, CCl_4 or benz; insol in w or petr eth; can be prepd by reacting aq Na peroxide & furoyl chloride at 0° (Refs 1, 2 & 3). Cooper (Ref 4) studied the effect of structure of diacyl peroxydes on their radical-induced decompn in vinyl monomers

Refs: 1) Beil **18**, [267] 2) H. Gelissen & J.D. van Roon, Rec **43**, 362 (1924) & CA **18**, 2515 (1924) 3) N.A. Milas & A. McAlevy, JACS **56**, 1219 (1934) & CA **28**, 4413 (1934) 4) W. Cooper, JCS **1952**, 2412 & CA **47** 3087 (1953)

Difuryl. See Bifuryl in Vol 2 of Encycl, p B113-L

Difurylacryloyl Peroxide or Bis(β-2-furylacrylyl) Peroxide (CA nomenclature)



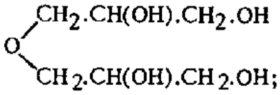
2; mw 274.22,

O 35.01%; wh ndls (from 1:1 ether/ethyl acet), mp 104° dec; explodes violently on heating in a flame; sol in acet & ethyl acet; sl sol in eth; was synthesized from aq Na peroxide & furylacryloyl chloride in anhyd eth soln at 0° (Ref 2)

Refs: 1) Beil - not found 2) N.A. Milas & A. McAlevy, JACS **56**, 1219 (1934) & CA **28**, 4413 (1934) 3) Not found in later CA's thru 1961

Diglycerol and Derivatives

Diglycerol; Diglycerin; Diglyceryl Ether; Diglyceryl Alcohol; Bisdihydroxy propyloxide; or 3,3'-Oxy-di(1,2-propanediol) (CA nomenclature) [called Diglycerin; Glycerin-1-(2.3-dihydroxy-propyläther) or Bis-(2.3-dihydroxy-propyl)-äther in Ger],



$$O\begin{cases}CH_2.CH(OH).CH_2.OH\\CH_2.CH(OH).CH_2.OH\end{cases}$$

; mw 166.17, O 48.14%; viscous, hygro liq, bp 265-70° at 14-15 mm press, d 1.279 at 20°, viscosity at 50° 7.316 g/cm sec, n_D 1.489 at 20°; v sol in hot water; sl sol in cold w; insol in eth; can be prepd by gently boiling glycerin for 5-6 hrs & distilling off the water; by heating glycerin to 290-95° in the presence of 0.5% Na_2CO_3 or Na_2S; by the oxidation of diallylether with $KMnO_4$ & H_2CO_3; or by other methods (Ref 1)· See Refs

It is used for the prepn of its expl tetranitro deriv

Refs: 1) Beil **1**, 513, (272), [591] & {2327} 2) Thorpe **6** (1943), 70 3) H.J. Wright & R.N. DuPuis, JACS **68**, 446 (1946) & CA **40**, 2795 (1946) 4) H. Wittcoff et al, JACS **71**, 2666 (1949) 5) Société Solvay et Cie, BelP 554639 (1957) & CA **53**, 21671 (1959) 6) Anon, IEC **50**, 41A (1958)

Diglycerol Tetranitrate; Tetranitrodiglycerin (TeNDG); or 3,3'-Oxydi(1,2-propanediol) Tetranitrate; Di(β,γ-dihydroxypropyl)-ether Tetranitrate; or Di(propylol)-ether Tetranitrate (called Diglycerin-tetranitrat; "Tetranitrodiglycerin", or Tetrasalpetersäureester des Diglycerins in Ger), $O[O_2N.O.CH_2.CH(O.NO_2).CH_2]_2$; mw 346.17, N 16.19%; thick, oily, nonhygro liq, fr p - none, bp - dec, d 1.33; insol in water; sl sol in alc & eth; like NG, it dissolves in most org solvs; can be prepd by nitrating Diglycerol with mixed acid in a manner similar to that of glycerol, but less acid is needed (Ref 1)

The expl props of TeNDG are summarized by Blatt (Ref 5) and Urbański (Ref 10) as follows:

Heat of expln - 1370 cal/g (Ref 8)

Impact sensitivity, 2 kg wt 8 to 10 cm; sensitivity increased by presence of entrapped air bubbles (Refs 6 & 7)

Power by Pb block test 86-87% as powerful as NG

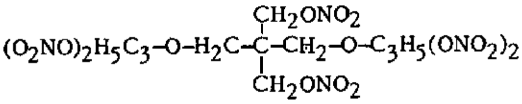
Velocity of detonation, 2050 m/sec (Ref 7)

TeNDG alone is not a satisfactory gelatinizer for NC, but mixed with NG it gelatinizes NC and, hence, has been used as a low-freezing ingredient of NG dynamites. It is superior to Dinitrochlorohydrin (qv) since it does not evolve HCl among its expln products. TeNDG came into broad use in Gt Britain between 1920 & WWII (Ref 10). Cook (Ref 3) patented an expl compn consisting of NH_4ClO_4 or $KClO_4$ 21-30, MnO_2 4-7, $NaNO_3$ 17-47.5, TNT 23-26 & TeNDG, NG or Dinitrochlorohydrin 2%, plus small amts of $CaCO_3$, S or woodmeal added

Refs: 1) Beil **1**, 515 & {2328} 2) C. Claessen, GerP 181754 (1906) & CA **1**, 2440 (1907); BritP 6314 (1906) 3) R.M. Cook, USP 1440767-68 (1923) & CA **17**, 1143 (1923) 4) Naoúm, NG (1928), 199-201 5) Blatt OSRD **2014** (Feb 1944) 6) R.G. Vines, Nature **160**, 400 (1947) 7) F.P. Bowden & O.A. Gurton, PrRoySoc **198A**, 353 (1949) 8) Orlova (1961), p 327, Engl trans p 620 9) Sax (1963), 722 10) Urbański **2** (1965), 139

Diglycerolpentaerythritol Ether, $C_{11}H_{24}O_8$; mw 284.30, O 45.02%; crysts, mp 72.5-74°, softens at 69° when heated rapidly; prepd by hydroxylating diallyl pentaerythritol ether (Ref 2); is the parent compd of its hexanitrate deriv

Diglycerolpentaerythritol Ether Hexanitrate,

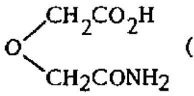


$$(O_2NO)_2H_5C_3{-}O{-}H_2C{-}\underset{\displaystyle CH_2ONO_2}{\overset{\displaystyle CH_2ONO_2}{\underset{|}{\overset{|}{C}}}}{-}CH_2{-}O{-}C_3H_5(ONO_2)_2$$

mw 554.30, N 15.16%; crysts, d 1.540 at 20°, n_D 1.4878 at 20°; was prepd by nitrating the parent compd (Ref 2). It is a possible gelatinizer for NC
Refs: 1) Beil - not found 2) R. Evans & J.A. Gallaghan, JACS **75**, 1249(1953) & CA **49**, 3811(1955)

Diglycol (Diglykol in Ger). Same as Diethyleneglycol (DEG or DEtGc)

Diglycolamic Acid or Diglycolic Acid Monoamide may be represented as



$$O{<}\begin{matrix}CH_2CO_2H\\ CH_2CONH_2\end{matrix}$$

(See also Diglycolamidic Acid)

(called Dimethyläther-α,α'-dicarbonsäure-monoamid; Diglykolsäuremonoamid; or Diglykolamidsäure in Ger), mw 133.10, N 10.52%; prisms, mp 135°, sinters at 125-30°; partially sol in w & alc; forms a *Barium salt*, $Ba(C_4H_6NO_4)_2 + H_2O$, crysts; sol in w; insol in alc (Ref). Other props & method of prepn are found in Beil
Ref: Beil **3**, 241-42

NOTE: No *Azido*, $C_6H_6N_4O_4$, or *Nitramino*, $C_4H_6N_2O_6$, derivs were found in Beil or in CA thru 1961

Diglycolamidic Acid and Derivatives

Diglycolamidic Acid or Iminodiacetic Acid, may be represented as $HN(CH_2COOH)_2$. Some of its derivatives are explosive. Eg: Diglycolamidic Diazide (qv) and Nitroso derivs (qv)

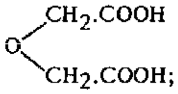
Diglycolamidic Diazide, Iminodiacetic Azide or Iminobisaceticazide (Diglykolamidsäure-diazid or Iminodiessigsäure-diazid in Ger), formerly called Diglycolamic Diazide $HN(CH_2.CO.N_3)_2$. Its *nitrite*, $C_4H_5N_7O_2 + HNO_2$, colorless needles (from eth), was obtained (Ref 2) in small quantity by treating the trihydrochloride of diglycolamic acid dihydrazide with $NaNO_2$ in aq soln. The nitrite exploded on heating and decomposed in storage
Refs: 1) Beil **4**, (482) 2) T. Curtius et al, JPrCh [2] **96**, 218(1917) & CA **12**, 2318(1918)

Nitrosodiglycolamidic Diazide, $ON.N(CH_2.CON_3)$; mw 212.13, N 52.83%; yel oil, expl on heating; insol in w; sol in eth; was prepd by reacting an acetic acid soln of Nitrosodiglycolamidic Dihydrazide & $NaNO_2$ with some HCl at low temp
Refs: 1) Beil **4**, 380 & (488) 2) T. Curtius et al, Ber **41**, 358(1908)

Nitrodiglycolamidic Acid (called Nitrodiglykolamidsäure or Nitroiminodiessigsäure in Ger), $O_2N.N(CH_2.COOH)_2$; mw 178.10, N 15.73%; yel lfts or crysts (from eth acetate), mp 148-55° (dec); sol in w, MeOH, alc, acet & ethyl acetate; insol in chlf & benz; was prepd by nitrating the parent acid compound; forms salts, some of which are expl (Refs):
Copper salt, $CuC_4H_4N_2O_6 + 2H_2O$, greenish microcryst powder, explodes on heating
Potassium salt, $K_2C_4H_4N_2O_6 + H_2O$, tablets (from w + alc), dec explosively at 195°
Silver salt, $Ag_2C_4H_4N_2O_6$, wh ppt, explodes on heating
Zinc salt, $ZnC_4H_4N_2O_6$, crysts, explosive
Refs: 1) Beil **4**, 380 & (488) 2) A.P.N. Franchimont & J.V. Dubsky, Rec **36**, 99(1917) & CA **11**, 45-48(1917) 3) J.V. Dubsky & M. Spritzmann, JPraktChem **96**, 108, 109, 117 (1917) & CA **12**, 1775(1918)

Nitrodiglycolamic Acid Dinitril, $O_2N.N(CH_2.CN)_2$; mw 140.10, N 39.99%; col ndls (from anhyd benz), mp - softens at 98° & sl decomp at 104-05°; sl sol in benz, alc & eth acet; v sl sol in w & chlf; insol in petr eth; was obtd when the nitrate salt of Iminodiacetic Acid Dinitrile, $HN(CH_2.CN)_2 + HNO_3$, in 15 parts abs HNO_3 was evaporated at RT (Refs 1 & 2)
Refs: 1) Beil **4**, (488) 2) A.P.N. Franchimont & J.V. Dubsky, Rec **36**, 106(1917) & CA **11**, 47(1917)

Diglycolic Acid, Diglycollic Acid or Oxydiacetic Acid [called Diglykolsäure; Dimethyläther-α.α'-dicarbonsäure or (Carboxymethoxy)-essigsäure in Ger].

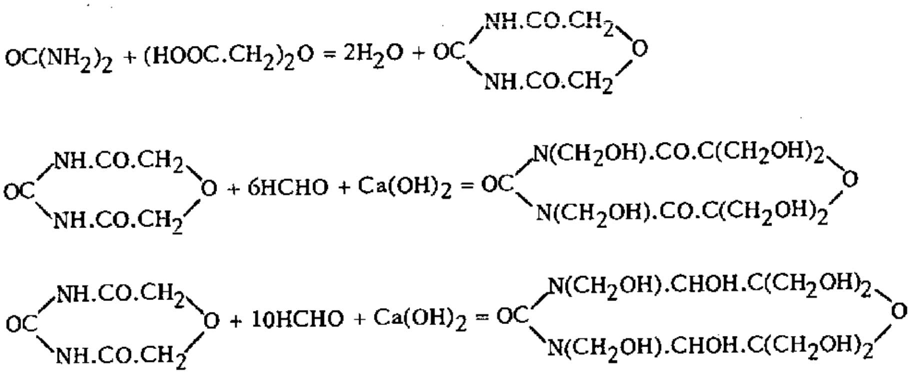


$$O{<}\begin{matrix}CH_2.COOH\\ CH_2.COOH\end{matrix}$$

; mw 134.09, O 59.66%; wh

crysts (from w), monoclinic prisms, mp 148°, bp - dec; forms a non-hygr monohydrate at RH above 72% & temp 25°; readily sol in w & alc; less sol in eth; can be formed together with glycolic acid on boiling monochloroacetic acid with $Ca(OH)_2$, $Ba(OH)_2$, or $Mg(OH)_2$; and by the oxidation of diethylene glycol (Ref 1 & 4)

Rinkenbach (Ref 2) conceived in 1953 a reaction between diglycolic acid & urea:



$$OC(NH_2)_2 + (HOOC.CH_2)_2O = 2H_2O + OC\left\langle\begin{array}{l}NH.CO.CH_2\\NH.CO.CH_2\end{array}\right\rangle O$$

$$OC\left\langle\begin{array}{l}NH.CO.CH_2\\NH.CO.CH_2\end{array}\right\rangle O + 6HCHO + Ca(OH)_2 = OC\left\langle\begin{array}{l}N(CH_2OH).CO.C(CH_2OH)_2\\N(CH_2OH).CO.C(CH_2OH)_2\end{array}\right\rangle O$$

$$OC\left\langle\begin{array}{l}NH.CO.CH_2\\NH.CO.CH_2\end{array}\right\rangle O + 10HCHO + Ca(OH)_2 = OC\left\langle\begin{array}{l}N(CH_2OH).CHOH.C(CH_2OH)_2\\N(CH_2OH).CHOH.C(CH_2OH)_2\end{array}\right\rangle O$$

which on nitration of the hexamethylol & octamethylol compds should yield hexanitrate & octanitrate derivs that would be powerful expls

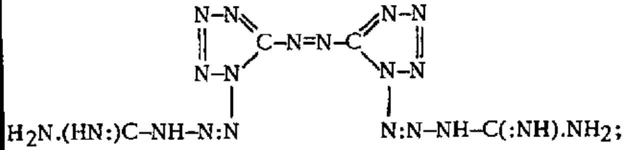
Diglycolic Acid is now used in the manuf of resins, plasticizers, and in org synthesis (Refs 3 & 4)

Refs: 1) Beil **3**, 234, (90) & {376} 2) W.H. Rinkenbach, Private Notes (2 March 1952) 3) CondChemDict (1961), 382 4) Kirk & Othmer, 2nd edit **10** (1966), 636

Diguanide. See Biguanide in Vol 2, p B114-L

Diguanidine. See Biguanidine in Vol 2, p B115-L

Di[1(3-guanyltriazene)-5-tetrazolyl]-diazene or Bis[1(guanyl-3-triazene-1-tetrazolyl-5]-diazene (called Bis-1,1(3-guanyltriazene)-5,5-azotetrazole by Lieber & Smith; and Azotetrazoldiazoguanidin by Thiele),



mw 336.26, N 83.32%; yel crysts, mp - not reported; decomp in warm water with evolution of gas; behaves like a salt, giving the same reactions as azotetrazoles & diazoguanidines; was prepd from guanyl azide & Na azotetrazole (Refs 2 & 3). This compd is of special interest because it contains two chains of seven nitrogen atoms. Its expl props were not detd

Refs: 1) Beil - not found 2) J. Thiele, Ann **303**, 61-62 (1898) 3) E. Lieber & G.B.L. Smith, ChemRevs **25**, 248 (1939)

Diharnstoff (Ger for Diurea). See Note under 4-Aminourazole in Vol 1, p A272-L and Diurea in this Vol

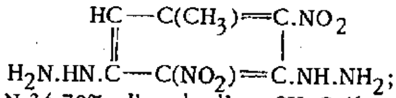
Di(hexahydrobenzoyl)-peroxide. See Bis(hexahydrobenzoyl)-peroxide in Vol 2, p B143-L

Dihydrazide-dihydro-tetrazine. See 3,6-Bis (hydrazide)-1,2-dihydro-1,2,4-tetrazine in Vol 2, p B143-R

Di(hydrazino)-benzene, Dinitro. See 1,3-Bis (hydrazino)-4,6-dinitrobenzene in Vol 2, p B143-R

Dihydrazinotoluene, $C_7H_{12}N_4$, may be considered the parent compd of its dinitro deriv, although not used to prep it:

2,4-Dinitro-3,5-dihydrazinotoluene (called 2.4-Dinitro-3.5-dihydrazino-toluol in Ger),



$$\begin{array}{l} HC-C(CH_3)=C.NO_2 \\ \;\;\| \qquad\qquad\quad | \\ H_2N.HN.C-C(NO_2)=C.NH.NH_2 \end{array}$$

; mw 242.20, N 34.70%; dk red ndls + $2H_2O$ (from dil alc), mp - dec at 173°; was obtd by heating 3,5-dibromo-2,4-dinitrotoluene with hydrazine-hydrate in alc on a water bath (Refs 1 & 2)
Refs: 1) Beil **15**, [267] & **25**, [545] 2) W. Borsche & W. Trautner, Ann **447**, 15 (1926) & CA **20**, 1222 (1926)

NOTE: No *Azido,* $C_7H_{11}N_7$, or *Trinitro,* $C_7H_9N_7O_6$, derivs were found in Beil or in CA thru 1961

Dihydrazone Oxamide (called Oxaldiimidsäure-dihydrazid, "Carbohydrazimin" or "Cyanhydrazin" in Ger), $(H_2N.N:)(H_2N)C.C(NH_2)(:N.NH_2)$ or $(H_2N.HN)(HN:)C.C(:NH)(NH.NH_2)$; mw 116.13, N 72.37%; ndls + H_2O (from w or 20% alc), mp - dec; sol in water, but decomp giving a wh ppt; insol in org solvs; can be prepd by slowly introducing $(CN)_2$ into $N_2H_4.H_2O$ & cold 50% alc, or in 60% yield from dithioxamide in 20% alc and $N_2H_4.H_2O$ added dropwise, and quantitatively from $[C(:NH)OC_2H_5]_2$ & excess $N_2H_4.H_2O$ (Refs 1 & 2)

Dihydrazone Oxamide forms salts, such as:
Hydrochloride, $C_2H_8N_6.2HCl$, yel ndls, swells w/o melting, more stable than its parent compd
Nitrate, $C_2H_8N_6.2HNO_3$, mother-of-pearl-colored cryst
Sulfate, $C_2H_8N_6.H_2SO_4$, wh crysts and other salts similar to those of NH_3 & Hydrazine Hydrate, some insol & others with characteristic colors (Ref 2)

The expl props of the compd and of its salts were not detd
Refs: 1) Beil **2**, 560 2) G. Dedichen, AvhandlNorskeVidenskaps-AkadOslo, I, Mat-Naturv,Klasse **1936**, No 5, 42 pp & CA **31**, 4985 (1937)

Dihydro-. A prefix indicating the presence of two additional hydrogen atoms in an organic compd

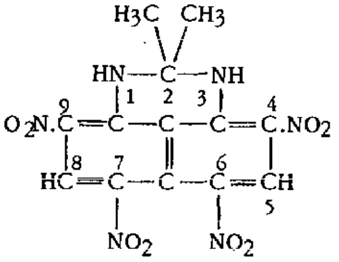
Di(hydroanthranol) or Dihydroxy-di(hydroanthracene). See Bis(hydroanthranol) in Vol 2, p B144-L

4',4-Dihydro-5',5-azinotetrazole; Azino-di[(4-hydroxytetrazole)-5]; (Ditetrazolyl-5)-hydrazine; 5,5'-Hydrazo-ditetrazole; or Hydrazo-di(tetrazolyl-5)· See Bis(5-tetrazole)-hydrazine in Vol 2, p B157-R and in the paper of F.R. Benson, ChemRevs **41**, 8(1947)

Dihydrodiketonebenzene; Dioxybenzene or Quinone. See Benzoquinone in Vol 2, p B79-R

1,3-Dihydro-2,2-dimethylperimidine [called 2.2-Dimethyl-2.3-dihydro-perimidin or N.N'-Isopropyliden-naphthylendiamin-(1.8) in Ger], $C_{13}H_{14}N_2$; mw 198.26, N 14.13%; ndls (from w), mp 117°, may be considered the parent compd of its tetranitroderiv
Ref: Beil **23**, 205 & (51)

1,3-Dihydro-2,2-dimethyl-4,6,7,9-tetranitroperimidine or 4,6,7,9-Tetranitro-dimethyl-dihydroperimidine,

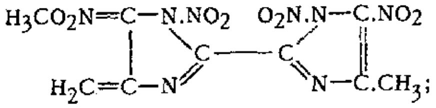


; mw 378.26, N 22.22%; orn-yel plates (from acet), mp 280° (dec); was obtd by tetrazotisation of the parent compd by nitrosylsulfuric-glacial acetic acid (Ref 2)
Refs: 1) Beil - not found 2) H.H. Hodgson & J.S. Whitehurst, JCS **1947**, 81 & CA **41**, 3452 (1947)

Dihydroimidazolone. Same as Imidazolidone

5,6-Dihydro-6-imino-2,4(1,3H)-s-triazinedione. See Ammelide in Vol 1, p A273-L

4,5-Dihydro-5-isonitro-4'-methyl-4-methylene-1,1',5'-trinitro-2,2'-biimidazole Methyl Ester or 4-Methyl-4'-methylene-1,1',5'-trinitro-2,2'-biimidazole-5'-nitronic Acid Methyl Ester,



mw 356.21, N 31.46%; lt-red crysts + H_2O, mp - 127° (dec), was obtd as one product when the Na salt of tetranitro-biimidazole was shaken with $(CH_3)_2SO_4$ at RT (Ref 2)
Refs: 1) Beil - not found 2) K. Lehmstedt, Ann **507**, 224-25 (1933) & CA **28**, 767 (1934)

1,2-Dihydro-1-methylnitro-2-nitroiminopyridine, (called 5-Nitro-2-methylnitramino-pyridin in Ger), $NC_5H_3(NO_2).N(CH_3).NO_2$; mw 198.14, N 28.28%; crysts (from w) or greenish plts (from petr eth), mp 59-60°; was obtd by nitrating 5-nitro-2-methylaminopyridine, or in addn to other products by the action of $(CH_3)_2SO_4$ on 5-nitro-2-nitraminopyridine in 10% NaOH or K_2CO_3 soln (Refs 1, 2 & 3)
Refs: 1) Beil **22**, [338] 2) A.E. Tschitschibabin (Chichibabin) & R.A. Konowalowa, Ber **58**, 1716 (1925) & CA **20**, 396 (1926) 3) A.E. Tschitschibabin (Chichibabin) & A.V. Kirssanow, Ber **61**, 1225 (1928) & CA **22**, 4125 (1928)

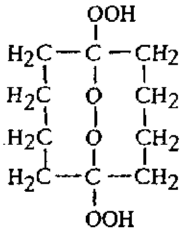
Dihydropentaborane. See Pentaborane, Unstable, under Boron Hydrides in Vol 2, p B254-L

Di(hydroperoxycyclohexyl)-peroxide or **Di(hydroperoxy)-dicyclohexyl-peroxide.** See 1,1'-Bis(hydroperoxycyclohexyl)-peroxide in Vol 2, p B144-L

1,1'-Dihydroperoxy-1,1'-dicyclohexylacetylene. See Vol 1, p A66-R under Acetylene Hydroperoxides and Peroxides

Di(hydroperoxydimethyl)-hexane. See 2,5-Bis(hydroperoxy-2,5-dimethyl)-hexane in Vol 2, p B144-R

1,6-Dihydroperoxy-(1,6-endoperoxy)-cyclodecane(?),



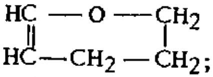
; mw 250.24, O 44.76%; ndls

(from benz), mp 130°(dec), explodes when heated in a flame; instantly liberates I_2 from KI and givès O when treated with $Pb(OAc)_4$; was obtd when the benz mother liquors from 1-hydroperoxy-1-methoxy-6-cyclodecanone were allowed to stand for several weeks (Ref 2)
Refs: 1) Beil - not found 2) R. Criegee & G. Wenner, Ann **564**, 14(1949) & CA **44**, 1942(1950)

9,9'-Di(hydroperoxy)-9,9'-fluorene. Same as 9,9'-Bis(benzoylperoxyfluorenyl)-peroxide in Vol 2, p B136-L

9,9'-Di(hydroperoxy)-9,9'-fluorene + 2 Fluorenones (Adduct). See 9,9'-Bis(hydroperoxy)-9,9'-fluorene + 2 Fluorenones (Adduct) in Vol 2, p B145-L. It is listed in CA's as *Fluorenone Compound with 9-Fluorenylidenedihydroperoxide*

2,3-Dihydro-4H-pyran or 1,5-Epoxy-pent-1-enes (called by Jones & Taylor),

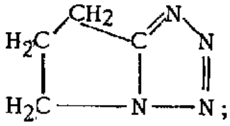


$$\begin{array}{ccccc} HC & - & O & - & CH_2 \\ \| & & & & | \\ HC & - & CH_2 & - & CH_2 \end{array}$$

; mw 84.11, O 19.02%; col mobile liq, having an ether-like odor, fr p -70°, bp 84.3°; flash p (Tag CC)° F, d 0.927 at 20°, n_D 1.4180 at 25°can be prepd by the catalytic dehydration of tetrahydrofurfuryl alcohol (Ref 3)

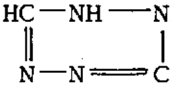
Fedoroff (Ref 2) detd that Dihydropyran completely gelatinizd NC of 11.95 & 12.65% N contents. Grades of NC of higher N content were only partially gelatinized
Refs: 1) Beil - not found 2) B.T. Fedoroff, Private communication, Letter (June 1949) 3) D.G. Jones & A.W.C. Taylor, QuarterlyRevs (London) **4**, 195, 204(1950)& CA **48**, 5172 (1954) 4) CondChemDict(1961), 384 5) Sax(1963), 724 (Toxicity & fire hazard)

6,7-Dihydro-5H-pyrrolotetrazole or Trimethylenetetrazole,



; mw 110.12, N 50.88%, col ndls (from xylene), mp 110°; previously obtd by an intramolecular cyclization of γ-azidobutyronitrile in chlf when treated with $ClSO_3H$ (Ref 4); was recently prepd by reacting 2-methylmercaptopyrroline & a chlf soln of Hydrazoic Acid (Ref 5). Châtel (Ref 2) showed that large doses of the compd could restore normal rhythm in the heart of dogs poisoned with chlf. Similar results were reported by Issekutz (Ref 3). The expl props of the compd were not detd
Refs: 1) Beil - not found 2) A. de Châtel, OrvosiHetilap **78**, 813(1934) & CA **29**, 1877 (1935) 3) B. Issekutz, ArchExptlPathPharmakol **177**, 415(1935) & CA **29**, 3721(1935) 4) V. Kereszty & E.Wolf, GerP 611692(1935) & CA **29**, 5495(1935) 5) J.H. Boyer & E.J. Miller Jr, JACS **81**, 4671, 4673(1959) & CA **54**, 3394(1960)

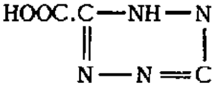
1,2-Dihydro-sym-tetrazine (called 1.2-Dihydro-1.2.4.5-tetrazin in Ger),



$$\begin{array}{ccccc} HC & - & NH & - & NH \\ \| & & & & | \\ N & - & N & = & CH \end{array}$$

; mw 84.08, N 66.64%; lt-yel prisms (from eth or benz), mp 125-26°(dec); readily sol in w & alc; sl sol in eth & benz; can be prepd by reduction of sym-tetrazine with H_2S in cold aq soln, or with Zn dust & glac acetic acid in ether; and from hydrazoformaldehyde-hydrazone by soln in dil glac acetic acid (Refs 1,2,3 & 4). Its expl props were not detd
Refs: 1) Beil **26**, 349 & [199] 2) T. Curtius et al, Ber **40**, 821, 828, 837(1915) 3) E. Müller & L. Herrdegen, JPraktChem **102**, 116, 123(1921) & CA **15**, 3849(1921) 4) V. vonRichter & R. Anschütz, "The Chemistry of the Carbon Compounds", ElsevierPubCo, NY, Vol **4**(1947), 324-25

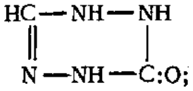
1,2-Dihydro-sym-tetrazine-3,6-dicarboxylic Acid [called 1.2-Dihydro-1.2.4.5-tetrazin-dicarbonsäure-(3.6); Bisdiazoessigsäure or "Triazoessigsäure" in Ger],



; mw 172.10, N 32.56%; yel plts + $2H_2O$ (from w) or anhyd yel prisms, mp 149-55° (dec), when heated on a Pt foil it burns with hissing as a proplnt; sol in acet & alc; insol in chlf, eth, CS_2, benz & cold w; can be prepd by heating 1,2-dihydro-sym-tetrazine-3,6-dicarboxylic acid diamide with NaOH and by other methods (Ref 1). When this compd was treated with HNO_2, some carmine and crysts, mildly expl, were formed. It forms numerous salts, some of which are unstable & expl when heated, Eg: *Silver salt*, $Ag_2C_4H_2N_4O_2$, lt-yel powd, explodes on heating; insol in w (Ref 2)

Refs: 1) Beil **26**, 567-68 2) T. Curtius & J. Lang, JPraktChem **38**, 531-58(1888); Ber **22**, Referate (1889), p 133 3) Chao-Han Lin et al, JACS **76**, 427(1954) & CA **49**, 3207(1955) [Describes some Methyl ester derivs, the prepn of 1,2-Dihydro-sym-tetrazine-3,6-dicarbonyl Hydrazide, mp 287-88° (dec); and their visible absorption spectra]

1,2-Dihydro-sym-tetrazine-3(4H)-one; or 4,5-Dihydro-sym-tetrazine-3(2H)-one,



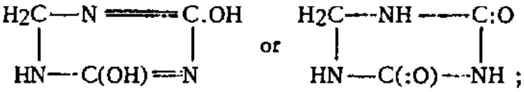
; mw 100.08, N 55.99%; crysts (from alc), mp 187-88°, bp 315°; was obtd when $(NH_2)_2.H_2O$ was heated 4 hrs at 175° under 900 atm of CO. This compd is reported to be useful as a synthetic resin intermediate, antishrinking agent for wool & rayon, and as a paper size (Ref 2)

Refs: 1) Beil - not found 2) H.J. Sampson, USP 2589290(1952) & CA **46**, 11234(1952)

Di[(4-hydrotetrazolyl)-5]-azine; Hydrazo-di (tetrazolyl-5); 4',4-Dihydro-5',5-azinotetrazole; Azino-di[(4-hydroxytetrazole)-5]; or (Ditetrazolyl-5)-hydrazine. See Bis(5-tetrazole)-hydrazine in Vol 2, p B157-R and in F.R. Benson, ChemRevs **41**, 8(1947)

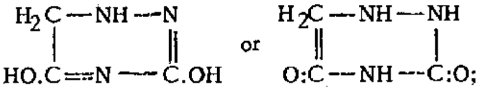
Dihydro-triazine-diol, $C_3H_5N_3O_2$; mw 115.09, N 36.51%. Two isomers are described in the literature:

5,6-Dihydro-s-triazine-2,4-diol (called 2.4-Dioxo-hexahydro-1.3.5-triazin in Ger)



; crysts (from formic acid), mp 245° (Refs 2 & 4)

1,6-Dihydro-as-1.2.4)-triazine-3,5-diol (called 3.5-Dioxo-hexahydro-1.2.4-triazin or 3.5-Dioxy-1.6-dihydro-1.2.4-triazin in Ger),

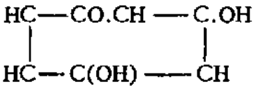


prismatic plates (from w), mp 221°; readily sol in w; diffc sol in alc; insol in common org solvs (Refs 1,3 & 5)

Refs: 1) Beil **26**, (63) 2) Beil **26**, [119] 3) J.R. Bailey & W.T. Read, JACS **36**, 1764 (1914) 4) H. Branderberger & R. Schwyzer Helv **38**, 1396(1955) & CA **50**, 5686(1956) 5) C.J. Grundmann et al, JOC **23**, 1522(1958) & CA **53**, 7193(1959)

Dihydroxy. A prefix indicating the presence of two hydroxyl groups, (OH), in an org compd. It corresponds to the German prefix "Dioxy"

2,4-Dihydroxyacetophenone [called 2.4-Dioxy-1-acetyl-benzol; 2.4-Dioxy-acetophenon; 4-Acetyl-resorcin; Resacetophenon or Methyl-(2.4-dioxyphenyl)-keton in Ger],



; mw 152.14, O 31.55%; pltls or ndls, mp 141-47°; prepn & other props are found in Beil (Ref 1). Its *5-Nitro-*, mp

142°; *3,5-Dinitro-*, mp 166-67°; and *2,4,6-Trinitro-* derivs were prepd by Jadhov & Merchant (Ref 2)

Refs: 1) Beil **8**, 266, (613) & [294] 2) G.V. Jadhav & J.R. Merchant, JIndianChemSoc **28**, 265 (1951); JUnivBombay,Sect A, **19**, Part 5 (Science No 29), 45 (1951) & CA **47**, 6375; 6376 (1953)

DIHYDROXYANTHRAQUINONE AND DERIVATIVES

Dihydroxyanthraquinone (called Dioxyanthrachinon in Ger), $HO.C_6H_3(CO)_2C_6H_3.OH$; mw 240.10, O 26.64%. The more commonly known Dihydroxyanthraquinones are described in Beil and listed in K & O (Ref 10)

1,2-Dihydroxy- or Alizarin (Ref 1)
1,3-Dihydroxy-; Purpuroxanthin or Xanthopurpurin (Ref 2)
1,4-Dihydroxy- or Quinizarin (Ref 3)
1,5-Dihydroxy- or Anthrarufin (Ref 4)
1,6-Dihydroxyanthraquinone (Ref 5)
1,7-Dihydroxyanthraquinone (Ref 5)
1,8-Dihydroxy- or Chrysazin (Ref 6)
2,3-Dihydroxy- or Hystazarin (Ref 7)
2,6-Dihydroxy- or Anthraflavin (Ref 8)
2,7-Dihydroxy- or Isoanthraflavin (Ref 9)

The 1,4- deriv is the most important of the Dihydroxyanthraquinones, many of which have trival names indicative of their derivation or color of dye which they produce. They are high melting solids, varying in color from yellow thru orange and red and some brown. They are practically insol in water; glacial acet ac, pyridine & higher-boiling aromatic solvs are used to crystallize them; they dissolve in alkali & concd H_2SO_4. Addn of boric acid to their sulfuric acid solns gives rise to distinctive color changes which are helpful in characterizing the various hydroxyanthraquinones (Ref 10). Other props and methods of prepn are given in Beil (Refs 1-9)

Refs: 1) Beil **8**, 439, (710) & [487] 2) Beil **8**, 448, (714) & [492] 3) Beil **8**, 450, (714) & [492] 4) Beil **8**, 453, (719) & [496] 5) Beil **8**, 457 & (721) 6) Beil **8**, 458, (722) & [500] 7) Beil **8**, 462, (723) & [504] 8) Beil **8**, 463, (723) & [504] 9) Beil **8**, 466, (724) & [505] 10) Kirk & Othmer, 2nd edit **2** (1963), pp 465ff by A.J. Cofrancesco

Nitro Derivatives of Dihydroxyanthraquinone

Mononitrodihydroxyanthraquinone, $O_2N.C_6H_3(CO)_2C_6H_2(OH)_2$; mw 285.20, N 4.91%. The following isomers are found in Beil:

3-Nitro-1,2-dihydroxy- or 3-Nitro-alizarin, long orn-yel ndls (from benz) or yel plts (from glac acet ac, alc or chlf), mp 244° (dec) (Ref 1)
4-Nitro-1,2-dihydroxyanthraquinone, golden-yel ndls (from alc or glac acet ac), mp 289° (dec) (Ref 2)
2-Nitro-1,4-dihydroxy- or 2-Nitro-quinizarin, red ndls (from nitrobenz or glac acet ac), mp - not reported (Ref 3)
5-Nitro-1,4-dihydroxyanthraquinone, red crysts (from glac acet ac), mp 244-45° (Ref 4)
4-Nitro-1,8-dihydroxy- or 4-Nitro-chrysazin, orn-yel crysts (from chlorobenz), mp 232-34° (Ref 5)
1-Nitro-2,3-dihydroxy- or 1-Nitro-hystazarin, yel crysts (from toluene), mp - not reported (Ref 6)

Other props & methods of prepn are given in Beil

Refs: 1) Beil **8**, 447, (713) & [491] 2) Beil **8**, 447 & [491] 3) Beil **8**, (716) 4) Beil **8**, 453 5) Beil **8**, 460 6) Beil **8**, 463

Dinitrodihydroxyanthraquinone, $HO.C_6H_2(NO_2)(CO_2)C_6H_2(NO_2).OH$; mw 330.20, N 8.48%. Five isomers are found in Beil:

X,X-Dinitro-1,3-dihydroxyanthraquinone, lt-yel ndls (from glac acet ac), mp 249-50° (Ref 1)
4,8-Dinitro-1,5-dihydroxyanthraquinone, yel cryst ppt, mp - not reported (Ref 2)
4,5-Dinitro-1,8-dihydroxyanthraquinone, lt-yel cryst flakes, mp - not reported (Ref 3)
1,4-Dinitro-2,3-dihydroxyanthraquinone, $C_6H_4(CO)_2C_6(NO_2)_2(OH)_2$; crysts (from alc), mp - not reported (Ref 4)
1,5-Dinitro-2,6-dihydroxyanthraquinone, yel ndls (from alc), mp >300° (Ref 5)

Other props & methods of prepn are found in Refs

Refs: 1) Beil **8**, 449 2) Beil **8**, 456 3) Beil **8**, 460 4) Beil **8**, 463 5) Beil **8**, 465 & [505]

Trinitrodihydroxyanthraquinone, $C_{14}H_5N_3O_{10}$, not found

Tetranitrodihydroxyanthraquinone, $C_{14}H_4N_4O_{12}$; mw 420.20, N 13.33%. The following isomers are described in the literature:

2,4,6,8-Tetranitro-1,5-dihydroxyanthraquinone or 2,4,6,8-Tetranitro-anthrarufin, $HO.C_6H(NO_2)_2(CO)_2C_6H(NO_2)_2.OH$; yel plates (from fuming HNO_3), mp - not reported; forms colored metallic salts (Refs 1 & 7)

2,4,6,8-Tetranitro-1,7-dihydroxyanthraquinone, structure as above, crysts, mp - not reported (Ref 2)

2,4,5,7-Tetranitro-1,8-dihydroxyanthraquinone; 2,4,5,7-Tetranitro-chrysazin or Chrysammic Acid; structure as above, golden yel shiny plates (from the aq soln of Ca salt + HNO_3) or yel plates (from fuming HNO_3), mp - deflgr with a flash on heating, bp - explodes on rapid heating; sol in hot w, alc & eth; forms salts, some of which are expl (Ref 3)

Barium salt, $BaC_{14}H_2N_4O_{12}$, brick-red crysts, explodes on heating; nearly insol in alc & water

Calcium salt, $CaC_{14}H_2N_4O_{12}$, golden ndls, explodes on heating; sol in alc

Copper salt, $CuC_{14}H_2N_4O_{12} + 4H_2O$, dk-red crysts, loses water on heating and then explodes

Lead salt, $PbC_{14}H_2N_4O_{12} + 5H_2O$, brick-red crysts, loses water on heating and then explodes

Potassium salt, $K_2C_{14}H_2N_4O_{12}$, greenish shiny crysts, explodes on heating; diffc sol in water

Silver salt, $Ag_2C_{14}H_2N_4O_{12}$, dk-brn crysts, explodes on heating

Sodium salt, $Na_2C_{14}H_2N_4O_{12} + 3H_2O$, possesses same props as K salt

1,3,5,7-Tetranitro-2,6-dihydroxyanthraquinone, structure same as 2,4,6,8-Tetranitro deriv, yel ndls, mp - explodes on heating to 308° w/o melting; sol in alc & eth; sl sol in water (Ref 4)

X,X,X,X-Tetranitro-2,7-dihydroxyanthraquinone, $C_{14}H_2O_2(NO_2)_4(OH)_2$; yel shiny plates (from dil HNO_3), mp >300°; almost insol in benz; sl sol in eth; v sl sol in glac acet ac & HNO_3; sol in alkali; forms salts, some of which are probably expl (Ref 5)

X,X,X,X-Tetranitro-X,X-dihydroxyanthraquinone, yel crysts, mp >300°; sol in benz; sl sol in alc (Ref 6)

Methods of prepn & other props are given in Beil

Refs: 1) Beil **8**, 457 & (720) 2) Beil **8**, (721) 3) Beil **8**, 461 & (723) 4) Beil **8**, 465, (724) & [505] 5) Beil **8**, 467 6) Beil **8**,, [505] 7) H. Hoyer, ZElektrochem **60**, 381 (1956) & CA **50**, 11823(1956) (Infrared spectrum)

Dihydroxyazobenzene and Derivatives. See under Azophenol (Azodiphenol) in Vol 1, pp A657-A658. Its nitrated derivative:

Hexanitrodihydroxyazobenzene was examined during WWII from the point of view of its utilization as a military expl, but was found to be rather expensive to prepare

Ref: A.H. Blatt & F.C. Whitmore, "A Literature Survey of Explosives", OSRD Rept **1085** (1942)

p-Dihydroxyazobenzene Dichromate, $[HO.C_6H_4.N]_2.Cr_2O_7$; N 12.23%; yel crysts; mp - expl ca 134° was prepd by Meldola & Eynon (Ref 2) on diazotizing p-aminophenol with Na nitrite in sulfuric acid, followed by treatment with Na dichromate

Refs: 1) Beil - not found 2) R. Meldola & L. Eynon, JCS **87**, 2-3(1905)

Dihydroxyazoxybenzene. See Azoxyphenol in Vol 1, p A671-L

DIHYDROXYBENZALDEHYDE AND DERIVATIVES

Dihydroxybenzaldehyde (called Dioxy-benzaldehyd in Ger), $(HO)_2C_6H_3.CHO$; my 138.12, O 34.75%. The following isomers are found in Beil: *2,3-Dihydroxy-*, sulfur-yel ndls (from benz), mp 102-08°, bp 235° at 754 mm press (dec) (Ref 1); *2,4-Dyhydroxy-*, almost col ndls (from eth + petr eth), or yel ndls (from w), mp 134-36° (Ref 2); *2,5-Dihydroxy-*, yel ndls (from hot benz), mp 98-99° (Ref 3); *3,4-Dihydroxy-*, flat crysts (from water), mp 153-54° (dec) (Ref 4); and *3,5-Dihydroxybenzaldehyde,* ndls (from eth acet + petr eth), mp 145-46° (Ref 5). Other props & methods of prepn are given in the Refs

Refs: 1) Beil **8**, 240,(600) & [267] 2) Beil **8**, 241, (603) & [272] 3) Beil **8**, 244 & [276] 4) Beil **8**, 246, (604) & [277] 5) Beil **8**, [291]

Nitro Derivatives of Dihydroxybenzaldehyde

Mononitrodihydroxybenzaldehyde, $(HO)_2C_6H_2(NO_2).CHO$; mw 183.12, N 7.65%. Some of the possible isomers are found in the literature: *5-Nitro-2,4-dihydroxy-*, yel-brn prisms (from benz), mp 146-49° (Ref 1 & 5); *2-Nitro-3,4-dihydroxy-*, yel ndls (from eth + petr eth), mp 176° (Ref 2); *5-Nitro-3,4-dihydroxy-*, yel ndls (from toluene or water), mp 106° (Ref 2); and *6-Nitro-3,4-dihydroxy-benzaldehyde*, crysts, mp 203-04° (dec) (Refs 3,4 & 6)

Refs: 1) Beil **8**, 244 2) Beil **8**, 261 3) A.H. Parijs, Thesis, Leiden **1928** & CA **23**, 4204 (1929) 4) Ibid, Rec **49**, 17 (1930) & CA **24**, 2446 (1930) 5) R.D. Desai et al, ProcIndianAcadSci **23A**, 338 (1946) & CA **40**, 6444 (1946) 6) R.J.S. Beer et al, JCS **1948**, 2223 & CA **43**, 3405 (1949)

3,5-Dinitro-2,4-dihydroxybenzaldehyde, $(HO)_2C_6H(NO_2)_2.CHO$; mw 228.12, N 12.28%; yel crysts (from alc), mp 170°; was obtd by nitrating the 2,4-dihydroxy deriv with fuming HNO_3

Ref: Beil **8**, 244

NOTE: No higher nitrated derivs of Dihydroxybenzaldehyde were found in Beil or in CA thru 1961

3,3'-Dihydroxybenzaldehyde-azine [called 3.3'-Dioxy-benzaldazin or Bis-(3-oxybenzal)-hydrazin in Ger], $HO.C_6H_4.CH{:}N.N{:}CH.C_6H_4.OH$; mw 240.25, N 11.66%; yel ndls (from alc), mp 204-05°; sl sol in eth & alkali; v sl sol in alc, benz & chlf; mod sol in hot w; insol in petr eth & w (Ref 1)

Its nitrated deriv **2,4,6,2',4',6'-Hexanitro-3,3'-Dihydroxybenzaldehyde-azine,** $HO.C_6H(NO_2)_3{\cdot}CH{:}N.N{:}CH.C_6H(NO_2)_3.OH$; mw 510.25, N 21.96%; lt-yel ndls, mp - explodes violently at 150-60°; sol in alc, 50% glac acet ac & alkali; insol in benz & chlf; was obtd by reacting hydrazine sulfate with 2,4,6-trinitro-3-hydroxybenzaldehyde in hot aq NaOH (Refs 2 & 3)

Refs: 1) Beil **8**, (526) 2) Beil **8**, [63] 3) H.H. Hodgson & H.G. Beard, JCS **1927**, 2379 & CA **22**, 64 (1928)

DIHYDROXYBENZENE AND DERIVATIVES

Dihydroxybenzene, $HO.C_6H_4.OH$; mw 110.11, O 29.06%. The three possible isomers are found in Beil:

1,2-Dihydroxybenzene; **Pyrocatechol** *or 1,2-Benzenediol* (called 1,2-Dioxy-benzol or Brenzcatechin in Ger), monoclinic crysts (from benz, water, petr eth or eth), mp 104-05°, bp 245° (sublimes); contact with skin & inhalation of dust should be avoided (Refs 1 & 5)

1,3-Dihydroxybenzene, **Resorcinol** *or 1,3-Benzenediol* (called 1.3-Dioxy-benzol or Resorcin in Ger), white orthorhombic crysts (from water, alc or acet), mp 110.7°, bp 281°; d 1.2717; flash point (open cup) 339°F; sol in w, alc, ether, glycerol, benzene and amyl alc; sl sol in chlf; less toxic than the 1,2-deriv but contact with skin and inhalation of dust should be avoided

Can be prepd by fusing benzene-meta-disulfonic acid with NaOH, and dissolving the melt in water. After acidifying the soln with HCl, the resorcinol is extracted with a volatile solvent and the solvent evaporated. Purification can be achieved by distillation

Uses: Besides its uses as a reagent in analytical chemistry, as substitute for camphor in celluloid, it serves as a parent material for manuf of mono-, di- and trinitro derivs used in the form of lead salts in priming expls

1,4-Dihydroxybenzene; **Hydroquinone** *or 1,4-Benzenediol* (called 1.4-Dioxy-benzol or Hydrochinon in Ger), wh trigonal ndls or prisms (from w), mp 166°, bp 285° at 730 mm press; oral ingestion causes irritation of intestinal tract, dermatitis can result from skin contact, and its vapors cause eye irritation (Refs 3 & 4)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **6**, 759, (378), [764] & {4187} 2) Beil **6**, 796, (398), [802] & {4292} 3) Beil **6**, 836, (413), [832] & {4374} 4) Kirk & Othmer **7** (1951), 755-62; 2nd edit **11** (1966), 483-92 5) Kirk & Othmer **11** (1953), 307-14; 2nd edit **11** (1966), 462-72 6) Kirk & Othmer **11** (1953), 711-20; 2nd edit **11** (1966), 472-83 6a) Sax (1963), p 889 (p-Hydroquinone), 1146 (Pyrocatechol), and 1156 (Resorcinol) 7) Urbański **1** (1964), 535-36

NITRODERIVATIVES OF DIHYDROXYBENZENE

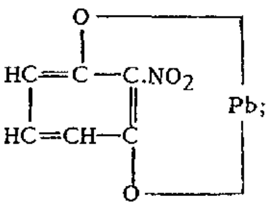
Mononitrodihydroxybenzene, $HO.C_6H_3(NO_2).OH$; mw 155.11, N 9.03%. All possible isomers are found in Beil:

3-Nitro-1,2-dihydroxybenzene, yel ndls (from petr eth), mp 86° (Ref 1)

4-Nitro-1,2-dihydroxybenzene, yel ndls (from eth + petr eth, benz or w), mp 174-76° (dec) (Ref 2)

2-Nitro-1,3-dihydroxybenzene or 2-Nitroresorcinol, orn prisms (from dil alc), mp 84-88°, bp 232-34°, d 1.3526 at 96.7° & 1.2531 at 180.5°; sol diagram with water from 20 to 140° is reported; forms yel solns with acids; forms a eutectic with Styphnic Acid; can be prepd by heating resorcinol & fuming H_2SO_4 at 100° for 30 mins, cooling to 25°, adding mixed acids & allowing to stand for 30 mins, addg water and distilling with steam (Ref 3)

Its **Lead Salt,** known as **Lead 2-Mononitroresorcinate (LMNR),**



mw 360.30, N 3.89%, NO_2 group 12.77%, Pb 57.51%; reddish-brn microscopic crysts; has slow burning props & low combustion temp; can be prepd by mononitration of resorcinol and allowing the slurry of MNR to react with $Pb(OH)_2$ at elevated temp

Uses. LMNR was patented by Jones & Moore (Ref 7) for use in the flashing of electric fuseheads. Foulon (Ref 8) suggested its use in other initiating materials. Odierno (Ref 17) reported that LMNR has been used in the "spot" (fusehead) of Electric Detonator M60 together with 70 mg of DLA (dextrinated Lead Azide) as a priming chge & 75 mg of PETN as a base chge. It has also been used as an upper chge (21 mg) in the same detonator. Accdg to Ref 16, LMNR can be used in other detonators, fuzes and in priming compositions. See also Refs 9, 10. 11, 12, 14 & 15

US requirements and tests for LMNR intended for use in detonators, fuzes, and priming compositions are given in US Specification MIL-L-46496 (Ord) (1961) and Amendment 1 (1962). LMNR is delivered wet with not less than 20% of dilute denatured alc (50% in w), placed in bags of 4-ounce (or heavier) duck, holding ca 25 lbs of material, on the dry basis

Requirements:

1) *Color and Appearance.* LMNR shall be lt brn in color and free from foreign material

2) *Apparent Density* - 0.20 to 0.35 g/ml

3) *Acidity, pH* - 5.0 to 6.5

4) *Lead Content* - 70.5 to 72.5%

5) *Nitro Group Content*- 7.2 to 7.6%

6) *Vacuum Stability at 120°* - 0.4 ml/40 hrs

Sampling. Use the same procedure as described under Normal LSt

Preparation of Dry Sample. Use the same procedure as described under Normal LSt

Tests:

1) *Color and Appearance.* Transfer ca 10 g of the wet sample as received, to a Büchner funnel and apply suction until the sample is almost dry. Spread the material on a large watch glass and place in an oven at 70-75° for at least 4 hrs or until constant wt is obtd. Take four 0.5-mg portions, each from different sections of the sample and spread them over an area of 2 sq cm on a glass microscopic slide. Examine the material using a magnification of ca 30 times

2) *Apparent Density.* Weigh into a 50-ml beaker, 2.00±0.01 g of dry sample, carefully add 95% ethyl alc until a thick paste is formed on mixing with a plastic spatula. Using the same spatula (or rod), followed by a stream of alc, transfer the paste to a 25-ml graduate having an ID of 20 mm. Cover the graduate with a rubber stopper and shake thoroughly until the sample is completely wetted. After shaking the graduate, allow it to stand for 2 hrs away from vibrations and jarring. Read the settled volume and calc the apparent density by dividing the wt of sample in grams by observed volume in mls

3) *Acidity.* Transfer 1.00 g of dry sample to a 150-ml beaker and add 100 ml of freshly boiled & cooled distd w having a pH of 6.0±0.5. Allow to stand, with occasional stirring, for 15 mins and filter it into a smaller beaker. Using a pH Meter, equipped with a glass electrode and a saturated calomel electrode,

which reads directly in pH units and is capable of measuring accurately within 0.1 pH unit, determine pH of filtrate at 25±2°. The apparatus shall be calibrated with standard buffer soln

Note 1: A Beckman Industrial Model M pH Meter (Fisher Scientific Co Catalogue 90, p 577) is satisfactory for detn of pH

Note 2: Prepare stock and buffer solns having the pH values listed below in the following manner:

Stock Solutions:

Solution A is a 0.1 molar soln of potassium dihydrogen phosphate, prepd by dissolving 13.61 g of ACS grade material in distd w and diluting to 1 liter

Solution B is a 0.05 molar soln of sodium borate (borax) prepd by dissolving 19.10 g of ACS grade material in distd w and diluting to 1 liter

Solution C is a 0.05 molar succinic acid soln, prepd by dissolving 5.9 g of material (previously purified by recrystallization from distd w and dried in a desiccator over calcium chloride) in distd w and diluting to 1 liter

Buffer Solutions. Prepare buffer solns of the following pH's at 25°C by mixing the stock solutions in the following proportions in milliliters:

pH Soln	A	B	C
4.0	-	1.78	8.22
5.0	-	3.68	6.32
6.0	8.77	1.23	-
7.0	6.23	3.77	-
8.0	4.65	5.35	-
9.0	1.75	8.25	-

4) *Lead Content.* Place in a 300-ml tall form beaker a 0.5 g accurately weighed dry sample and wet it with ca 5 ml distd w. Add 20 ml of dilute sulfuric acid & 5 ml of concd nitric acid, cover the beaker with a ribbed watch glass and place on a hot plate turned on a low heat. Increase the heat gradually during the evaporation in order to keep the soln fuming. After evaporating to ca half volume, cool the beaker and add 10 ml of concd nitric acid. Reheat it until the liquid becomes clear and dense white fumes are given off. After the 2nd evaporation, cool the beaker with contents and rinse down the watch glass and side of beaker until the vol in the beaker is increased to ca 100 ml. After allowing to stand for 2 hrs filter off the ppt of Pb sulfate thru a sintered-glass filtering crucible. Use a stream of 95% alc (ca 50 ml) for transferring the ppt from the beaker to the crucible and three 50-ml portions of alc for rinsing the ppt on the filter. Dry the crucible with ppt for 1 hr at 100°, cool in a desiccator and weigh

$$\text{Percent of Lead} = \frac{68.32A}{W},$$

where: A = wt of ppt; and
W = wt of dry sample

5) *Nitro Group Content by Titanous Chloride Method.* Transfer ca 0.2 g of accurately weighed dry sample into a titration flask, add 25 ml of 95% alc and a few glass beads. Allow carbon dioxide, generated in a Kipp's apparatus, to pass thru the flask for ca 10 min in order to displace the air and continue the flow of CO_2 throughout the test. Add 50 ml of 0.2N titanous chloride soln from the buret, install a vertical (reflux) water-cooled, condenser in the neck of the flask and place the flask on a hot plate. After boiling gently for ca 5 mins, increase the flow of CO_2 and place the flask in a cold w bath to cool. Disconnect the condenser and titrate the soln with 0.15N ferric ammonium sulfate until the disappearance of dark color of the titanium. Add 10 ml of Amm thiocyanate indicator soln and continue the titration with ferric ammonium sulfate soln until a reddish color, which remains for 1 min is obtd. Conduct a blank detn using the same reagents as above minus the sample

$$\text{Percent of } NO_2 \text{ Group} = \frac{0.7668(A-B)N}{W}.$$

where: A = ml of ferric ammonium sulfate soln used for blank;
B = ml of ferric ammonium sulfate soln used for sample;
N = normality of ferric ammonium sulfate soln; and
W = wt of dry sample

Note: Nitro group can also be detd by Titanous Sulfate Method, which is offered as an alternate method on pp 9-11 of Spec MIL-L-46496. In this method the soln of titanous sulfate is standardized against p-nitraniline

6) *120° C Vacuum Stability Test.* Using a 2.3 g dry sample and adjusting the apparatus described in Vol 1 of Encycl, p XXVI to 120°, determine the amt of gas evolved in 40 hrs of heating. More detailed description of the test is given in MIL-STD-286A (1961). Method 403.1.2 and in PATR **3278** (1965), pp 19-22 & Fig 15

4-Nitro-1,3-dihydroxybenzene, lemon yel ndls + ½H_2O (from water), anhyd crysts (from chlf or CCl_4), mp 120-22° (Ref 4)

5-Nitro-1,3-dihydroxybenzene, crysts (from w), mp 157-59° (Ref 5)

2-Nitro-1,4-dihydroxybenzene, dk-red crysts (from w), mp 132-34° (Ref 6)

Other props & methods of prepn are given in Beil

Refs: 1) Beil **6**, 787, [789] & {4263} 2) Beil **6**, 788, (391), [790] & {4263} 3) Beil **6**, 823, (404), [822] & {4343} 4) Beil **6**, 823, (404), [822] & {4344} 5) Beil **6**, 825 & {4347} 6) Beil **6**, 856, (418), [848] & {4442} 7) D.T. Jones & E.F. Moore, BritP 312952 (1928) & CA **24**, 967 (1930) 8) A. Foulon, SS **27**, 361 (1932) 9) ICI, Ltd, FrP 781646 (1935) & CA **29**, 6430 (1935) (Describes a compn which is fired directly by an elec current and is comprised of LMNR 30, finely divided Zr 70 parts, and a sufficient amt of a 5% soln of NC in amyl acetate to make a creamy mixt) 10) E.B. Kerone & C.C. Carroll, USP 2177657 (1939) & CA **34**, 1176 (1940) [Use of LMNR with $Ba(NO_3)_2$ & LA or MF in priming compns] 11) K.S. Warren, PATR **1448** (1944) 12) S. Kinoshita & T. Sakamati, JapP 2498 (1958) & CA **48**, 6700 (1954) (Compns for elec detonators consist of Sb or Cu powder 80, LMNR 10 & $KClO_3$ 10%, or Cu powder 90 & LMNR 10%, both compns made with binding agents) 13) S. Kinoshita, Kokayoshi **17**, 202 (1956) & **18**, 135 (1957) 14) K. Okazaki, Kôgyô-KayakuKyôkaishi **23** (4), 177-85 (1962) & CA **60**, 1528 (1964) [PA Translation **139** (Nov 1968); Priming characteristics of powdered ignition chges] 15) W. G. Finnegan et al, USP 3055780 (1962) & CA **58**, 407 (1963) [Highly effective initiation compn for bridge wires is formed from LMNR 98 & poly(2-methyl-5-vinyl-tetrazole) 2%, a water, insol stable binder] 16) US Specification MIL-L-46496 (Ord) (1961) & Amendment 1 (1962) 17) S.J. Odierno, "Information Pertaining to Fuzes", PicArsn, Dover, NJ, Vol IV (1964) pp IVa & Va

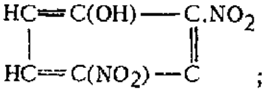
DINITRODERIVATIVES OF DIHYDROXYBENZENE

Dinitrodihydroxybenzene, $HO.C_6H_2(NO_2)_2.OH$; mw 200.11, N 14.00%. Five derivs are found in Beil:

3,5-Dinitro-1,2-dihydroxybenzene, yel ndls (from alc), mp 164° (Ref 1)

4,5-Dinitro-1,2-dihydroxybenzene, hygr yel crysts (from nitrobenz + benz), mp 166-69° (dec); crysts + H_2O, mp 80°; sol in several org solvs, water & alkali. Its **Disodium salt,** $Na_2C_6H_2N_2O_6$, steel-blue crysts, explodes on heating (Ref 2)

2,4-Dinitro-1,3-dihydroxybenzene or 2,4-Dinitroresorcinol (2,4-DNR)



$$\begin{array}{l} HC{=}C(OH){-}C.NO_2 \\ | \qquad\qquad\quad \| \\ HC{=}C(NO_2){-}C \end{array}$$

; yel plates (from w), mp 147-48°; deflagrates on rapid heating. Was first described in 1881 edition of Beilstein (Beil VII, p 885) and then in 1883 by P. Typke in Ber **16**, p 551 (See Ref 3)

Accdg to Urbański (Ref 6, p 537), 2,4-DNR cannot be prepd by the conventional method for phenols (ie sulfonation of resorcinol followed by the action of nitric acid), because this method yields the trinitro compd. There are, however, two methods of prepn possible: 1) nitrosation of resorcinol, followed by oxidation of dinitrosoresorcinol or 2) nitration of resorcilic acid, followed by decarboxylation

For nitrosation, a weak (2.75%) aqueous soln of 1 mole resorcinol is acidified with 2 moles of sulfuric acid. The soln is cooled to ca -2°, and a 10% aqueous soln of Na nitrite is added dropwise. The pptd yellowish crysts are washed with w and separated on a sintered glass filtering crucible, applying a vacuum suction. The separation may also be conducted in a centrifuge

K.S. Warren stated in PATR **1328** (1943) that he nitrosated the purified resorcinol, mp

107-09°, prepd by Eastman Kodak Co, by using the method of W.R. Orndorff & M.L. Nichols, described in JACS **45**, 1536(1923), except that he used dilute sulfuric acid instead of acetic acid. He stated that average yields of 68% crude DNR were obtd by this method in which resorcin was nitrosated as described above, and this product dried and subsequently oxidized with 48% nitric acid at ca -5°. Urbanski describes the method of oxidation of moist dinitroresorcinol using nitric acid of 50% strength at -5°, but his yields were only 30% of the theoretical and mp of resulting product was 147°

Since yields obtd by Warren were claimed to be 68%, we are describing here his method, which does not seem to differ much from that described by Urbański (Ref 6, p 538)

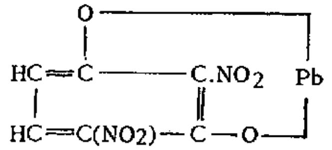
After removing moisture from dinitrosoresorcinol by washing with alc, followed by ether and suction of air thru the product in a sintered glass crucible, the following procedure was used: The desired wt of 48% nitric acid (the amt is not stated), was cooled in a 250-ml beaker placed in an ice-salt bath. When the desired temp of ca -5° was reached, 30 g of pulverized dinitrosoresorcinol was added in 0.5 g portions over the space of 10 mins. At the end of this period, the mixture was rapidly transferred to a suction sintered glass filtering crucible and washed immediately with several 20-ml portions of cold w to remove most of the residual acid. After air-drying, the product had mp 147-48°. In order to determine its impurities, the product was extracted with four 75-ml portions of ether and the combined ether extracts were washed with w and evaporated to dryness for subsequent examination

In the method described by Urbanski, the oxidized product was drowned, together with acid, in a large volume of ice water and the resulting mixture transferred to a vacuum filter

Warren also examined methods of prepn of DNR utilizing as starting materials 2,4-Dinitrophenol; 2,4-Dinitroaniline or meta-dichlorobenzene, but found them unsuitable on accnt of very low yields. The same applies to the methods of oxidation of dinitrosoresorcinol by agents other than nitric acid, such as electrolytic oxidation or hydrogen peroxide oxidation

2,4-Dinitroresorcinol forms several salts, some of them explosive, as for example: *Potassium 2,4-Dinitroresorcinate*, when obtd in anhydrous state, $KC_6H_3N_2O_6$, dk-red crysts, explodes on heating, while its monohydrate, yel ndls (from w) is not explosive

Lead 2,4-Dinitroresorcinate, abbreviated as **LDNR**



; mw 405.30, N 6.91%. It exists in two forms, yel & orn differing in physical characteristics, but possessing, accdg to K.S. Warren, similar expl props; crystal d 3.2, loading d 2.23; insol in w, alc, eth, acet, benz, carbon tetrachloride and ethylene dichloride.

Accdg to Urbanski (Ref 7, p 221), L DNR ignites readily from a direct flame and burns with great velocity. Its initiating action is weaker than that of LSt and it is less sensitive to impact and friction than Pb styphnate. It has therefore recently found application as a component of cap compns. It is valuable because it is safer to handle than LSt Accdg to K.S. Warren, PATR **1328**(1943), LDNR had been studied at PicArsn in 1934 and found to have special interest as a constituent of primer compns. It was prepd by J.D. Hopper, PATR **480**(1934) by adding slowly, with constant stirring, a soln of 10 g Pb nitrate in 60 ml of boiling w to a soln of 5 g purified 2,4-DNR and 2.65 g of anhydrous Na carbonate in 500 ml of boiling w. Carbonate was added to form the soluble Na dinitroresorcinate which, in turn, reacted with Pb nitrate to form the insoluble PbDNR. Stirring was continued for ca 1 hr while the mixture was cooled to RT. The resulting ppt of LDNR was filtered, and washed thoroughly first with w, then with alc and finally with eth. It was dried in a steam oven (Ref 8, p 189)

Properties of LDNR were examined at PicArsn by A.J. Phillips PATR **1079**(1941), Warren and others. Most of these properties are listed in Ref 8, pp 187-89: *Brisance* - 20 g of sand crushed by 0.4 g sample ignited by BkPdr fuse (Phillips gave for Western Cartridge Co sample 3.0 g); *Explosion Temperature* - 265°/5sec (Phillips gave 201°/5sec); *Heat of Explosion* - 270 cal/g; *100° Heat Test* - % loss of wt in 1st 48 hrs 0.20, in 2nd 48 hrs 0.02 and no expln after 100 hrs; *Hygroscopicity* at 30° & 90% RH - 0.73%; *Impact Sensitivity*, BM Apparatus, 1 kg wt 30 cm with 20 mg sample (Phillips gave 68 cm with 2 kg wt); *Initiating Efficiency* - 0.4 g of LNDR did not initiate Tetryl pressed at 3000 psi; *Minimum Detonating Charge of MF* - 0.24 g; *Sensitivity to Flame* - deflagrates; *Sensitivity to Spit of BkPdr Fuse* - deflagrates; *Vacuum Stability at 120°* - exploded in 73 mins

Uses: As an initiating expl, principally in electric detonators (Ref 8, pp 187-89). See also Ref 5a

Phillips examined the following mixture: MF 42, Ba & K nitrate 33, Sb sulfide 20 & LDNR 5%, used by the Western Cartridge Co in the modified No 3 Battery Cup Primer for Aircraft Signals and M-9 Flares and found it to be more satisfactory than previously used mixtures MF 44.5, K chlorate 28.5 & Sb sulfide 27.0%

There is no US Military Specification for LDNR

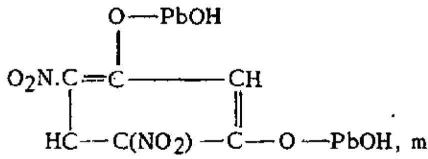
4,6-Dinitro-1,3-dihydroxybenzene, or 4,6-Dinitroresorcin (4,6-DNR), yel prisms (from eth acetate), mp 212-15°. Was first prepd in 1882 by Weselsky, Benedikt & Hübl and in 1883 by Typke (Ber **16**, 551), who hydrolyzed the nitration product of resorcin diacetate. A more direct and economical method suitable for production scale manuf was developed during WWII by the British. It consisted of direct nitration of granulated resorcin. A detailed description of laboratory method of prepn, given in Ref 8, p 192, is as follows:

Fuse ca 100 g of pure resorcin in a porcelain casserole and immediately pour on a Pyrex glass plate. Cool and grind the cake in a mortar to pass a US Standard No 6 mesh screen. Pour 400 g of 98% nitric acid into a pint size Dewar jar and add, in small portions, while mechanically stirring, snow of carbon dioxide, until the temperature drops to -20° C. Add, in small portions and with constant stirring, 40 g of granulated resorcin, while maintaining the temp between -20 and -15° with the aid of occasional addn of carbon dioxide snow. Five mins after the last portion of resorcinol is introduced, cool the mixture to -50° and then drown it, with vigorous stirring into 5 times its volume of cracked ice, in water. After allowing to stand for 1 hour, decant the bulk of supernatant liquid and filter the slurry. Wash the crude 4,6-DNR with w, and partially dry it. Purify it by first dissolving the DNR in an aq 5% NaOH soln (17.4 g NaOH in 340 ml w), and then neutralizing the soln by gradually adding it to a boiling soln of 21.4 g of 98% sulfuric acid in 150 ml of w. Filter the resulting ppt of purified 4,6-DNR on a suction filter, rinse with w and air-dry. The yield is ca 27.5 g (37.8% of the theoretical) (Ref 8, p 192)

See also K.S. Warren PATR **1448**(1944), pp 8-9 and Ref 9

Its **Barium Salts** are: $Ba(C_6H_3N_2O_3)_2$, yel ndls, and $BaC_6H_2N_2O_6$, carmine-red flakes having a violet reflection. They explode violently on heating (Ref 4)

Its **Basic Lead Salt,** known as **Lead 4,6-Dinitroresorcinate, Basic (LDNR, Basic),** or *Diplumbic Dinitroresorcinol,*



, mw 646.53, N 4.3%; exists in two forms: 1) microcrystalline, low density form and 2) a denser brick-red form; density values are not given in Ref 6; both products have, accdg to K.S. Warren, PATR **1448**(1944), the same value for sensitivity to impact

Its laboratory method of prepn, given in Ref 8, p 192 and in PATR **1448**, p 9, consists of two steps: prepn of Pb hydroxide and of LDNR, Basic. To a soln of 18.96 g of Pb acetate in 67 ml of warm distd w add gradually, with stirring, 4.0 g of NaOH dissolved in 67 ml of w and continue stirring for 5 mins. After allowing to settle, wash the white ppt of Pb hydroxide, by decantation three times with 100 ml of distd w, and use immediately for the next operation

Disperse in 270 ml of distd w, by vigorously beating with motor stirrer, 5.56 g of 4,6-DNR, prepd as described under 4,6-Dinitro-1,3-dihydroxybenzene, and heat the dispersion to 90°. Introduce in small portions and with constant stirring the freshly prepd slurry of Pb hydroxide. Continue agitation for 3 hrs while maintaining the temp at 90°. Wash the resulting ppt of Basic LDNR once by decantation and again on the filter with alcohol, and dry overnight in a desiccator over Ca chloride. The yield of product is ca 15.6 g

For large scale manuf, the British developed during WWII a method consisting of direct nitration of granulated resorcin with concd nitric acid, and allowing the resulting DNR to react, without purification, directly in slurry with an excess of Pb hydroxide at 90° (Ref 8, p 192)

Properties of Basic LDNR: *Brisance* - 15 g of sand crushed by 0.4 g sample ignited by BkPdr fuse (31.3% TNT); *Explosion Temperature* - 295°/5 secs; *Heat of Explosion* - not given; *100° Heat Test* - % loss of wt in 1st 48 hrs 0.4, in 2nd 48 hrs 0.0 and no expln in 100 hrs; *Hygroscopicity* - not given; *Impact Sensitivity*, BM App, 1 kg wt 60 cm with 20 mg sample; *Initiating Efficiency* - 0.4 g of Basic LDNR does not initiate Tetryl pressed at 3000 psi; *Vacuum Stability* - not given

Uses: As an initiating expl, principally in electric detonators (Ref 8, pp 190-92). BritP 165069 (1921) of Peters Cartridge Co, listed in CA **16**, 648 (1922), a priming compn for small arms cartridges contains "diplumbic dinitroresorcinol" together with other ingredients

There is no US Military Specification for Basic LDNR

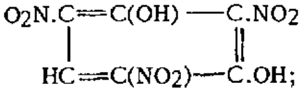
2,6-Dinitro-1,4-dihydroxybenzene, golden-yel crysts + H_2O (from w), mp 93-94°, anhyd crysts, mp 136-37°. Its **Barium Salt**, $BaC_6H_2N_2O_6 + H_2O$, dk-blue crysts, when anhyd is very explosive (Ref 5)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **6**, 791, (394) & [793] 2) Beil **6**, {4274} 3) Beil **6**, 827, (404), [823] & {4351} 4) Beil **6**, 828, (405), [824] & {4352} 5) Beil **6**, 858, (418), [850] & {4444} 5a) G.C. Hale & W.H. Rinkenbach, USP's 214568, 214569 and 214570 (1938) & CA **32**, 7668 (1938) (Use of normal LDNR in mixts with either BkPdr, Tetracene or NC) 6) Urbański **1** (1964), 536-38 6a) Kirk & Othmer, 2nd ed, **8** (1965), 591 (2,4- & 2,6-Lead Dinitroresorcinate) 7) Urbanski **3** (1967), 220-21 8) W.R. Tomlinson, Jr & O.E. Sheffield, "Properties of Explosives of Military Interest", **AMCP 706-177** (1967), pp 187-89 (Lead Dinitroresorcinate, Normal); 190-92 (Lead Dinitroresorcinate, Basic) 9) G.W.C. Taylor & S.E. Napier, BritP 1094921 (1967) & CA **68**, 5003 (1968) (Prepn of granular, free-flowing Pb 4,6-dinitroresorcinate from $Pb(OH)_2$ & 2,4-DNR)

TRINITRODERIVATIVES OF DIHYDROXYBENZENE

2,4,6-Trinitro-1,3-dihydroxybenzene; 2,4,6-Trinitroresorcinol; or STYPHNIC ACID (called 2.4.6-Trinitro-1.3-dihydroxy-benzol; 2.4.6-Trinitro-resorcin or Styphninsäure in Ger), abbreviated as **TNR** or **StA**



$$\begin{array}{l} O_2N.C{=}C(OH)\text{—}C.NO_2 \\ \quad | \qquad\qquad\quad \| \\ \quad HC{=}C(NO_2)\text{—}C.OH \end{array}$$

; mw 245.11, N 17.14%; sl hygr crysts exist in two modifications: stable form, mp 176-177°, and unstable form, mp 165-66°; dyes animal fibers intensely; sl sol in w (9.45 g of StA dissolves in 100 ml w at 15° & 0.68-0.69 at 25°); readily sol in glycol diacetate (13 g in 100 ml at RT); sl sol in nitric and sulfuric acid and practically insol in their mixtures; heat of combustion 537 cal/mol. Styphnic Acid of high purity is prepd by sulfonation (93-98% H_2SO_4) of resorcinol at 50° and nitrating with 90% HNO_3 at not over 65°. Low purity material is obtd by addg 10-20 parts of Na nitrite to the H_2SO_4 & resorcinol and nitrating with sl more HNO_3 under the same conditions as above (Ref 1). Detailed description of prepn is given by Urbański (Ref 18, p 540), who also mentions its manuf by continuous method by Meissner and by the method of Wilkinson. Both Meissner and Wilkinson methods produce impure brown acids of low mp - max 170°. Same manufacturers claimed that when the acid is used to produce its Pb salt, better results are achieved with impure acid of mp

170° than with a purified product of mp 176-77° (Ref 18, p 539)

Explosive Properties, listed by Blatt (Ref 8) and Urbański (Ref 18), are as follows: *Brisance* by Sand Test - 94% TNT or 75% MF; *Hygroscopicity* - slight (Ref 18, p 539); *Impact Sensitivity* - same as Picric Acid or 35% TNT for 50% impact work explns; *Initiation Sensitivity* - min initiating chge for 0.5 g sample pressed with initiator to 3400 psi in a No 8 detonator is Diazodinitrophenol 0.110 g, MF 0.225 g & LA 0.075 g; *Power* by Ballistic Pendulum 94% PA, by Lead Block Expansion 82% MF; *Pressure* in Bomb - develops 2840 kg/sq cm at loading d 0.25 or 100% TNT; *Rate of Detonation* - ca 1/2 that of MF; *Sensitivity to Heat* - explodes at 314°

US Military requirements are not covered by Specification

Styphnic Acid forms numerous salts & addn compds, which are expl, such as:

Barium salt, $BaC_6HN_3O_8 + H_2O$, orn plates, explodes on heating above 160°. See also Ref 25

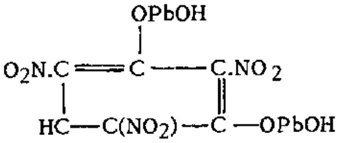
Potassium salt, $KC_6H_2N_3O_8 + H_2O$, lt-yel prisms (from w), explodes when heated on a Pt block, becomes anhyd on heating to 100°

Dipotassium salt, $K_2C_6HN_3O_8 + H_2O$, orn ndls (from w), explodes violently on heating, becomes anhyd at 120°

Sodium salt, $NaC_6H_2N_3O_8 + 3H_2O$ (from w), explodes violently on heating betwn 120 & 130°

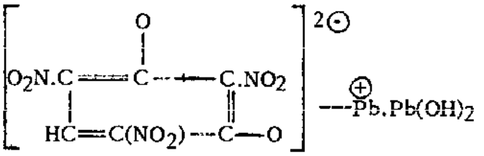
The most important salts of Styphnic Acid are its lead salts:

LEAD STYPHNATE, BASIC; Lead Hydroxide Styphnate; or Lead Hydroxide-2,4,6-Trinitroresorcinate

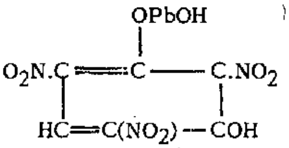


According to this structure it is a dibasic salt; mw 705.53, N 6.0%; exists in two forms: 1) a yel modification, ndls, d 3.878 and 2) red modification, prisms 4.059; apparent d 1.4 to 1.6; practically insol in w and org solvents listed under normal LSt; sol in Amm acetate

Accdg to Urbański (Ref 21, p 214), it seems that P. Griess, Ber **7**, 1224 (1874) was the first to prep this salt, although he ascribed to his product a different structure than that shown above. The usual method of prepn of Basic LSt is by the reaction of Pb acetate with Na or Mg styphnate. R.A. Zingaro, JACS **76**, 816 (1954) prepd a dibasic salt by slowly adding a soln of normal Pb styphnate in a 2% soln of NaOH at 65-70° to an aqueous soln of Pb nitrate. Zingaro also reported that the structure



is possible for his salt. Depending on the condition of reaction, there is a possibility of formation of monobasic salt,



In the patents of Brün (Ref 4), basic LSt was prepd by addg Pb nitrate to styphnic acid in the presence of NaOH. See also prepn reported by Hitchens & Garfield (Ref 5)

Properties of Basic LSt. Very few expl props of Basic LSt have been detd at Picatinny Arsenal: Activation energy is 75.4 kcal/mol, Ignition temp 295° for 5 sec value. It might be assumed that the expl props of Basic LSt are similar to those of the normal salt (qv)

Husten (Ref 16) reports that Basic LSt is a violent expl; in addn to its heat, friction, and impact sensitivity, it also has an electrostatic sensitivity which renders handling & loading particularly hazardous. He describes a process for fine milling which results in uniform particle size distribution in the 5-micron range. Milled Basic LSt resembles other styphnates in color & texture. It should be stored with a water-alcohol soln or isopropyl alc for ½-lb portions. To prevent improper use, all styphnates, in or out of storage, should be properly identified (Ref 16)

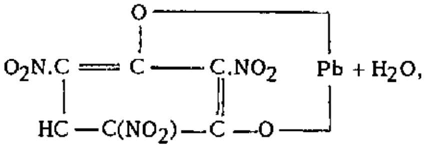
Destruction of Basic LSt by Chemical Decomposition can be achieved by a method similar to that for Normal LSt

Uses of Basic LSt. It is used as a component of priming compositions, of which the best known is the *NOL No 130 Primer Mixture:* Basic LSt (Commercial) 40±2., Tetracene (Commercial) 2.±0.5, Antimony Sulfide, Grade II, Class E 15.±1.5, Barium Nitrate 20.±2 and LA (Spec MIL-L-3055) 20±2% (Ref 17, p VII F). This mixture has been used as a top chge in: Stab Primer M97 - 85 mg with Base Charge of LA (200 mg) (p XII A); Stab Primer T96 - 120 mg with 180 mg of LA as an intermediate chge and 165 mg of LA as a base chge (p XII B); Percussion Primer T98 (p XIII B); Stab Detonator M46 - 115 mg with 300 mg LA as an intermediate chge and 145 mg of RDX as a base chge (p VII B); and Stab Detonator M47 - 15 mg with 110 mg LA & 34 mg RDX (p VII B). In Stab DetonatorsXM61E2, T33E1, T37, T51E1, T54 & T58 listed on p VII C, and in T59, T82, T83, T84E1, T86 & T87 listed on p VII D, NOL No 130 is used in conjunction with LA as an intermediate chge and RDX as a base chge. In Stab Detonator T57 listed on p VII C, 50 mg of NOL No 130 are used in conjunction with 150 mg of LA and 120 mg of Tetryl; and in T64, 15 mg of NOL No 130 are used with 40 mg of PVA LA and 10 mg of Tetryl; and in T71 listed on p VII D, 16 mg of NOL No 130 are used in conjunction with 54 mg of PVA LA and 16 mg of PETN (Ref 17, p VII D)

Analytical procedures for a mixture of Basic LSt, LA, Tetracene, Ba nitrate & Sb sulfide are given in Vol 1 of Encycl, pp A 586 & A587

US military requirements and tests for Basic Lead Styphnate are given after the Refs for Trinitrodihydroxybenzene and Derivatives

LEAD STYPHNATE, NORMAL (LSt) or Lead 2,4,6-Trinitroresorcinate (LTNR) [called Bleistyphnat, Bleitrinitroresorcinat or Trizinat in Ger; Styphnate de plomb in French; Stifnato di piombo in Ital; Stifnat svintsa or Trinitroresortsinat svintsa (Teneres) in Russian; and Tricinato (Trizinato) or Trinitroresorcinato de plomo in Spanish],



mw of monohydrate 468.30, N 9.0%; yel-orn or reddish-brn rhombic needle-like crysts, d 3.02 at 30° and 2.9 for anhyd salt; apparent d of uncompressed material is 1.4 to 1.6; mp - explodes at 260-310° depending on rate of heating; v sl sol in w (0.04% at RT); less sol in acetone and ethanol; and insol in ether, chlf, CCl_4, CS_2, benz, toluene, concd HCl & glac acetic acid. Sl sol in glycol diacetate (0.1% at RT) and soluble in 10% aq Amm acetate (Refs 20, 21 & 24). It is decompd by concd nitric or sulfuric acid (Ref 24, p 7-11)

LSt was first prepd in 1914 by E. vonHerz by adding a soln of Mg styphnate to a soln of Pb acetate, followed by adding nitric acid to convert the basic LSt to the normal salt (Ref 1). In a laboratory procedure described in Ref 20, p 195, the soln of 14.4 g Pb acetate in 320 ml distd w (acidified with 1 ml of glac AcOH), is added, under agitation, to a soln of 2,4,6-TNR in 80 ml distd w contg 1.73 g of anhyd Na carbonate. The temp is kept betw 70 & 75° during the addn and 3 hrs afterwards while the agitation is continued. Then the mixt is cooled to RT and allowed to stand for 5 hrs. Finally, the mixt is evaporated to 1/3 its volume, cooled and filtered. The resulting ppt of LSt is washed with distd w to neutrality and stored in a wet condition (Ref 20, p 195)

Accdg to Ref 24, p 7-11, in commercial production, the Mg salt (prepd by the reaction of Mg oxide and styphnic acid) is used to react with Pb nitrate or acetate in presence of an acid

The following methods of manufg Normal LSt are briefly described by Urbański (Ref 21, pp 218-20): 1) Method used at Wolfratshausen, Germany and 2) Continuous Method of J. Meissner. In both methods starting materials are MgO, TNR & Pb nitrate. In the 1st part of manuf, a suspension of Mg reacts with TNR in w to form soluble Mg Styphnate, while in the 2nd part MgSt reacts with Pb nitrate to form insol LSt. The apparatus used in Meissner process is the same as for manuf of LA. It is shown in Fig 50 of Ref 21

Methods of prepn of LSt have been examined recently at PicArsn and the following confidential reports were issued:

A) R.J. Gaughran & J.P. Picard, "The Preparation of Colloidal Normal Lead Styphnate", PATR **2077** (Oct 1954) (Conf)
B) A.C. Forsyth & Pvt M.M. Jones, "The Development of New Preparative Methods for Normal Lead Styphnate", PATR **2306** (Dec 1955) (Conf)
C) H.A. Bronner & H.J. Jackson, "Normal Lead Styphnate: Development of Standardized Preparative Methods", PATR **3079** (Aug 1963) (Conf)

Properties of Normal LSt: *Activation Energy* is accdg Ref 14, p 194 75.39 kcal/mole & Induction Period 0.5-10 sec; accdg to Ref 24, p 7-12, the "calculated activation energy for the decomposition" of LSt is 61.5 as compared with a value of 25.2 for MF; *Brisance* - 24 g sand crushed by 0.4 g sample when initiated by MF vs 48 g for TNT and only 11 g when ignited by BkPdr fuse (Ref 20, p 193 & Ref 24, p 7-12); *Explosion Temperature* - 282°/5 sec (Refs 20 & 24); *Exudation* - none; *Friction Sensitivity* - detonates by steel and fiber shoes; *Gas Volume* 368 cc/g; *Heat of Combustion* - 1251 cal/g; *Heat of Explosion* - 457 cal/g; *Heat of Formation* - 92 cal/g; *100°C Heat Test* - % loss in 1st 48 hrs 0.38, in 2nd 48 hrs 0.73 and no expln in 100 hrs; *Hygroscopicity* - gains 0.05% at 25° & 100% RH and 0.02% at 30° & 90% RH (Ref 24); *Ignition* - much more easily ignited by flame or electric spark than MF, LA or DADNPh (Ref 24, p 7-12); *Impact Sensitivity*, BM App, 1 kg wt - 17 cm with 20 mg sample and PicArsn App, 8 oz wt - 8 inches with 22 mg sample (Ref 24, p 193) (Compare with values given in Ref 21, p 215); *Initiation Efficiency* - poor; while it detonates straight 60% Dynamite and sometimes Gelatin Dynamite, it does not detonate any of the military HE's except unpressed PETN. Its minimum detonating chge is 0.55 g, as compared with 0.30 g of MF or 0.04 g of LA (Ref 24, p 7-12); *Power*, by Lead Block Expansion Test (Trauzl Test) - 40% TNT or 39% PA (Refs 8 & 20); *Rate of Detonation* - 5200 m/sec at 2.9, when initiated with blasting caps (Refs 20 & 24); *Sensitivity to Electrostatic Discharge* - 0.0009 joules; *Specific Heats, cal/cm/°C* - 0.141 at −50°, 0.164 at 25°, and 0.167 at 50°; *Stability in Storage at 75-80°* - not affected by being stored for 2 months; *Vacuum Stability at 100-120°* - 0.4 cc/40 hrs (Refs 8, 20 21 & 24)

Destruction of LSt by Chemical Decomposition. LSt is decomposed by dissolving it in at least 40 times its weight of 20% NaOH soln (or in 100 times its wt of 20% Amm acetate soln) and adding Na dichromate equal to half the wt of LSt, dissolved in 10 parts of tap water

Uses: The ease of ignition of Normal LSt renders it suitable for use as an ignition charge for LA, and as an ingredient of priming compns (Ref 9). Moisak [Trans of ButlerovInstChemTechnolKazan' **2**, 81-5 (1935)] showed that LSt could be used as a covering material for LA charges, providing protection from mechanical and chemical influences and, at the same time, increasing the detonating ability of the total charge (Ref 20, p 195)

Accdg to K.S. Warren, PATR **1448** (1944), LSt was used in Ger & Ital primer and in PATR **2145** (1955), p Rus 13, several Rus percussion compns contd LSt (See Tables A & B). In PATR **2510** (1958), p Ger 13, it is stated that LSt was used in Germany as an initiating expl since 1920 when the so-called "Trizinatkapseln" were put on the market by the Rheinisch-Westfälische Sprengstoff AG

Accdg to StdMethodsChemAnalysis (Ref 14, p 1367), the following modern noncorrosive types of primer compositions contain Normal LSt: a) LSt 38, Ba nitrate 39, Pb peroxide 5, Ca silicide 11, Sb sulfide 5 & Tetracene 2%; b) MF 25-40, LSt 5-10, Ba nitrate 24-44, Sb sulfide 0-16, Pb dinitrosalicylate 4-10 &abrasive 0-30%; and c) MF 30-31.5, LSt 10-5.5, Ba nitrate 29-30.5, Pb sulfocyanate 10-10.5 & abrasive 20-21%.

LSt has been used extensively abroad as igniting charge for LA and to some extent in the US as an ingredient of priming compositions (Ref 24, p 7-12). On p 7-18 of Ref 18, it is stated that LSt has been used in electric primers and the following compn is used for that purpose: LSt 39.5, Ba nitrate 44.4, Ca silicide 14.1, carbon black 1.0 & gum arabic 1.0%

Accdg to Odierno of Picatinny Arsenal, Dover NJ (Ref 17), Normal LSt has been used in primers. It is designated in his work as "MLSt" which means "milled product with gum binder" and if it is designated as "NLSt", it means "Normal LSt with NC binder"

The following mixtures contg NLSt are listed in Ref 17:

Mixture No 92 - NLSt 40.±2 & Sb sulfide (Grade I, class a) 60.±2% (p VII G). This mixt has been used as a primer chge (20 mg) in Stab Detonators T92 & T93 with LA (90 mg) as an intermediate chge and RDX (34 mg) as a base chge (p VII E)

Straight MLSt has been used as a "spot" (fusehead) chge in the following Electric Detonators: M36A1, M52 (T72), XM64, XM67 (T76) (p IV A); and T22E1 (p IV B). As a priming chge in these detonators, DLA (dextrinated Lead Azide) has usually been used with PETN as a base chge

Straight NLSt has been used as an upper chge (25 mg) with 65 mg of LA (RD-1333) as a lower chge in Relay XM10 (pX A) and as a primer mixt in Percussion Primer M27 (p XIII A)

Accdg to US Spec MIL-D-46209(MU), 29 Feb, 1968, primer mixt for Detonator M44E1 may contain either Normal or Basic LSt; other ingredients of mixt are Ba nitrate, Tetracene, LA and Sb sulfide

US military requirements and tests for Normal LSt are given after the spec requirements and tests for Basic LSt

Refs: 1) Beil **6**, 830, (405), [825] & {4354} 2) Beil **6**, {4356} 3) H.R. Hailes, TrFaradSoc **29**, 544 (1933) (Thermal decompn of Lead Styphnate) 4) W. Brün, USP 1942274 (1934) & CA **28**, 1866 (1934); USP 2137234 (1938) & CA **33**, 1942 (1939) (Process for making Basic LSt) 5) A.L. Hitchens, Jr & F.M. Garfield, USP 2265230 (1941) & CA **36**, 2146 (1942) (Process for making Basic LSt) 6) R. Schmitt, SS **38**, 148-49 (1943) (Quantitative detn of Styphnic Acid by pptn with phenylacridine soln. Lead Styphnate also may be detd by same method) 7) Davis (1943), 140 & 169 8) Blatt, OSRD **2014** (1944) (Trinitroresorcinol & Lead Styphnate) 9) All & EnExpls (1946), 70 (Lists primer compns as Pb St) 10) ADL PureExplCompds, Part 1 (1947), 74; Part 2 (1947), 238 11) Stettbacher (1948), 98 12) H. Ficheroulle & A. Kovache, MP **31**, 8, 9, 24 & 25 (1949) 13) US Specification MIL-L-17186(1) (Oct 1958) (Lead Styphnate, Normal, "Commercial Grade") 14) StdMethodsChemAnalysis **2B** (1963), 1346, 1367 & 1369 15) Sax (1963), 757 (Incorrectly lists Styphnic Acid as 2,4-Dinitroresorcinol) 16) B.F. Husten, "Basic Lead Styphnate, Fine Milling of", **NAVORD OD 23996** (March 1963) 17) S.J. Odierno, "Information Pertaining to Fuzes", Picatinny Arsenal, Vol IV (1964) (Explosive Components) 18) Urbański **1** (1964), 538-43 (Styphnic Acid) 19) Kirk & Othmer, 2nd edit **8** (1965), 590 (Normal LSt) 20) W.R. Tomlinson, Jr & O.E. Sheffield, "Properties of Explosives of Military Interest", AMCP **706-177** (March 1967), 193-96 (Lead Styphnate) 21) Urbański **3** (1967), 213-20 (Lead Styphnates) 22) US Specification JAN-L-757A (Sept 1967) and Amendment 1 (Sept 1968) (Lead Styphnate, Normal) 23) US Specification MIL-L-16355A (Sept 1967) (Lead Styphnate, Basic) 24) Anon, "Military Explosives", **TM 9-1300-214/TP 11A-1-34** (1967), pp 7-10 to 7-12 (Lead Styphnate); p 7-19 (Electric Primer and Squib Primary Compositions Containing LSt) 25) G.W.C. Taylor et al, BritP 1094981 (1967) & CA **68**, 5921 (1968) (Ba Styphnate prepd from Mg Styphnate soln, Ba acetate soln at 75° with HNO_3 added after 20 mins. The Monohydrate crysts had a bulk d of 1.49 g/cc) 26) Y. Ishiwata, JIndExplsSoc (Japan) **30**, No 2, 18 (1969) (Describes two methods of prepg Normal LSt)

Lead Salts of Styphnic Acid. Analytical Procedures. Two lead salts of styphnic acid are used in military primary compns: basic and normal

The following qualitative test is described in StdMethodsChemAnalysis **2B** (1963), p 1346:

Procedure: Wet, in a small beaker or flask, ca 0.1 g of material to test (usually light orange to reddish-brown in color), with ca 10 ml of distd water, and then add slowly 10 ml of 20% soln of Amm acetate. Agitate the mixture until soln is complete and add 10 ml of 10% K dichromate soln. The appearance of a bright yel ppt indicates the presence of Pb

Add to another 0.1 g of material in a small beaker, 10 ml of a 10% HCl soln and heat the mixt on a steam bath to dryness. Remove the beaker from the bath, cool it and add 10 ml of diethyl ether. Mix the contents, allow to settle and decant or filter off the ether into another beaker. Allow the ether to evaporate at RT, dissolve the residue in 25 ml of distd w, and add 0.1 g of solid KCN. The absence of color indicates that the sample is LSt

Note: This method does not make any distinction between basic and normal LSt

Accdg to A.R. Lusardi of PicArsn, LSt does not form any ppt with Nessler's Reagent. The presence of lead can be verified by hydrogen sulfide test, potassium dichromate test or dithizone test

Basic Lead Styphnate (Basic LSt)

US Military requirements and tests for Basic LSt for use in priming compositions are described in US Specification MIL-L-16355A (1967), superseding MIL-L-16355 (1966) and FA-PD-2473 (1961)

Basic LSt is delivered wet with not less than 20% of water, in bags of rubberized cloth, each bag containing ca 25 lbs of material (dry weight)

Requirements:

1) *Appearance.* The material shall be free from visible impurities and aggregates of crysts

2) *Crystal Form.* The material shall consist of discrete crysts

3) *Crystal Dimension.* 95% of the crysts shall have a max dimension betw 7 & 20 microns. The length of the crysts shall not be greater than 3 times it width

4) *Granulation* - 100% shall pass thru US Std sieve No 100

5) *Apparent Density* - 1.40 to 1.60 g/cc

6) *Acidity, pH* - 6.0 to 7.0 in 1% soln

7) *Material Insoluble in Ammonium Acetate* - maximum 0.2%

8) *Material Soluble in Ether* - maximum 0.1%

9) *Lead Content* - 59.6 to 60.2%

10) *Nitrogen Content* - 5.97 to 6.17%

11) *Instantaneous Flash Point* - 330 to 350°C

Sampling. Basic LSt arrives with not less than 20% of w, packed in the same manner as described under Normal LSt. Prepn of samples for testing is the same as for Normal LSt

Preparation of Dry Sample. Use the same procedure as for Normal LSt

Tests:

1) *Appearance.* Use the same procedure as described under Normal LSt

2) *Crystal Form.* Use the same procedure as described under Normal LSt

3) *Crystal Dimension.* Use the same procedure as for Normal LSt

4) *Granulation.* Use the same procedure as for Normal LSt

5) *Apparent Density.* Use the same procedure as described under Normal LSt

6) *Acidity, pH.* Use the same procedure as described in test 3, under Lead Mononitroresorcinate

7) *Material Insoluble in Ammonium Acetate.* Use the same procedure as for Normal LSt

8) *Material Soluble in Ether.* Use the same procedure as for Normal LSt

9) *Lead Content.* Use the same procedure as for Normal LSt

10) *Nitrogen Content* is determined by *titanous chloride* method as described below. Before proceeding with the test, it is necessary to prepare the following reagents:

a) Potassium Dichromate Solution (0.2000N). Dry the NBS $K_2Cr_2O_7$ standard 136 (or equivalent) in an oven at 180° for 2 hrs, cool, dissolve 9.8070 g in ca 900 ml distd w and dilute to 1 liter in a volumetric flask. This is a primary standard

b) Acetic Acid Solution (25%). Add 250 ml of glac AcOH to 750 ml distd w

c) Titanous Chloride Solution (0.2N). Can be prepd from either 20% titanous chloride

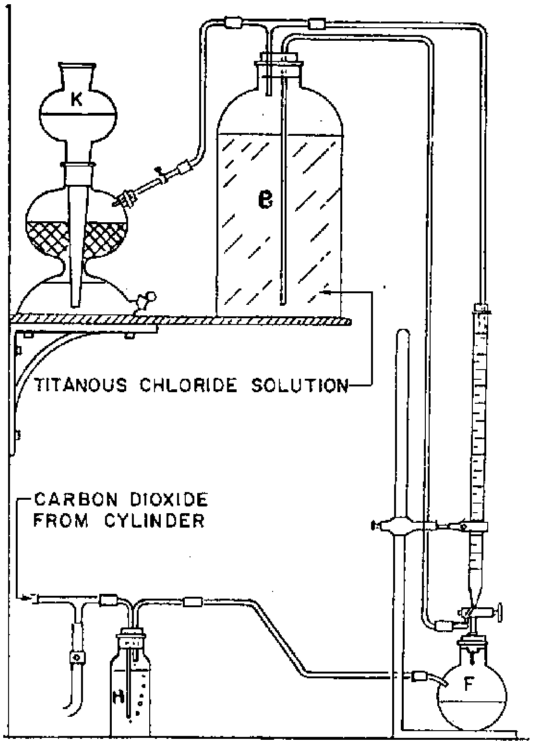


Figure 1 APPARATUS FOR TITANOUS CHLORIDE TEST

soln or from titanium hydride. We are describing here the 1st method

Filter ca 150 ml of 20% titanous chloride soln thru a glass filtering crucible, add ca 100 ml concd HCl and mix by means of oxygen-free carbon dioxide (or nitrogen). Add 750 ml of freshly distilled water, boiled and cooled in absence of oxygen, and mix again using the same gas. Store in an amber bottle (B) under carbon dioxide supplied by a Kipp generator (K) as shown in Fig 1. The soln need not be standardized as it is compared with std ferric ammonium sulfate soln each time analysis is made

d) Ammonium Thiosulfate Solution (20%). Dissolve 20 g of NH_4CNS in 100 ml of distd w

e) Sodium Acetate Solution (20%). Dissolve 20 g of CH_3COONa in 100 ml of w

f) Stannous Chloride Solution. Dissolve 50 g of $SnCl_2.2H_2O$ in 100 ml of concd HCl and dilute to 1 liter with w

g) Barium Diphenylamine Sulfonate Indicator. Add 0.095 g of Ba diphenylamine-sulfonate to 100 ml of w and stir until complete dissolution. Add 10 ml of dilute (1 to 1) sulfuric acid, let stand for several hrs, and filter. Add 500 ml of 95% phosphoric acid and dilute to 1 liter

h) Ferric Ammonium Sulfate Solution (0.2N). Dissolve 100 g of $FeNH_4(SO_4)_2.12H_2O$ in 1 liter of 5% sulfuric acid and determine its normality as follows:

Pipet 25 ml of the above soln in a 500 ml wide mouth Erlenmeyer flask (F), showh in Fig 1 and Fig 2, add 25 ml of concd HCl and heat nearly to boiling. Add the above stannous chloride soln dropwise and with stirring until the yel color of ferric chloride disappears and then add exactly one drop in excess. Dilute to ca 200 ml with distd w and let stand for 5 min with occasional shaking. Add 15 ml of diphenylamine sulfonate indicator and titrate with std 0.2000N K dichromate soln until the change at the end point is from lt green to purple

Normality of ferric ammonium sulfate soln is determined from the expression (AxN) / B, where A = ml of K dichromate soln; N = its normality (0.2000); and B = ml of ferric ammonium sulfate taken for titration

Procedure. Transfer 0.6 to 0.8 g of the dry sample weighed to 0.1 mg to a 100 ml beaker. Add 50 ml of 25 percent acetic acid soln and stir to dissolve. Transfer to a 250 ml volumetric flask and dilute to the mark. Attach the cylinder of oxygen-free carbon dioxide to the titration flask as shown in Fig 1 and allow the gas to flow for 5 minutes thru gas humidifier (H) to displace the air. Continue the flow of gas during the entire titration procedure. Pipet a 25 ml aliquot of the solution of the sample into the titration flask (Fig 2). Add 30 ml of 20% Na acetate soln and 20.00 ml of 0.2N titanous chloride soln and swirl the flask for about 20 secs. Add 25 ml of 15% HCl. Titrate with ferric ammonium sulfate soln until the purple color begins to fade, then add 5 ml

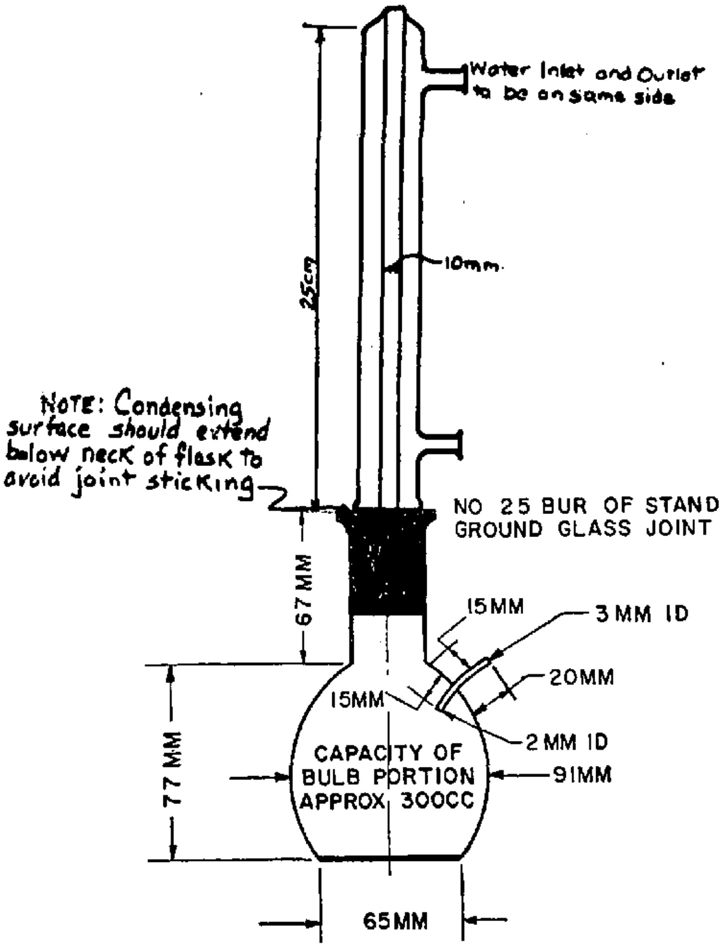


Figure 2 TITRATION FLASK FOR TITANOUS CHLORIDE TEST

of 20% ammonium thiocyanate soln and continue the titration to a faint permanent pink. A blank run should be made using the quantities of reagents as above, but omitting the sample. This will provide a value for the amount of ferric ammonium sulfate equivalent to the 20.00 ml of titanous chloride soln. Calculate the percent nitrogen in the sample as follows:

$$\text{Percent Nitrogen} = \frac{(A-B) \times 0.00233N \times 1000}{W}$$

where: A = ferric ammonium sulfate soln for the blank, ml
B = ferric ammonium sulfate soln for the sample, ml
N = normality of ferric ammonium sulfate soln
W = weight of sample, grams

Note: This method, suitable for detn of nitro-nitrogen content, is also described in MIL-STD-650(1962) Method 409.1. Preparation of 0.2N Titanous Chloride Solution is described in Method 605.1; prepn of Barium Diphenylamine Sulfonate Solution in Method 705.1 and prepn of 0.15N Ferric Ammonium Sulfate in Method 603.1

11) *Instantaneous Flash Point.* Use sample of the size that may be held on about 1/16 inch of the flat end of an ordinary toothpick and a heated Dennis melting point bar. The instantaneous flash point is determined by noting the times required for the explosion of samples dropped on the bar at several temperatures slightly lower than the instantaneous flash point. Then by plotting a time-temperature curve and extrapolating to zero time, the instantaneous flash point is obtained

Analytical procedures for mixtures contg Basic LSt, LA, Tetracene, Ba nitrate and Sb sulfide are described in Vol 1 of Encycl, p A586 & A587

Normal Lead Styphnate (Normal LSt)

US Military requirements and test for Normal LSt for use in priming compositions are described in US Military Specification MIL-L-757A(1967) & Amendment 1(1968)

Normal LSt is delivered wet with not less than 20% of water in bags of rubberized cloth, each bag contg ca 25 lbs of material (dry weight). The bags are placed inside metallic barrels or drums

Requirements:

1) *Appearance.* The material shall be free from visible impurities and free from aggregates which are larger than the maximum allowable crystal dimensions permitted, as stated under "Crystal Dimension"

2) *Crystal Form.* The material shall be crystalline and the longest dimension of any crystal shall not be greater than 3 times the next longest axis. Hexagonal plates are acceptable

3) *Crystal Dimension.* The largest dimension of any crystal shall not exceed 0.5 mm

4) *Granulation.* One hundred percent shall pass thru a US Standard Sieve No 100
5) *Apparent Density.* 1.30 to 1.60 g/ml
6) *Acidity, pH.* 5.0 to 6.0
7) *Material Insoluble in Ammonium Acetate,* max 0.3%
8) *Material Soluble in Ether,* max 0.1%
9) *Lead Content* - 43.2 to 43.3%
10) *Nitrogen Content* - 8.87 to 9.07%
11) *Instantaneous Flash Point* - 310 to 330°C

Sampling: Take from each of the barrels (or drums) in a lot (which shall not exceed 500 lbs), a 100 g sample, using a horn spoon. Mix each of these "primary samples" and place ca 30 g out of each 100 g in a bottle, close it tightly with a rubber stopper and attach a label in such a manner that the barrel from which the sample was taken can be identified. Blend the remaining 70 g portions of the "primary" samples on a smooth surface with a horn spatula, spread the material out and divide into squares ca 0.5-inch on a side. Remove from all of these squares enough material to form a "composite" sample of ca 50 g LSt. Mix this "composite" sample thoroughly and place in a bottle provided with a rubber stopper. Close the bottle tightly and label it giving the name of the material, name of manufacturing plant, purchase order and number of pounds in the lot. All acceptance tests shall be made on this "composite" sample representative of the lot. Hold the "primary" samples for possible future examination should the "composite" sample fail to meet the specification requirements

Preparation of Dry Normal LSt. If visible inspection of a wet sample does not show the presence of foreign matter, transfer 10 g of material from the bottle contg ca 50 g of "secondary" sample, prepd as described above. Spread on a medium texture filter paper placed in a Büchner funnel and apply suction until the sample is almost dry. While working behind a safety shield, remove the sample from filter and spread it over a watch glass (No safety shield is required when working with Lead Mononitroresorcinate)

Place the watch glass with the sample in a desiccator over concd sulfuric acid for 24 hrs or for at least 2 hrs in an oven at 70-75°. Use this sample for all tests, unless otherwise specified

Tests:

1) *Appearance.* Take four 0.5 mg samples each from different places in the dried sample and spread them over an area of 2 cm^2 on a glass microscopic slide. Examine material first macroscopically for impurities under daylight illumination and then microscopically for impurities and aggregates of crystals using a magnification of ca 30 times and reflected light

2) *Crystal Form.* Examine the same crysts microscopically using a magnification of ca 350 times and reflected light

3) *Crystal Dimensions.* Using the same slide as above, measure the longest dimension of the largest crysts observed when scanning the field with the aid of a mechanical stage. Use transmitted light and an ocular micrometer which has been calibrated by use of a stage micrometer. Consider the longest dimension to be the distance between the two most remote points of any one crystal

4) *Granulation.* Place a portion of wet sample, contg ca 10 g LSt on a dry basis, on a US Stand Sieve No 100 (conforming to Spec RR-S-366) and wash by lifting and lowering the seive in a container of water. After most of the material has passed thru the sieve, wash the remainder with a gentle stream of w played over the sieve. There should be no residue left on the screen

5) *Apparent Density.* Place 3 ml of n-butyl alcohol into a 5 ml graduate which has an ID of ca 7.5 mm and is graduated in 0.05-ml divisions. Add an accurately weighed, exactly 2.00 g dry sample to the graduate, in small portions, and wash down the material adhering to the walls of the graduate with a thin stream of n-butyl alcohol. Fill the graduate slight-

ly above the 5 ml mark, close with thumb and invert at least 5 times in order to assure thorough wetting of the material. After allowing to stand for 3 hrs at RT, observe the exact volume occupied by ppt.

Apparent Density = W/V,

where: W = weight of sample in grams; and
V = observed volume in mls

6) *Acidity, pH.* Use the same procedure as described in test 3 under Lead Mononitroresorcinate (See p D 1271)

7) *Material Insoluble in Ammonium Acetate.* Transfer an accurately weighed 1 g dry sample to a 250 ml beaker, add 100 ml of distd w and then, slowly, with constant stirring 100 ml of 20% Amm acetate soln. Stir the mixture for ca 15 mins until no further dissoln of sample is apparent and filter the mixture thru a tared sintered-glass crucible of fine porosity. Transfer any residue in the beaker to crucible with a stream of distd w and wash the ppt in the crucible with w until the washings are colorless. Dry in an oven at 100° to 105° for 1 hr, cool in a desiccator and weigh

$$\text{Percent of Material Insoluble in Acetate} = 100A / W$$

where: A = wt of residue in crucible; and
W = wt of dry sample

8) *Material Soluble in Ether.* Transfer an accurately weighed 2 g sample of the dry material to a 150 ml beaker, add 50 ml of anhydrous ethyl ether and allow to stand, with occasional stirring, for 15 mins. Decant the ether into a funnel contg a dry No 41 Whatman (or its equivalent) filter paper and catch the filtrate in a tared 100 ml beaker. Wash the ppt with a total of 20 ml of ether. Evaporate off the ether in the beaker on a steam bath or by means of a current of air (under a hood) and finally dry it to constant wt in a vacuum desiccator over concd sulfuric acid. Run a blank detn of 70 ml of ether

$$\text{Percent of Material Soluble in Ether} = \frac{100(A-B)}{W}$$

where: A = wt of residue from sample;
B = wt of residue from blank; and
W = wt of dry sample

9) *Lead Content.* Transfer 0.45 to 0.55 g, accurately weighed sample, to a 250 ml beaker and add 10 ml of concd nitric & 10 ml of concd sulfuric acids. Cover the beaker with a ribbed watch glass and heat at moderate temp on a hot plate until dense fumes of sulfur trioxide are evolved. Allow the beaker to cool for ca 2 mins, add 5 ml of concd nitric acid and heat again until dense white fumes are evolved. Repeat the addn of 5 ml portions of nitric acid until all organic matter is destroyed and the soln becomes practically colorless. Cool the beaker and wash down the watch glass and side walls of the beaker with ca 10 ml of w. Heat the uncovered beaker again until the evolution of dense wh fumes, allow to cool and then cautiously dilute with w to ca 100 ml. After allowing to cool to RT, add 25 ml of ethyl alc and let stand for at least 2 hrs. Filter thru a tared, sintered porcelain crucible of fine porosity (or a Gooch crucible with an asbestos mat), transferring the ppt from the beaker by means of stream of 5% sulfuric acid. Wash the crucible 3 times with 5% sulfuric acid and twice with 95% ethyl alc. Dry in an oven at 100 to 105° for 30 mins and then ignite in a muffle furnace at 600° for 15 mins. Cool in a desiccator and weigh the crucible with ppt of $PbSO_4$

Percent Lead = 68.32A / W,

where: A = wt of ignited residue; and
W = wt of dry sample

Note 1. A slightly different detn of Pb content is described as test 4, under Lead Mononitroresorcinate (See p D 1272)

Note 2: An older Spec MIL-L-17186 (BUORD) (1952) described detn of Pb by electrolysis with formation of PbO_2 deposit

10) *Nitrogen Content* is detd by Titanous Chloride Method in the same manner as described under Basic Lead Styphnate, Test 10

11) *Instantaneous Flash Point* is detd in the same manner as described under Basic Lead Styphnate, Test 11

Note: In the old Spec JAN-L-757A (1947) are described the following tests which are not included in the new Specs:

a) *Purity.* This method is also described in MIL-STD-650 (1969), Method 408.1 and recommended for detn of LSt content in unknown sample. For this an accurately weighed 0.25 g portion of sample is placed in a sintered glass crucible of fine porosity, and extracted with 50 ml of 20% Amm acetate soln, followed by rinsing with w until the filtrate collected in a 250 ml volumetric flask is clear. After diluting with w to the mark and mixing, a 25 ml aliquot is pipetted to a 1 liter volumetric flask and diluted with w to the mark. A portion of this soln is transferred to a Corex glass cell having a width of ca 1 cm to be used in a Quartz Ultraviolet Spectrophotometer, such as Beckman Model DU or Model D manufd by the National Technical Laboratories, South Pasadena, Calif. [This apparatus is described in the JAmerOpticalSoc **31**, 638 (1941)] Then the optical density of soln at a wave length of 410 millimicrons is determined and a similar cell filled with 20% Amm acetate solvent is tested to be a reference. Since the cell holding the soln of sample is not identical with that holding the solvent alone, it is required to introduce an optical correction. For this purpose the cell contg the soln of sample is emptied and filled with solvent alone. After detg its optical density at a wave length of 410 millimicrons, a correction is introduced and the % of LSt is calcd from the equation

$$\frac{\text{Percent LSt}}{\text{Monohydrate}} = \frac{29.41A}{WD}$$

where: A = optical density (corrected) of LSt soln;
D = width of the Corex cell in cm; and
W = wt of original sample

b) *Metals Other Than Lead,* such as Fe, Al, Cr, Cu, Mg, Ca, Mn, Na, K & Si were detd by a very tedious procedure which we are not describing since it is not included in the new Spec

c) *Brisance Index.* We are describing it here although it is not included in the new Spec

Using five Cu, Al, or gilding metal caps of the same size as the No 6 Blasting Cap (ID ca 0.217 inch), transfer to each cap accurately weighed 0.400 g portions of LSt. Place each cap in a loading block (located behind a barricade), insert a plunger 0.20-inch in diam and subject the LSt to a pressure of 3000 psi for 3 mins. Add to each cap accurately weighed 0.300 g portions of LA (complying with Spec MIL-L-3055), insert a reinforcing cap above LA and subject to a pressure of 3000 psi for 3 mins. With a pin, prick the powder train in one end of a piece of safety (Bickford) fuze, 8-9 inches long. Crimp the pricked end to each of the caps described above, taking care that the end of the fuse is held firmly against the charge in the cap. Crimp near the mouth of the cap so as to avoid squeezing the charge. Pour 80.0±0.1 g of standard Ottawa sand, which passes thru a No 20 US Standard sieve and is retained on a No 30 US Standard sieve, into the cavity of the sand test bomb (See Fig on p XXII of Vol 1 of Encycl) and level it by striking the bomb vigorously with a hammer at least 5 times while rotating the bomb on its axis. Lower the fuse and cap into the bomb cavity so that the cap is centered at the axis of the bomb and just touches the sand. Hold the cap and fuse in this position and carefully pour 120.0±0.1 g more of the sand into the bomb, being careful not to disturb the position of the cap. To avoid possible error caused by the explosion blowing crushed sand thru the hole in the cover, slip a piece of rubber tubing, (about 1/8 inch long and of such inner diam that it fits the fuse snugly) over the fuse and adjust at a point on the fuse so that the rubber will be against the inner side of the bomb cover, with the loaded cap in position as described above. Carefully insert the fuse thru the hole in the bomb cover and lower the cover into position and fasten securely. Then strike the bomb vigorously with a hammer five times while rotating the bomb on its axis. Light the fuse and after the explosion has taken place empty the sand onto a sheet of smooth (glazed) paper taking care to remove any sand which may adhere to the sides of the bomb, the bomb cover, or to pieces of the detonator cap or burnt fuse. Transfer all of the sand to a No 30 US Standard sieve fitted with a bottom pan and cover. Weigh the sand which passes thru the sieve after shaking mechanically for three minutes. Determine the weight of sand crushed by each charge as directed above, and correct this by subtracting the weight of sand found to be crushed by a charge of 0.300 g of LA. Record the av of the corrected values obtd in this manner. Determine the av weight of sand crushed by TNT complying with Spec JAN-T-248, Grade I, ground to pass a 100 mesh screen) using five charges consisting of 0.400 g of TNT, 0.05 g of Tetryl (complying with Spec JAN-T-339, Grade I) and 0.200 g of LA. Determine the total weight of sand crushed in each case, correct by subtracting the weight of sand found to be crushed by 0.05 g of Tetryl and 0.200 g of LA and calc the average of the corrected value for TNT. Calc the "Brisance Index" of LSt by dividing the av corrected value obtd for LSt by the av corrected value for TNT

Analytical procedure for a mixture consisting of LSt, LA, Tetracene, Ba nitrate and Sb sulfide is described in Vol 1 of Encycl, p A586-L and in Std-MethodsChemAnalysis **2B** (1963), p 1369

In a polarographic procedure employed by D.W. Traas & H.L. Ligteneberg, Chem & Ind (London) **1962**, 2040 & CA **58**, 3262 (1963), LSt is removed from a primer mixt contg Tetracene as other ingredient, by pptg LSt with casein. For this test weigh out a percussion mixture (30 mg) into a centrifuge tube and add 2.5 ml 4N sulfuric acid. Shake the tube for 15 mins to dissolve the Tetracene and make the volume up to 25 ml with distd w. Add 5 g finely powdered casein and shake the tube again for 15 mins. Centrifuge the tube and its contents and pipet 10 ml from the supernatant clear soln. Neutralize the soln with 25% tetramethylammonium hydroxide soln, using methyl red indicator. Pass nitrogen thru the soln to expel oxygen and polarograph the soln at 25° for detd Tetracene as detd by A.M. Wild in Chem & Ind **1957**, 1543. Percentage of LSt is calcd by subtracting from 100% the percent of Tetracene

In a polarographic procedure of A.M. Wild, Chem & Ind **1963**(20), 819-20 & CA **59**, 3710(1963), Lead Dinitroresorcinate, LSt, NC (varnish), and Tetracene are detd from their mixts in the following manner. Weigh accurately contents of cap into a tared microsinter, extract NC with dry acetone, dry the residue and weigh. Extract Ba nitrate using minimum amt of cold w, wash the residue with alc, dry and reweigh. Extract LSt with 0.01M HCl at RT using 20 ml of acid in four 5 ml portions, wash the sinter with alc, dry and weigh. Extract Tetracene with 2.60M HCl and polarograph it in acid-tartaric medium. Polarograph also the extract contg LSt, in acid-tartaric medium and if LDNR is present a different wave will be obtd

Analytical procedures for LSt are also given on pp 213-15 of "Analytical Methods for Powders and Explosives", by A.B. Bofors, Nobelkrut, Bofors, Sweden (1960). The procedures include: a) Appearance b) Content of Styphnate and c) Acidity.

E. Kurz & G. Kober, Analyst (London) **92** (1095), 391-95 (1967) & CA **68**, 4074 (1968) report that LSt and its production, intermediates and effluents were detd, and primary compns analyzed by Bolligan's extraction, titration of polynitrophenols with methylene blue by using $PhNO_2$ as an extractant. NO_3^- interference is avoided by extn of the Styphnic Acid with iso-BuCOMe before the titration

Other Lead Salts of Trinitroresorcinol

Other Pb salts and derivatives of Trinitroresorcinol include:

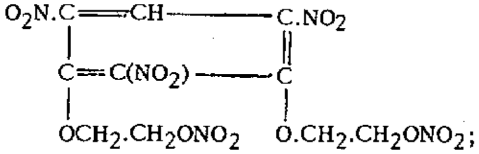
Lead Acetate Styphnate, $Pb_2(C_2H_3O_2)_2(C_6HN_3O_8)$, yel crysts, mp - explodes at 315°, d 3.1 at 30°, explodes on impact & by friction; was obtd by treating Styphnic Acid with 2 moles of Pb acetate in w at 55°, or from Pb Styphnate & aq Pb acetate soln (Ref 1a)

Lead Triethyllead Hydroxide Styphnate, $Pb[Pb(C_2H_5)_3](OH)(C_6HN_3O_8)$, yel crysts, mp - explodes at 298°; obtd by treating an aq soln of triethyllead acetate with 1 mol of Na hydrogen styphnate at 65° and introducing alkaline $Pb(NO_3)_2$ soln

Bis(Hydroxydiethyllead) Styphnate. See Bis(diethyl) Lead Styphnate, Basic in Vol 2 of Encycl, p B141-L and Ref 1a

Bis(Triethyllead) Styphnate. See Bis(triethyl) Lead Styphnate in Vol 2 of Encycl, p B160-L and Ref 1b

2,4,6-Trinitroresorcinol-3,5-di(β-nitroxy) Ethyl Ether or Trinitroresorcindiglycolether Dinitrate or 2,4,6-Trinitro-1,3-Bis(2-hydroxyethoxy)benzene Dinitrate in CA [called 2.4.6-Trinitro-1.3-bis-(2-nitryloxy-)-benzol in Ger],



mw 423.21, N 16.55%; ndls (from alc), mp 75°, bp - dec at 175° & deflgr at 265°; readily sol in acet & benz; mod sol in alc, eth & chlf; sl sol in petr eth & w; was prepd by nitrating 4,6-dinitro-1,3-bis(2-hydroxy-ethoxy)-benzene with anhyd HNO_3 & concd H_2SO_4 (Refs 1b, 2 & 4)

Blatt (Ref 3) reported the compd has FI Impact Sensitivity value of 48% PA and a Pb Block Expansion value of 116% PA

Refs: 1a) Beil **6**, {4356} 1b) Beil **6**, {4363} 2) A.H. Blatt & F.C. Whitmore, OSRD **1085** (1942), p 117 3) Blatt, OSRD **2014** (1944) 4) J.J. Blanksma & P.G. Fohr, Rec **65**, 816, 817 (1946) & CA **41**, 2704 (1947)

2,4,5,6-Tetranitroresorcinol (called Tetranitroresorcin in Ger), $HO.C_6(NO_2)_4.OH$; mw 290.11, N 19.31%; col crysts (from chlf or CCl_4), has bitter taste, mp 152°, bp - explodes; gives yel colored solns in w or alc; was prepd by nitrating 5-nitroresorcinol with concd HNO_3 & concd H_2SO_4 (Refs 1, 2 & 3)

Its **Monomethylether** deriv, $HO.C_6(NO_2)_4O.CH_3$; mw 304.13, N 18.42%, col crysts (from chlf or CCl_4), bitter tasting, mp 115°, bp - explodes; and **Monoethylether** deriv, $HO.C_6(NO_2)_4O.C_2H_5$; mw 318.16, N 17.61%; col crysts (from chlf or CCl_4), bitter tasting, mp 110°, bp - explodes; were prepd by Blanksma (2)

Refs: 1) Beil **6**, 833 2) J.J. Blanksma, Rec **27**, 35, 36 (1908); PrKAkadWetenschAmsterdam **9**, 278-80 (1907); CA **1**, 1553 (1907) & JCS **92** I, 127 (1907) 3) A.H. Blatt & F.C. Whitmore, OSRD **1085** (1942), 89 (Compd claimed to be too reactive & too sensitive for use as an expl) 4) Blatt OSRD **2014** (1944) (Listed under Nitro Compounds)

DIHYDROXYBENZOIC ACID AND DERIVATIVES

Dihydroxybenzoic Acid, $(HO)_2C_6H_3.COOH$; mw 154.12, O 41.53%. The five possible isomers are described in Beil: *2,3-Dihydroxy-* [called Brenzcatechin-carbonsäure-(3) in Ger], crysts + ½H_2O (from saturated aq soln, mp - becomes anhyd at 100°, mp 203-06° (Ref 1); *2,4-Dihydroxy-* [called Resorcin-carbonsäure-(4) in Ger], ndls + $3H_2O$ (from eth), mp - anhyd at 100° & dec 200-06° (Ref 2); *2,5-Dihydroxy-* [called Hydrochinon-carbonsäure-(2) or Gentisinsäure in Ger], ndls or prisms (from w), mp 196-200° (Ref 3); *2,6-Dihydroxy-* [called Resorcin-carbonsäure-(2) or γ-Resorcylsäure in Ger], ndls + H_2O (from w), mp 148-67° with decompn (Ref 4); *3,4-Dihydroxy-* [called Brenzcatechin-carbonsäure-(4) or Protocatechusäure in Ger], ndls or plates + H_2O (from w), anhyd at 100° & melts at 194-98° (dec) (Ref 5); and *3,5-Dihydroxybenzoic Acid* [called Resorcin-carbonsäure or α-Resorcylsäure in Ger], prisms or ndls + 1½H_2O, becoming anhyd at 100-05° & melting at 232-40° (Ref 6)

Other props & methods of prepn are found in Beil

Refs: 1) Beil **10**, 375, (173) & [248] 2) Beil **10**, 377, (176) & [251] 3) Beil **10**, 384, (180) & [257] 4) Beil **10**, 388, (186) & [259] 5) Beil **10**, 389, (187) & [260] 6) Beil **10**, 404, (195) & [266]

Nitroderivatives of Dihydroxybenzoic Acid

Mononitrodihydroxybenzoic Acid, $O_2N.C_6H_2(OH)_2.COOH$; mw 199.12, N 7.03%. The following derivs are found in Beil & in CA thru 1967: *4-Nitro-2,3-dihydroxybenzoic Acid,* golden ndls (from hot w), mp 194° (Refs 4 & 6) & 208-10° (Ref 7); *5-Nitro-2,4-dihydroxybenzoic Acid,* yel plts or ndls + ½H_2O (from w), mp 215° (dec); forms several colored cryst salts (Ref 1); *3-Nitro-2,5-dihydroxybenzoic Acid,* yel ndls (from benz or w), mp 158° (Ref 2); *4-Nitro-2,5-dihydroxybenzoic Acid,* brn crysts (from w), mp 242°, bp - sublimes w/o decompn (Refs 4 & 6); *4-Nitro-3,5-dihydroxybenzoic Acid,* dk-red ndls (from w), mp - dec 238-40° (Ref 2)

Other props and methods of prepn are given in the Refs

Refs: 1) Beil **10**, 382, (179) 2) Beil **10**, (184) 3) Beil **10**, (196) 4) S.C. Bhattacharyya & D.E. Seymour, JCS **1950**, 1139 (1950) & CA **44**, 10685 (1950) 5) K.G. Rosdahl, SwedP 127434 (1950) & CA **44**, 8374 (1950) 6) D.E. Seymour & S.C. Bhattacharyya BritP 694238 & 694300 (1953) & CA **48**, 8826 (1954) 7) D.H. Powers et al, JACS **78**, 907-11 (1956) & CA **50**, 10041-42 (1956)

3,5-Dinitro-2,4-dihydroxybenzoic Acid (called 3.5-Dinitro-2.4-dioxy-benzosäure or 3.5-Dinitro-β-resorcylsäure in Ger), $(O_2N)_2C_6H(OH)_2.COOH$; mw 244.12, N 11.48%; lt-yel ndls + 1½H_2O (from 2.5N HCl or chlf), mp - (anhyd salt) 195-205°; can be prepd by nitration of β-resorcylic acid (Ref 1). Its **Ammonium salt,** $(NH_4)_2C_7H_2N_2O_8$, citron-yel ndls, deflgr on heating; **Potassium salt,** $KC_7H_3N_2O_8$, lt-yel ndls (from w), mp - explodes; and **Dipotassium salt,** $K_2C_7H_2N_2O_8$, lt-orn ndls (from w), mp - explodes (Refs 1 & 2)

Refs: 1) Beil **10**, 383, (180), [256] 2) H. Einbeck & L. Jablonski, Ber **56B**, 1907 (1923) & CA **18**, 534 (1924)

Trinitrodihydroxybenzoic Acid, $(O_2N)_3C_6(OH)_2.-COOH$, was not found in Beil nor in CA thru 1967

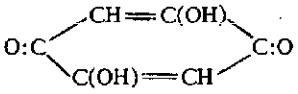
2,4-Dihydroxybenzonitrile. Its dinitro deriv was prepd by Lindemann et al (Refs 1 & 2): *3,5-Dinitro-2,4-dihydroxybenzonitrile* (called 3.5-Dinitro-2.4-dioxy-benzonitril in Ger), $(HO)_2C_6H(NO_2)_2.CN$; mw 225.12, N 18.67%; yel crysts (from dil HCl), mp 175°; was prepd by reaction of 5,7-dinitro-6-hydroxyindoxyazine with 10% NaOH and acidifying with dil HCl. The **Disodium salt,** $Na_2C_7HN_3O_6$, crysts (from a little w), mp - detonates at 230°; sol in alk or glac acet ac; sl sol in benz & Nitrobenz; was obtd on acidifying the above reaction mixt with acetic acid

Refs: 1) Beil **10** [256] 2) H. Lindemann et al, Ann **456**, 292 (1927) & CA **21**, 3363 (1927)

Dihydroxybenzophenyltriazole. See Benzophenyltriazole, Dihydro in Vol 2, p B79-L

DIHYDROXYBENZOQUINONE AND DERIVATIVES

2,5-Dihydroxy-p-benzoquinone [called 2.5-Dioxy-benzochinon-(1.4) in Ger],



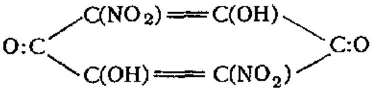
mw 140.09, O 45.68%; exists in two modifications: metastable form, lt-yel lfts or prisms (from acet ac) which, on warming in light or moistening with water or ether, transforms to a stable form, orn-yel ndls or lfts (from alc) or rhomb (thru sublimation), mp - sublimes undecompd at 100-50° & dec at 211°; mod sol in hot alc, acet, glac acet ac & w; was prepd by reacting 4 mol of K ferric cyanide & 1 mol of 1,2,4-trihydroxyanthraquinone in dil KOH at 15°. Other props are given in Refs

Refs: 1) Beil **8**, 377, (680) & [432] 2) R. Scholl & P. Dahll, Ber **57**, 82 (1924) & CA **18**, 2157 (1924)

Monoazido, $C_6H_3N_3O_4$, and *Diazido*, $C_6H_2N_6O_4$, derivs were not found in Beil

3-Nitro-2,5-dihydroxy-1,4-benzoquinone, $C_6H_3NO_6$; mw 185.09, N 7.57%; golden-yel ndls, mp-not reported; mod sol in w; was prepared by warming 1 part of 3-nitro-2-hydroxy-5-amino-1,4-benzoquinone-1-imide with 10-15 pts of 10% KOH (Refs 1 & 2)
Refs: 1) Beil **8**, 384 2) R. Nietzki & (?).Schmidt, Ber **22**, 1661(1899)

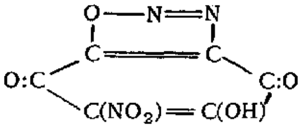
3,6-Dinitro-2,5-dihydroxy-1,4-benzoquinone or Nitranilic Acid [called 3.6-Dinitro-2.5-dioxy-benzochinon-(1.4) or Nitranilsäure in Ger]



mw 230.09, N 12.18%; hygr lfts, mp 86-87°, bp-deflgr at 170°; mod sol in w & alc; insol in eth; was prepd by reacting quinone with fuming HNO_3 and keeping the mixt at −5° to −10° for several days (Refs 1 & 2), or by nitrating hydroxyquinone diacetate with mixed acids (Ref 6), which gives a 75% yield (Ref 7)

Some of its salts are expl, such as:
Barium salt, $BaC_6N_2O_6$, golden-yel leaflets, deflgr on heating; insol in w (Refs 1 & 2)
Calcium salt, $CaC_6N_2O_6$, yel crysts (Refs 1 & 2)
Disodium salt, $Na_2C_6N_2O_8$, dk-red monoclinic prisms, mp-deflgr violently on heating (Refs 1 & 4)
Hydroxylamine salt, $2NH_3O + C_6H_2N_2O_8$, dk-yel lfts, mp-explodes on heating (Refs 1 & 5)
Lead salt (basic), $PbC_6H_3N_2 + 2PbO$, red powd, mp-deflgr at 215°; explodes on impact or frictional influences; insol in hot w (Refs 1 & 8)
Potassium salt, $K_2C_6N_2O_6$, lt-yel ndls, existing in two modifications, both monoclinic, mp, deflgr more violently than Sodium salt; very sol in hot w; diffc sol in cold w (Refs 1 & 4)
Silver salt, $Ag_2C_6N_2O_6$, yel crysts, mp-deflgr on heating (Refs 1 & 2)
Sodium salt. See Disodium salt, above
Refs: 1) Beil **8**, 384, (683), [433] & {3351}
2) R. Nietzki, Ann **215**, 140 & 141(1882)
4) J.U. Nef, Ber **20**, 2028(1887) 5) J.U. Nef, JACS **11**, 21(1889) 6) R. Nietzki, Ber **43**, 3458(1910) 7) H.O. Meyer, Ber **57B**, 327(1924) & CA **18**, 2123(1924)
8) A.G. Lignose, GerP 407416(1925)
9) G. Schwarzenbach & H. Suter, Helv **24**, 617(1941) & CA **35**, 7806 (1941) (Absorption spectrum) 10) E. Müller, ZPhysiolChem **268**, 245(1941) & CA **36**, 3817-18(1942) (Use of Nitranilic Acid as precipitation reagent for N-contg biological bases)

Anhydro-(6-nitro-2,5-dihydroxy-p-benzoquinone-3-diazohydroxide){called Anhydro-[6-nitro-2.5-dioxy-p-chinon-diazohydroxyd-(3)]; Anhydro-[6-nitro-4.5-dioxy-o-chinon-diazohydroxyd-(3)]; or 3-Diazid des 6-Nitro-cyclohexanpentons-(1.2.3.4.5) in Ger};



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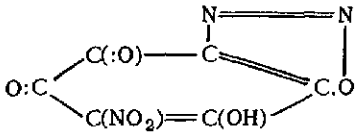

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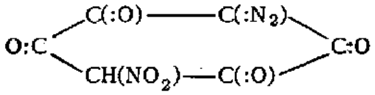


mw 211.09, N 19.91%; yel plts or ndls + $4H_2O$ (from w + concd HCl under cooling), mp-70°, dec above 100° & deflgr on rapid heating; mod sol in cold w & alc; sl sol in eth, eth acet & glac acet ac; insol in common org solvs; can be prepd by treating 6-nitro-3-amino-1,2,4,5-tetroxybenzene with $NaNO_2$ & HCl, aq HNO_3, or nitrosylsulfuric acid. Its *Sodium salt*, $NaC_6N_3O_6 + 2H_2O$, yel ndls (from w at 50°) explodes violently on heating the dry material (Refs 1 & 2)
Refs: 1) Beil **16**, 542 2) R. Nietzki & (?).Benckiser, Ber **18**, 501(1885) 3) No later Refs found

Di(hydroxybenzyl)-peroxide. See Bis(α-hydroxybenzyl)-peroxide in Vol 2, p B145-L

Dihydroxybiphenyl or Diphenol and Derivatives. See Biphenol and Derivatives in Vol 2, p B122-L

1,1'-Dihydroxy-(5,5'-bistetrazole). See 5,5'-Bis(1-hydroxytetrazole) in Vol 2, p B148-L

Dihydroxybitolyl or Dihydroxydimethylbiphenyl. See Bicresol in Vol 2, p B112-R

Dihydroxybutane or Butyleneglycol. See Butanediol in Vol 2, p B368-R

Dihydroxy-iso-butane or iso-Butyleneglycol. See iso-Butanediol in Vol 2, p B370-L

Di(hydroxycyclohexyl) -peroxide or Dihydroxydicyclohexyl-peroxide. See Bis(1-hydroxycyclohexyl) -peroxide in Vol 2, p B145-R

Dihydroxydiazapentane. Same as Dihydroxytrimethylene-diamine

Dihydroxydiethyl-amine. Same as Diethanolamine or Diethylol-amine

Dihydroxydiethyl-nitramine. See under Diethylolamine and Derivatives

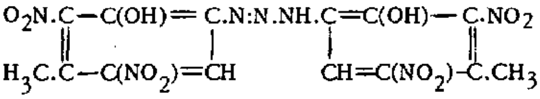
Dihydroxydiethyl-oxamide. See Diethylol-oxamide and Derivatives

Dihydroxy-di(hydroanthracene). See Bis-(hydroanthranol) in Vol 2, p B144-L

Dihydrodiketobenzene. One of the names for Benzoquinone which is described in Vol 2, p B79-R

Dihydroxydimethyldiazoaminobenzene or Di(hydroxytoluene)-triazene. Its tetranitro deriv:

2,2'-Dihydroxy-4,4'-dimethyl-3,5,3',5'-tetranitrodiazoaminobenzene (called 3.5.3'.5'-Tetranitro-2.2'-dioxy-4.4'-dimethyldiazoaminobenzol in Ger),



$$\begin{array}{l} O_2N.C{-}C(OH){=}C.N{:}N.NH.C{=}C(OH){-}C.NO_2 \\ H_3C.C{-}C(NO_2){=}CH \qquad CH{=}C(NO_2){-}C.CH_3 \end{array}$$

mw 437.28, N 22.42%; golden-yel plates, mp-explodes violently on heating to 160°; was prepd by treating an alcoholic soln of 2,6-dinitro-4-amino-3-hydroxy-1-methylbenzene with nitrous acid (Refs 1 & 2)

Refs: 1) Beil **16**, 720 2) O. Emmerling & A. Oppenheim, Ber **9**, 1095(1876) 3) Not found in later Refs

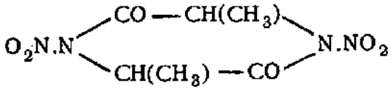
Dihydroxydimethylethylmethane or 2,2-Dimethyl-1,3-propanediol, $(CH_3)_2C(CH_2OH)_2$ (Ref 1). Its dinitrate:

2,2-Dimethyl-1,3-propane Dinitrate (called Dimethyloldiethylmethane Dinitrate in CA), $(CH_3)_2C(CH_2.ONO_2)_2$; mw 194.15, N 14.43%; lt-yel liquid explosive; was prepd by Spaeth (Ref 2) by nitrating 2,2-dimethyl-1,3-propanediol with mixed acid at 24-40°. It was claimed to be suitable for use with NC & NG (Refs 1, 2 & 3)

Refs: 1) Beil **1**, 483, (251), [550] & {2199} 2) C.P. Spaeth, USP 1883045(1933) & CA **27**, 845-46(1933) 3) M.S. Fishbein, VoennayaKhim **1933**, No 6, 3-8; ChemZentr **1934** II, 1074-75 & CA **29**, 7077(1935)

Dihydroxydimethyldioxypiperazine. Its dinitro deriv:

1,4-Dinitro-3,6-dioxy-2,5-dimethylpiperazine,



mw 232.16, N 24.14%; ndls (from MeOH or chlf), mp-dec 136°; readily sol in hot chlf; sol in eth; almost insol in cold w; dec in hot w; was prepd from 3,6-dioxo-2,5-dimethylpiperazine by reaction of abs HNO_3 in the presence of acetic anhydride

Refs: 1) Beil **24**, 299 2) Not found in later Refs

Dihydroxydimethyl-oxamide. See Di(methylol)-oxamide

1,1'-Dihydroxy-5,5'-ditetrazolyl-tetrazole. See 5,5'-Bis(1-hydroxytetrazole) in Vol 2, p B148-L and in the paper of F.R. Benson, ChemRevs **41**, 16(1947)

Di(hydroxyethoxy)-benzene or Di(hydroxyethyl)-phenylene Ether. See Bis(hydroxyethoxy)-benzene in Vol 2, p B145-R

1,3-Di(2-hydroxyethoxy)-2,4,6-trinitrobenzene Dinitrate, listed in CA's as **2,4,6-Trinitro-1,3-bis(2-hydroxyethoxy)-benzene Dinitrate.** See 1,3-Bis(2-hydroxyethoxy)-2,4,6-trinitrobenzene Dinitrate in Vol 2, p B146-L

Di(hydroxyethyl)-amine. See Diethylol-amine

Di(hydroxyethyl)-ether. See Diethyleneglycol

Di(hydroxyethyl)-oxamide. See Diethylol-oxamide

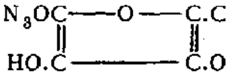
Di(hydroxyethyl)-peroxide. See Diethylol-peroxide

Di(hydroxyethyl)-phenylene Ether. See Bi-(hydroxyethoxy)-benzene in Vol 2, p B145-R

Di(hydroxyethyl)-piperazine or Diethylol-piperazine. See N,N'-Bis(2-hydroxyethyl)-piperazine in Vol 2, p B146-R

Di(hydroxyethyl)-sulfamide. See Diethylol-sulfamide

3,4-Dihydroxy-2,5-furandicarbonyl Diazide or 2,5-Bis(azidodicarbonyl)-3,4-dihydroxyfuran



$$\begin{array}{lcl} N_3OC & —O— & C.CON_3 \\ \| & & \| \\ HO.C & ——— & C.OH \end{array}$$

; mw 238.12, N 35.30%; cryst, mp 148-50° (explodes); was obtd when the salt, $C_8H_8O_7.2N_2H_4$(mp 168°), resulting from reaction of the ester dimethyl-3,4-dihydroxyfuran-2,5-dicarboxylate with two parts $N_2H_4.H_2O$ in alc, was treated with $NaNO_2$ in dil HCl (Ref 2). This compd was also patented by Priest & Vanallen (Ref) for use in radiation-sensitive materials. No expl props are reported

Refs: 1) Beil-not found 2) A. Darapsky & M.Stauber, JPraktChem **146**, 209-18(1936) & CA **31**, 396(1937) 3) W.J. Priest & J.A. Vanallen, BritP 956336(1964) & CA **61**, 16026(1964)

Dihydroxylaminobenzene and Derivatives

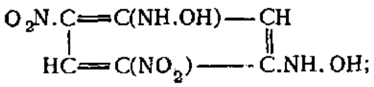
Dihydroxylaminobenzene (called Dihydroxylamino-benzol or Phenylenedihydroxylamin in Ger), $C_6H_4(NH.OH)_2$, may be considered as the parent compd of its derivs:

1,4-Di(nitrosohydroxylamino)-benzene (called 1.4-Bis-nitrosohydroxylamino-benzol or "p-Dinitroso-phenylen-dihydroxylamin" in Ger),

HO.N(NO).C_6H_4.N(NO).OH or
HO.N:N(:O).C_6H_4.N(:O):N.OH;

known only in the form of salts (Refs 1 & 2)
Disodium Salt, $Na_2C_6H_4N_4O_4 + H_2O$, reddish yel, deflagrates >250° (See also Ref 3)
Ferric Salt, dk-red ppt, explodes at 215°; almost insol in acet & pyridine
Silver Salt, not explosive
Refs: 1) Beil **16**, [347] 2) D. Bigiavi & F. Franceschi, Gazz **57**, 376(1927) & CA **21**, 3049(1927) 3) B.J. Danzig et al, JOChem **25**, 1071(1960) & CA **54**, 20827 (1960)

4,6-Dinitro-1,3-dihydroxylamino-benzene [called 4.6-Dinitro-1.3-dihydroxylamino-benzol or 4.6-Dinitro-phenylen-(1.3)-dihydroxylamin in Ger),



$$\begin{array}{lcl} O_2N.C & = C(NH.OH) — & CH \\ | & & \| \\ HC & = C(NO_2) —— & C.NH.OH; \end{array}$$

mw 230.14, N 24.35%, brn-yel ndls + H_2O (from dil hot alc), mp-decomposes explosively at 184°, stable for months when pure; when allowed to stand for days at RT in dil alc, it separates in thick brick-red tablets, mp 172°(decomp), changing back into yel form on recrystn from dil alc; sol in dil NaOH or NH_4OH with deep red-brn color; can be prepd by boiling 4,6-dinitroresorcin-diphenylether, 3,4,6-$HONH(O_2N)_2C_6H_2OC_6H_5$, in alc with hydroxylamine, NH_2OH, filtering, evaporating and again boiling in alc with hydroxylamine (Refs 1 & 2)

Refs: 1) Beil **15**, [22] 2) W. Borsche & E. Feske, Ber **59B**, 819(1926) & CA **20**, 2667(1926) 3) No later refs found to 1966

NOTE: No higher nitrated derivs of Dihydroxylaminobenzene were found to 1966

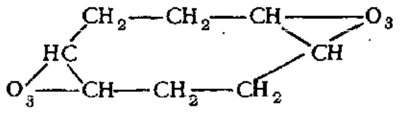
Dihydroxylaminomethane, $CH_2(NHOH)_2$, may be considered the parent compd of dinitroso deriv, although not found in the literature nor used to prep it:

N,N'-Dinitroso-N,N'-dihydroxylamino-methane or N,N'-Dinitroso-N,N'-dihydroxy-methanediamine (called Bis-nitrosohydroxylamino-methan, Methylen-bis-nitrosohydroxylamin or Methylendiisonitramin in Ger), $CH_2[N(NO).OH]_2$ or $CH_2[N(:O):N.OH]_2$; mw 136.07, N 41.18%; was obtd when nitric oxide was passed into an alcoholic soln of acetone to which NaOH or Na ethoxide was added; by reaction of NO on Na isopropylate or Na-α-phenylethylate soln; and by other methods (Ref 1); known free only in soln; UV & IR spectra (Ref 5)

Its salts are explosive. Urbański (Ref 4) prepd many salts and determined their decreasing order of expl effect, when primed with MF in the Lead Plate Test, as follows: Tl, Sn, Na, K, Fe, Cu, Cd & Ba. Von Herz (Ref 2) patented the use of some of these metallic salts in mixts with Pb Picrate, Pb Styphnate, and others as compns for detonators & percussion caps

Refs: 1) Beil **1**, 592, [651] & {2608} 2) E. von Herz, USP 1625966(1927) & CA **21**, 2065(1927); GerP 424380(1926) 3) H. Wieland et al, Ber **61B**, 2382-87 (1928) & CA **23**, 1385(1929) 4) T. Urbański, IX CongrInternQuimPuraAplicada **4**,438-46(1934) & CA **30**, 3649(1936) 5) M. Piskorz & T. Urbański, BullAcadPolon, SerSciChim **11**(11), 607-13(1963) (English) & CA **60**, 12791(1964)

m-Dihydroxylone Diozonide (called Diozonid des α-Cyclooctadiens in Ger),



mw 198.13, O 48.45%, col transparent solid, explodes on heating; d 1.284; diffc sol in org solvents; sol in dil NaOH giving a brn color; obtd by action of ozone on α-cyclooctadien in CCl_4 soln (Refs 1 & 2)

Refs: 1) Beil **5**, 117 & (62) 2) C. Harries & H. Neresheimer, Ber **39**, 2846-50 (1906) & CA **1**, 50(1907) 3) No later refs found thru 1966

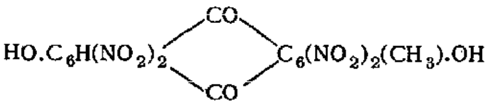
Dihydroxymethylanthraquinone and Derivatives

1,8-Dihydroxy-3-methylanthraquinone, Chrysophanic Acid, Methylchrysazin, Chrysophanol (called 4,5-Dioxy-2-methyl-anthrachinon; 3-Methyl-chrysazin; Chrysophanol; or Chrysophansäure in Ger), $HO.C_6H_3(CO)_2C_6H_2(CH_3).OH$; mw 254.23, O 25.17%, golden yel pltlts (from alc, benz or petr eth), mp 194-96°, bp-sublimes & explodes; can be prepd by diazotizing 5-amino-4-hydroxy-2-methylanthraquinone in concd H_2SO_4 and warming the soln to 120°; or by heating 4-hydroxy-5-methoxy-2-methylanthraquinone with acetic acid & aq HBr; and by many other methods listed in Beil (Ref 1)

Bedier (Ref 2) patented the use of this compd in an expl consisting of $KClO_3$ 736, sugar 480, hard wheat bolted flour 320, chrysophanic acid 2 & curcuma (coloring material) 1 part

Refs: 1) Beil **8**, 470, (725), [510] & {3808} 2) V.L. Bedier, USP 981969(1911) & CA **5**, 1190(1911)

1,8-Dihydroxy-3-methyl-2,4,5,7-tetranitro-anthraquinone or 2,4,5,7-Tetranitro-3-methyl-chrysazin (called Tetranitrochrysophanol or Tetranitrochrysophansäure in Ger),



$$HO.C_6H(NO_2)_2\begin{matrix}CO\\ \\ CO\end{matrix}C_6(NO_2)_2(CH_3).OH$$

mw 434.23, N 12.90%; long orn ndls (from acet acid), mp-decomp at 300°; sol in glac acet acid; almost insol in cold w; can be prepd by nitration of chrysophanol with excess concd HNO_3; forms red crystalline salts of K, Mg & Ca (Refs 1 & 3). On heating with HNO_3(d 1.5) the Tetranitro compd gives 2,4,6-Trinitro-m-hydroxybenzoic Acid (Ref 2). No expl props of the Tetranitro compd were reported

Refs: 1) Beil **8**, 473 & (726) 2) E. Leger, CR **154**, 281–83(1912) & CA **6**, 1132(1912) 3) F.E. King et al, JChemSoc **1952**, 4580 & CA **48**, 1308(1954)

NOTE: No higher nitrated derivs of Chrysophanic Acid were found in the literature thru 1966

Di(hydroxymethyl)-oxamide. See Dimethylol-oxamide in this vol

Di(hydroxymethyl)-peroxide. See Dimethyl-peroxide in this vol

Dihydroxynaphthalene and Derivatives

Dihydroxynaphthalene or Naphthalenediol (called Dioxy-naphalin, Naphthohydrochinon or Hydronaphthoquinon in Ger), $HO.C_{10}H_6.OH$; mw 160.16, O 19.98%. All of the isomers as parent compds of their nitro derivs are found in Beil

Refs: Beil **6**, 975, 978, 984, 985, (468, 474, 480, 482), [944, 948, 950, 952-56] & {5283}

Dinitrodihydroxynaphthalene, $HO.C_{10}H_4(NO_2)_2.OH$; mw 250.16, N 11.20%. The following derivs are described in Beil:

1,8-Dinitro-2,7-dihydroxynaphthalene, yel crysts (from anisole), mp-dec at 250° (Ref 1)

2,4-Dinitro-1,5-dihydroxynaphthalene, golden-brn ndls (from acet acid), mp-dec 247° (Ref 2, p 5273)

2,6-Dinitro-1,5-dihydroxynaphthalene, orange colored pltlts (from acetic acid), mp-dec 246° (Ref 2, p 5274)

2,4-Dinitro-1,8-dihydroxynaphthalene, red prisms (from MeOH or H_2O), mp-dec 180-82° (Ref 2, p 5284)

4,5-Dinitro-1,8-dihydroxynaphthalene, brn prisms (from MeOH), mp-dec at 225° (Ref 2, p 5285)

2,4-Dinitro-1,6-dihydroxynaphthalene, yel threads (from 50% alc), mp-dec 220° (Ref 3)

1,5-Dinitro-2,6-dihydroxynaphthalene, crysts (from benz), mp-darkens at 200°, dec at 212° (Ref 4)

Their methods of prepn and other props are given in the Refs

Refs: 1) Beil **6**, 987 2) Beil **6**, {5273, 5274, 5284, 5285} 3) S.V. Bogdanov & Z.V. Todres-Selektor, ZhVsesKhimObshch im D.I. Mendeleeva **7**, No 1, 118-19(1962) & CA **57**, 16510(1962) 4) P. Heinz & G. Zimmer, JPraktChem **18**, 219-24(1962) & CA **58**, 11289(1963)

2,4,6-Trinitro-1,5-dihydroxynaphthalene, $HO.C_{10}H_3(NO_2)_3.OH$; mw 295.16, N 14.24%; yel ndls (from aq acet ac), mp-dec 260°; was prepd by heating 2,4,6-trinitro-5-acetoxy-1-naphthole or 2,6,8-trinitro-5-methoxy-1-acetoxynaphthalene with 2% NaOH (Refs 1 & 2). No expl props are reported

Refs: 1) Beil **6**, {5275} 2) R.H. Thomson et al, JCS **1947**, 350, 353 & CA **41**, 5495 (1947)

2,4,6,8-Tetranitro-1,5-dihydroxynaphthalene, $HO.C_{10}H_2(NO_2)_4.OH$; mw 340.16, N 16.47%; yel ndls (from acet), mp-decomp violently between 250-65° depending on rate of heating; was prepd by nitrating 2,4-dinitro-5-acetoxy-1-naphthol with HNO_3(d 1.5) at 0°, or by direct nitration of 1,5-dihydroxynaphthalene in acetic anhydride by diacetyl orthonitric acid in acet anhyd at 0° during 1½hrs (Refs 1, 2 & 3). No expl props were reported

Refs: 1) Beil **6**, {5275} 2) R.H. Thomson et al, JChemSoc **1947**, 350, 353 & CA **41**, 5495(1947) 3) D.C. Morrison & D.W. Heinritz, JOrgChem **27**, 2229-31(1962) & CA **57**, 5855(1962)

Tetranitro-2,7-dihydroxynaphthalene, $HO.C_{10}H_2(NO_2)_4.OH$; mw 340.16, N 16.47%; yel crysts (from acetic acid), mp 227° with expl decompn; was prepd by adding dropwise fuming HNO_3 to 2,7-dihydroxynaphthalene in acetic acid (Ref 2). No other expl props were reported

Refs: 1) Beil **6**, not found 2) F. Bell & J.R. Gorrie, JChemSoc **1961**, 4259 & CA **56**, 2392(1962)

NOTE: No higher nitrated derivs of Dihydroxynaphthalene were found in the literature thru 1966

Dihydroxypentamethylene-tetramine. See its nitrated derivs 1,9-Dinitroxy-1,3,5,7,9-pentamethylene-2,4,6,8-tetranitramine and 1,8-Dinitroxy-1,3,5,6,8-pentamethylene-2,4,7-trinitramine in this Vol

Di(hydroxyphenyl)-anthranone and its **Hexanitrate.** See under Bis(hydroxyphenyl)-anthranone and Derivatives in Vol 2, p B147-L

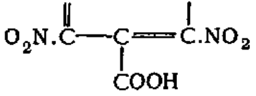
Di(hydroxyphenyl)-dimethyl-methane. See Di(phenylol)-dimethyl-methane in this Vol

Di(hydroxyphenyl)-propane and its **Dinitro Compound.** See under Bis(hydroxyphenyl)-propane in Vol 2, p B147-R

Di(hydroxyphenyl)-sulfone and its **Nitro Compound.** See under Bis(hydroxyphenyl)-sulfone and Derivatives in Vol 2, p B148-L

Dihydroxypyridinecarboxylic Acid, $C_6H_5NO_4$, mw 155.11, N 9.03 %. Several isomers are described in Beil (Ref 1). The 2,6-Dihydroxypyridine-4-carboxylic Acid is the most important (Ref 1, p 254). Its dinitro deriv is expl:

3,5-Dinitro-2,6-dihydroxypyridine-4-carboxylic Acid [called 3.5-Dinitro-2.6-dioxy-pyridin-carbonsäure-(4); 3.5-Dinitro-2.6-dioxy-isonicotinsäure; or Dinitrocitrazinsäure in Ger],



mw 245.11, N 17.14%; yel ndls + $2H_2O$ (from acet acid), mp-explodes at 115-20°; sol in alc & w; sl sol in benz & petr eth; was prepd by nitrating 5-nitro-2,3,4,6-tetroxo-piperidine with HNO_3 (d 1.42) (Refs 2 & 3)

Refs: 1) Beil **22**, 251, 253, 254, (562) & [190] 2) Beil **22**, 258 3) (?). Sell & (?). Easterfield, JChemSoc(London) **65**, 831, 833(1894) 4) No later refs found in the literature thru 1966

Dihydroxyquinone. See Dihydroxybenzoquinone in this Vol

Dihydroxytetramethylenetriamine or Dihydroxytriazaheptane,
$HO.CH_2.NH_2.CH_2.NH_2.CH_2.NH_2.CH_2.OH$, may be considered as the parent compound of its nitrated deriv although not used to prep it

1,7-Dinitroxy-1,3,5,7-tetramethylene-2,4,6-triamine; 1,7-Dinitroxy-2,4,6-trinitro-2,4,6-triazaheptane; or 2,4,6-trinitro-2,4,6-triazaheptanediol-1,7-dinitrate (Code name ATX & NBSX),
$O_2NO.CH_2.N(NO_2).CH_2.N(NO_2).CH_2.N(NO_2)-.CH_2.ONO_2$; mw 360.16, N 31.10%; crysts, mp 153-54°(dec), sol in 1/1 dioxane/CCl_4 mixt; insol in w; was obtd by nitrolysis of 1,7-diacetoxy-2,4,6-trinitro-2,4,6-triazaheptane at 20°(Refs 3,4 & 7)

ATX is a powerful expl very sensitive to impact. It was claimed in Germany to be more powerful than RDX as judged by Trauzl Lead Block Test (Ref 2)

Refs: 1) Beil-not found 1a) L. Pauling, OSRD Rept **5953**(1945) (Absorption spectra) 2) G. Römer, "Reports on Explosives (Germany), PBL Rept 85160(1946) 3) W.J. Chute et al, CanJRes **27B**, 513-14(1949) & CA **43**, 9074(1949) 4) K.W. Dunning & W.J. Dunning, JChemSoc **1950**, 2925-28 & CA **45**, 6643(1951) 5) W.A. Schroeder et al, AnalChem **23**, 1740-47(1951) & CA **46**, 5434(1952) (UV & visible spectrum) 6) E.W. Malmberg et al, AnalChem **25**, 901(1953) & CA **47**, 12095(1953) (Chromatographic estimation as an impurity in RDX) 7) R. Reed Jr, JACS **78**, 801-04(1956) & CA **50**, 12864(1956) 8) Not found in later refs thru 1966

Dihydroxytetrazanonane or Dihydroxypentamethylenetetramine. See its nitrated derivs 1,9-Dinitroxy-1,3,5,7,9-pentamethylene-2,4,6,8-tetranitramine and 1,8-Dinitroxy-1,3,5,8-pentamethylene-2,4,7-trinitramine in this Vol

Dihydroxy-tetrazole. See Bis(hydroxytetrazole) in Vol 2, p B148-L

Di(1-hydroxy-tetrazolyl-5), called by Benson **1', 1-Dihydroxy-5',5-ditetrazolyl.** See 5,5'-Bis(1-hydroxytetrazole) in Vol 2, p B148-L and in F.R. Benson, ChemRevs **41**, 16(1947)

Dihydroxytoluene and Derivatives

Dihydroxytoluene (called Dioxy-methyl-benzol or Dioxy-toluol in Ger), $CH_3.C_6H_3(OH)_2$; mw 124.13, O 25.78%. All of the known isomers are found in Beil

Ref: Beil **6**, 872, 874, 878, 882, 896, 897, (426, 428, 431, 437, 439), [858, 859, 861, 865, 875, 877, 881, 882] & {4492, 4495, 4512, 4514, 4531}

Mononitrodihydroxytoluene, $CH_3.C_6H_2(NO_2)(OH)_2$; mw 169.13, N 8.28%. The known isomers are found in Beil

3-Nitro-2,4-dihydroxytoluene, red ndls (from dil alc), mp 111-12° (Ref 1)

5-Nitro-2,4-dihydroxytoluene, orn-yel ndls (from gasoline-benzene), mp 125°(Ref 1)

6-Nitro-2,5-dihydroxytoluene, scarlet red ndls (from eth + petr eth), mp 117-18°(Ref 2)

X-Nitro-2,5-dihydroxytoluene, yel-brn ndls (from petr eth) or yel-red leaflets (from dil alc), mp 122-24° (Ref 3)

5-Nitro-3,4-dihydroxytoluene, golden-yel pltlts (from w) or ndls (from petr eth), mp 82-83° (Ref 4)

6-Nitro-3,4-dihydroxytoluene, lt-yel ndls (from benz or w), mp 180-82° (dec)(Ref 5)

2-Nitro-3,5-dihydroxytoluene, yel ndls + H_2O (from w) or brn crysts + C_6H_6 (from benz), mp - becomes free of solvents at 122°; forms crystalline salts (Ref 6)

4-Nitro-3,5-dihydroxytoluene, orn-colored ndls (from alc), mp 127°; forms salts (Ref 6)

Other props & methods of prepn are found in the Refs

Refs: 1) Beil **6**, [861] & {4496} 2) Beil **6**, 876 3) Beil **6**, 877 4) Beil **6**, 881 & [868] 5) Beil **6**, 881, (433) & {4522} 6) Beil **6**, 889

Dinitrodihydroxytoluene, $CH_3.C_6H(NO_2)_2(OH)_2$; mw 214.13, N 13.08%; The known isomers are as follows:

3,5-Dinitro-2,4-dihydroxytoluene, yel ndls (from aq alc), mp 90° (Ref 1, p873)

4,6-Dinitro-2,5-dihydroxytoluene, yel-brn prisms + H_2O (from w or 50% alc) or yel tablets (from chlf) or orn-yel ndls (from benz), mp 149-53° (Ref 1, p877)

2,6-Dinitro-3,4-dihydroxytoluene, yel prisms (from w), mp-dec 172° (Ref 2)

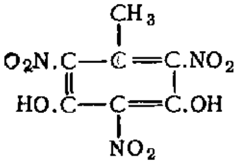
2,4-Dinitro-3,5-dihydroxytoluene, yel pltlts (from alc), mp 164.5°; forms a Ba salt (Ref 1, p890)

X,X-Dinitro-X,X-dihydroxytoluene, golden-yel ndls (from alc), mp 109-110° (Ref 1, p891)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **6**, 873, 877, 890, 891 2) Beil **6**, [871]

2,4,6-Trinitro-3,5-dihydroxytoluene (called 2.4.6-Trinitro-3.5-dioxy-1-methyl-benzol or *eso*-Trinitro-orcin in Ger),



mw 259.13, N 16.22%; yel ndls + $2C_6H_6$ (from benz) or lemon-yel rodlets (from MeOH), mp 171-72° with weak explosion; was prepd by heating 2,4,5,6-Tetranitro-m-cresol (See Vol 3, p C558-L) with water; readily sol in hot w, hot benz; sl sol in cold w & ether; forms salts such as $K_2C_7H_3N_3O_8$, orn ndls; $Ag_2C_7H_3N_3O_8$; $BaC_7H_3N_3O_8 + 3H_2O$, yel ndls; $PbC_7H_3N_3O_8$, yel ndls; and with naphthalene $C_7H_5N_3O_8 + C_{10}H_8$, yel ndls, mp 120° (Refs 1 & 2)

No expl props were reported for the trinitro compd

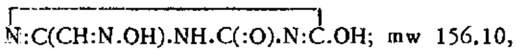
Refs: 1) Beil **6**, 890 2) H. Musso & H. Beekem, ChemBer **92**, 1416-22(1959) & CA **54**, 397(1960)

Di(hydroxytoluene)-triazene. Its tetranitro deriv, 3,5,3',5'-Tetranitro-2,2'-dihydroxy-4,4'-dimethyl-dibenzene-triazene is described in this Vol under Dihydroxydimethyldiazoaminobenzene

Dihydroxy-triazaheptane. See Dihydroxytetramethylene-triamine

Dihydroxytriazaoctane. Same as Dihydroxypentamethylene-triamine

Dihydroxytriazinylformaldoxime, Dihydroxytriazinylmethanol Oxime or 3,4-Dihydro-6-hydroxy-4-oxo-s-triazine-2-carboxaldehyde Oxime (CA Nomenclature).



$\overline{N:C(CH:N.OH).NH.C(:O).N:C}.OH$; mw 156.10, N 35.89%. This compd and many of its salts were prepd and described by Ostrogovich & Crasu (Refs 2 & 3). The salts and their description are too many & detailed to list here. The hydrated mono-Ba salt, $(C_4H_3N_4O_3)_2Ba.3H_2O$, honey-yel crysts, becomes orange-ochre when heated on a Pt foil and decomposes violently with a "Pharoah's serpent" effect

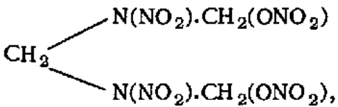
Refs: 1) Beil - not found 2) A. Ostrogovich & V. Crasu, JGenChem (USSR) **4**, 629-31 (1934) & CA **29**, 2168 (1935) 3) Ibid, GazzChimItal **66**, 653-62 (1936) & CA **31**, 3488 (1937) 4) Not found in later Refs

Di(hydroxytrichloroethyl)-peroxide. See Bis(α-hydroxy-β,β,β-trichloroethyl)-peroxide in Vol 2, p B148-R

Dihydroxytrimethylenediamine or Dihydroxydiazapentane,

$OH.CH_2.NH_2.CH_2.NH_2.CH_2OH$, may be considered as the parent compd of its nitrated deriv, although not used to prep it:

1,5-Dinitroxytrimethylene-2,4-dinitramine or 1,5-Dinitroxy-2,4-diazapentanediol-1,5-dinitrate



$CH_2\left\langle\begin{matrix}N(NO_2).CH_2(ONO_2)\\N(NO_2).CH_2(ONO_2)\end{matrix}\right.$, mw 382.13, N 22.00%, mp 98–101°. This compd was prepd during WWII in GtBritain by nitrating cyclonite oxide (See Vol 3, p C601-R). It is described in British Rept **AC 4628**, which was secret

This compd can also be prepd from MEDNA (methylenedinitramines)

Diimidazole. See Biimidazole in Vol 2 of Encycl, p B115-L

Diimidazoline. See Biimidazoline in Vol 2, p B115-R

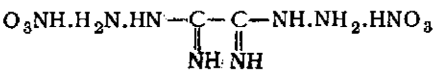
Diiminooxalic Acid Dihydrazide & Derivitives

Diiminooxalic Acid Dihydrazide,

$H_2N.HN{-}C(:NH){-}C(:NH).NH.NH_2$, mw 116.13, N 72.37%; ndls (from dil alc), mp >250°; mod sol in w; sl sol in alc; insol in eth. It was prepd by Curtius & Dedichen (Ref 2) on interaction of cyanogen $(CN)_2$ with 2 moles of hydrazine hydrate

Some of its salts are explosive, such as:

Diimino-oxalic Acid Dihydrazide Dinitrate,

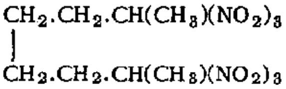


$O_3NH.H_2N.HN{-}\underset{NH}{\overset{}{C}}{-}\underset{NH}{C}{-}NH.NH_2.HNO_3$,

mw 242.14, N 46.28%; lt yel crysts, mp dec at 147–149° with evoln of gas; detonates when struck with a hammer, but only gasses vigorously when heated in a flame or dropped on a hot plate. Was prepd at US Rubber Co Laboratory, Passaic, NJ (Ref 3) by adding concd nitric acid to an aqueous soln of diiminooxalic acid hydrazide, cooling to 0° and adding ethanol. A more detailed description of prepn is given on p34 of Ref 3

Its **Silver salt,** $C_2H_8N_6 + 2AgNO_3$, explodes when heated above 100° (Ref 1, p 1594)

Diimino-oxalic Acid-bis(4,4,4-trinitro-3-methylbutylidene) Dihydrazide,



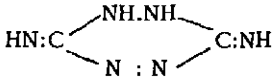
$$\begin{matrix}CH_2.CH_2.CH(CH_3)(NO_2)_3\\|\\CH_2.CH_2.CH(CH_3)(NO_2)_3\end{matrix}$$

mw 526.40, N 31.93%; burned quite vigorously, but could not be detonated by a hammer. Was prepd at US Rubber Co Lab (Ref 4)

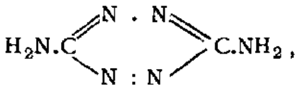
Refs: 1) Beil **2**, 560 & {1594} 2) T. Curtius & G. Dedichen, JPraktChem **50**(2), 254(1894) & 52(2), 272(1895) 3) Anon, USRubberCoQuartRept **No 3**, Apr 30 to July 31, 1948, Contract Nord 10129, pp 33–4 4) *Ibid*, Rept **No 5**, November 1, 1948 to 30 January 1949, pp44–5

1,4-Diimino-p-quinone. See Benzoquinone-1,4-diimine in Vol 2 of Encycl, p B83-L

[3,6-Diimino-1,2,3,6-tetrahydro]-sym-tetrazine or 3,6-Diamino-sym-tetrazine,



or



mw 112.10, N 74.98%; violet leaflets (from ammoniacal water), mp – expl ca 120°; may be prepd by evaporating an aq soln of aminoguanidine hydrochloride with KOH soln, while bubbling air thru the soln

Forms salts, some of which are explosive, eg:

Hydrobromide, yel lfts, expl at ca 120°

Picrate, yel amorphous pdr; expl

Refs: 1) Beil **26**, (130) 2) G. Ponzio & G. Gastaldi, Gazz **43**(II), 132(1913) 3) N. Mataga, BullChemSocJapan **36**(12) 1607–18(1963) & CA **60**, 8773(1964) (Electronic spectrum & electronic structure)

Diiodoacetylene or Diiodoethyne, IC:CI, mw 277.86; col crysts, mp 78.5°, bp decomp >80°; extremely toxic. Can be prepd by dissolving iodine in liq NH_3 and passing acetylene into the soln; explodes by frictional forces, deflagrates on heating (Ref 1)

Refs: 1) Beil **1**, 246, (106) [222] & {919}
2) CondChemDict (1961), p 386-R

Diiododiacetylene or Diiodobutadiyne, IC:C.C:CI, mw 309.86; crysts, mp – explodes violently on heating, by friction & by impact; was obtd by reaction of Ag diacetylene with an aq soln of iodine in KI (Ref 2) and by other methods (Ref 1)

Refs: 1) Beil **1**, 266, [246] & {1057}
2) A. Baeyer, Ber **18**, 2276(1895)

Diiododiphenyl Diazide, Diazidobiphenyl Diiodide or Ditriazobiphenyl Diiodide, $(N_3).I.C_6H_4-C_6H_4.I(N_3)$, mw 490.07, N 17.15%. Exists in three isomers:

ortho: crysts, mp 166°; practically insol in all solvs; chromate of o- compd explodes at 168° (Ref 2)

meta: crysts, mp 171°; same props as ortho (Ref 2)

para: crysts, mp 174°; same props as ortho (Ref 2)

Above isomers obtd when molecular proportions of iodsophenylazide, $N_3.C_6H_4.IO$, and iodoxyphenylazide, $N_3C_6H_4.IO_2$, with calcd amt of Ag oxide were made into a paste with water and shaken 3–4 hrs. The filtered liq, having been saturated with S dioxide, was treated with K iodide, which pptd the colorless products (Ref 2)

Refs: 1) Beil – not found 2) M.O. Forster & J.H. Schaeppi, JCS **101**, 1365–66 (1912) 3) Not found in later refs thru 1966

Diiodofumaric Acid (Dijodfumarsäure in Ger) (Called Acetylencarbonsäure by Bruck) HOOC.CI:CI.COOH, mw 303.90; ndls (from eth + benz) or crysts (from eth + toluene), mp – dec at 192°; sol in w, alc, eth, acet acid; insol in benz, chlf & petr eth; prepd by heating acetylene dicarboxylic acid with iodine in alc or in chlf at 100°

Its *silver salt*, $Ag_2C_4O_4I_2$, puffs off ca 140°

Refs: 1) Beil **2**, 747 & [641] 2) P. Bruck, Ber **24**, 418–19(1891) and **26**, 845 & 848(1893)

4,4'-Diiodosodiphenyl (called 4.4'-Dijodosodiphenyl in Ger), $OI.C_6H_4.C_6H_4.IO$; mw 438.02, chocolate-brn pwd, mp 127° & explodes at 198°; was obtd by reaction of Cl in a chloroform soln with 4,4'-diiododiphenyl & NaOH. The compd is insol in cold glac acet acid. Its salts are also expl

4,4'-di(iododichloro)-diphenyl, $Cl_2I.C_6H_4.C_6H_4.ICl_2$, red-brn unstable powd or yel amorph ndls, mp – dec at 154°

4,4'-Tetraacetyl Ester of Diiododiphenyl,

$(C_2H_3O_2)_2I.C_6H_4.C_6H_4.I(C_2H_3O_2)_2$, almost col crysts (from benz), mp 203°, but explodes on heating above its mp (Refs 1, 2 & 3)
Refs: 1) Beil **5**, 581 2) C. Willgerodt & G. Hilgenberg, Ber **42**, 3826–27(1909) & CA **4**, 209(1910) 3) A. Werner, JChemSoc **89**, 1633–34(1906) 4) Not found in later refs

4,4'-Diiodoxydiphenyl (Dijodo-diphenyl in Ger), $O_2I.C_6H_4.C_6H_4.IO_2$, mw 470.02, amorph solid, mp – explodes at 218°; crystn from acetic acid yields 4,4'-diiododiphenyl; prepd from the tetrachloride of 4,4'-diiododiphenyl by reaction with Na hypochlorite & boiling acetic acid (Refs 1 & 2)
Refs: 1) Beil **5**, 581 2) C. Willgerodt & G. Hilgenberg, Ber **42**, 3827(1909) & CA **4**, 209(1910) 3) Not found in later refs

Diisoalkylphthalates. The compds having 3 to 6 carbon atoms in the alkyl chain were patented by Cook as the deterrent coating on smokeless propellant grains. It was claimed that using 2 to 15% of such compds prevented deterioration of coatings caused by migration of the deterrent during storage. Tests showed that breech pressure increased after 3 months storage at 140°F, but was much less for proplnt grains coated with diisoalkylphthalate deterrent than those coated with n-DBuPh(n-dibutylphthalate)

One compd, Diisobutylphthalate, $C_6H_4(COO)_2[(CH_3)_2CH.CH_2]_2$, is an example of a coating deterrent for propellant grains
Ref: R.L. Cook, Assignee: Olin Mathieson Chem Corp, USP 2865728(1958), "Smokeless Powder" & CA **53**, 5678(1959)

Di(isobutylammonium)-triazo-copper. See under Di(butylamino) diazo- and triazo-copper compounds, p D1196-R in this Vol

Diisobutyl Ketone (2,6-Dimethyl-4-heptanone), $(H_3C)_2CH.CH_2.CO.CH_2.CH(CH_3)_2$, mw 136.19, col liquid, sp gr 0.8089 at 20/20°, bp 168.1°; flash p 140°F. Can be prepd by heating isopropyl alcohol over Zn-Cu-Cd-Cr catalyst at 360° (Ref 1). It is suitable as a solvent for NC (Ref 2)
Refs: 1) Beil **1**, 710, (366), [763] & {2892}
2) CondChemDict (1961), p 387-R

Diisobutylphthalate. See under Diisoalkylphthalate

Diisopropanolamine. See under Dipropylolamine and Derivatives

Diisopropanolamine Trinitrate. See under Dipropylolamine Trinitrate

Diisopropyl Ether. See under Dipropyl Ether

Diisopropyl-urea. See under Dipropyl-urea

Di(isoquinoline)-diazido-copper. See under Diquinolino-diazido-copper

Diketopiperazine and Derivatives

Diketopiperazine; 3,5-Dioxopiperazine; Glycine Anhydride; Cycloglycylglycin or Lactam of Glycylglycin

$HN.CO.CH_2.NH.CO.CH_2$, mw 114.10, N 24.55%; col plates, mp – subl 260°; bp dec 275° (Lange), 295° (Ref 4). Can be prepd either by methods given in Beil (Ref 1) or by dehydrating intramolecular glycine, $H_2N.CH_2.COOH$, on heating it in glycerol as described in Ref 3

On nitration it gives an expl dinitro compd and also forms salts, some of which are expl, as for example:

Silver Diketopiperazine, $Ag_2C_4N_2O_2H_4$; expl on heating (Ref 1, p 265)

1,4-Dinitrodiketopiperazine,
$(O_2N).N.CO.CH_2.N(NO_2).CO.CH_2$, mw 204.10, N 27.45%, OB to CO_2 –31.4%, white crysts, mp dec 143-147°; bp – deflagrates and burns quietly w/o expln. Can be prepd, accdg to direction of Franchimont & Friedmann (Ref 2), by nitrating diketopiperazine with a mixture of white nitric acid and acetic anhydride. The crude product can be purified by recrystallizing from

a mixture of ethyl acetate and chloroform. It is an expl comparable in sensitivity with TNT, but unstable in storage (decomposing in 48 hours on heating to 75°); hygroscopic and is hydrolized by water. May be considered unsuitable for military purposes (Ref 4)
Refs: 1) Beil **24**, 264–65, 269 and (295) 2) A.P.L. Franchimont & H. Friedmann, Rec **27**, 192 (1908) 3) Y. Shibata & T. Asahina, BullChemSoc(Japan) **1**, 71 (1926) & CA **20**, 2502 (1926) 4) R. Adams & C.S. Marvel, OSRD **86**(1941),, pp9–10, 27–28 & 56

Dilauroyl Peroxide *(Alperox C)* or **Dodecanoyl Peroxide,** $(C_{11}H_{23}CO)_2O_2$, mw 398.61, O 16.06%; active O 8.04%. The commercial product of the Lucidol Div, contains a minimum of 95.0% of dilauroyl peroxide with min active O 3.76%. It consists of wh soft granules with mp 48–50°; insol in w; sl sol in alcohols; sol in esters, vegetable oils and petr solvents; very sol in chlorobenzene, chlf, CS_2, CCl_4, toluene, dichloromethane, ethylenedichloride and trichloroethylene. Can be prepd by treating the appropriate acid chloride or anhydride with Na or H peroxide in the presence of a base (Ref 4)

Alperox C is stable at ord temp, but because of its rather low mp, it should be stored in a cool place; prolonged storage at temps close to or above its mp causes decompn

It burns upon ignition but without violent decompn characteristic of most organic peroxides; should not be subjected to friction, such as by grinding
Uses: Catalyst for the polymerization of synthetic resins; ingredient of rubber compounding and propellent compositions
Refs: 1) Beil – not found 2) Lucidol Division, Wallace & Tiernan Corp, "Organic Peroxides" Pamphlet (Data Sheet No 3), Buffalo, NY (1947) 3) CondChemDict (1961), p654 4) Kirk & Othmer, 2nd edit **14** (1967), p799

Dimazine. A code name for unsym-Dimethylhydrazine, described in this Vol under Dimethylhydrazine and Derivatives

Dimedone. See Dimethylcyclohexanedione, p D133-R

Dimensional Analysis is a method by which the variables characterizing a phenomenon may be related. Accdg to Eschbach (Ref 2), it is fundamentally identical with the analysis of physical equations, and in particular, with the analysis of physical differential equations. Methods of Lord Rayleigh and of E. Buckingham are used in ballistics, thermodynamics and fluid mechanics

Dimensional analysis is simply a mathematical tool. In all cases, it will reduce the number of experimental variables to be correlated, and often it will point out the best experimental approach to a problem. It will not give quantitative information, however; experiments must still be performed (Ref 3)
Refs: 1) D.Q. Kern, "Dimensional Analysis", pp 133–41 in Kirk & Othmer's, Vol **5**(1950) 2) O.W. Eshbach, "Handbook of Engineering Fundamentals", Wiley, NY (1952), pp133–42 3) J.H. Perry, "Chemical Engineers' Handbook", McGraw-Hill, NY (1963), pp2–87 to 2–90 4) Kirk & Othmer, 2nd Edit **7**(1965), 176–90

Dimeres Fluorenonperoxyd. A Ger name for 9,9'-Bis(fluorenyl)-diperoxide, described in Vol 2 of Encycl, p B143-L

Dimeric Acetone Peroxide. See Acetone – peroxide, Dimeric in Vol 1, p A41-R

Dimeric Cyclohexanone Peroxide. See Comp VI, under Cyclohexanone Peroxide in Vol 3, p C598-R

Dimethanol-amine. See Di(methylol)-amine

Dimethanol-benzene. See Di(methylol)-benzene

Dimethanol-ethylenediamine. See Di(methylol)-ethylenediamine

Dimethanol-methylmethane. See Di(methylol)-methylmethane

Dimethanol-oxazolidone. See Di(methylol)-oxazolidone

Dimethanol-peroxide. See Di(methylol)-peroxide

Dimethoxyaniline and Derivatives

Dimethoxyaniline, $(H_3CO)C_6H_3(NH_2)(OCH_3)$; mw 153.18, N 9.14%. Several isomers are found in the literature:
2,3-Dimethoxyaniline; oil, bp 137° at 10mm press (Ref 2)
2,4-Dimethoxyaniline, colorless oil, bp 75–80° at 6×10^{-3}mm pressure (Refs 2 & 5); was prepd from $2,4(CH_3O)_2C_6H_3COOCH_3$ by treating with NH_3 and oxidizing the amide by treating with hypochlorite in alk medium (Ref 7); and by hydrogenating 2,4-dimethoxynitrobenzene in acetic acid with palladium as catalyst
2,5-Dimethoxyaniline, wh crysts (sublimation), mp 74–76°; was obtd when 2,5-dimethoxybromobenzene reacted with diethyl malonate in the presence of a catalyst (Ref 6)
2,6-Dimethoxyaniline, crysts (from lt petr eth), mp 75.5–77°; was prepd by hydrogenating $2,6\text{-}(MeO)_2C_6H_3NO_2$ in alc over Raney Ni at 80° (Ref 3)
3,4-Dimethoxyaniline, crysts, mp 86°, bp 172–74° at 24mm press- prepd by treating a boiling soln of 4-nitroveratrole in alc contg Pd-C catalyst with $N_2H_4.H_2O$ (Ref 8)
3,5-Dimethoxyaniline, crysts, mp 46°, bp 115° at 0.5mm press; obtd by hydrolysis of $3,5\text{-}(MeO)_2C_6H_3NHAc$ with 15% alc NaOH by refluxing for 4 hrs (Ref 4)

Other props are given in the Refs

Refs: 1) Beil **13**, 780, (307) & [465] 2) Beil **13**, (305) & [464] 3) T. Ekstrand & N. Löfgren, ActaChemScand **6**, 1016(1952) (Engl) & CA **47**, 8028(1953) 4) F. Šorm & L. Novotný, ChemListy **49**, 901(1955) & CA **49**, 13244(1955) 5) J.D. Loudon & J. Ogg, JChemSoc **1955**, 742 & CA **50**, 4963 (1956) 6) B.A. McFadden & G.G. Still, JOrgChem **25**, 275(1960) & CA **54**, 14180 (1960) 7) S.N. Solodushenkov & V.N. Erikhov, RussP 137924(1960) & CA **56**, 8637(1962) 8) C. Alberti & C. Tironi, Farmaco (Pavio) EdSci **19**(5), 459(1964) & CA **61**, 5633(1964)

Mononitrodimethoxyaniline, $(H_3CO)C_6H_2(NH_2)(NO_2)(OCH_3)$; mw 198.18, N 14.14%. The following isomers are found in the literature:
4-Nitro-2,3-dimethoxyaniline (called 3-Amino-6-nitroveratrole by Bell & Millar), prisms (from chlf-lt petroleum), mp 93° (Ref 5)
5-Nitro-2,3-dimethoxyaniline (called 3-Amino-5-nitroveratrole by Bell & Millar) (called 5-Nitro-3-amino-brenzcatechin-dimethyläther in Ger), red ndls (from alc), mp 103–07° (Refs 1 & 5)
6-Nitro-2,3-dimethoxyaniline (called 3-Amino-4-nitroveratrole by Bell & Millar), prisms, mp 87–89° (Ref 5)
2-Nitro-3,4-dimethoxyaniline (called 3-Nitro-4-amino-veratrol or 3-Nitro-4-amino-brenzcatechin-dimethyläther in Ger), rd ndls (from dil alc), mp 74° (Ref 2)
6-Nitro-3,4-dimethoxyaniline (called 4-Amino-5-nitroveratrole by Nakamura et al and 5-Nitro-4-amino-brenzcatechin-dimethyläther in Ger), orn-colored prisms or terracotta ndls (from alc), mp 169–75° (Refs 3&6)
4-Nitro-2,5-dimethoxyaniline. Its use as an excellent fast dye on acetate artificial silk is described. No other info given in patent abstract (4)

Their methods of prepn & other props are given in Refs

Refs: 1) Beil **13**, (306) & [464] 2) Beil **13**, (311) 3) Beil **13**, (311) & [466] 4) W. Kirst, GerP 889738(1953) & CA **52**, 11434(1958) 5) F. Bell & A.S. Millar, JChemSoc, Org **1966**(4), 376 & CA **64**, 9625(1966) 6) M. Nakamura et al, ChemPharmBull(Tokyo) **14**(7), 773(1966) (Eng) & CA **65**, 13588(1966)

Dinitrodimethoxyaniline,
$(H_3CO)C_6H(NH_2)(NO_2)_2(OCH_3)$, mw 243.18, N 17.28%. Three isomers are described in the literature:
4,5-Dinitro-2,3-dimethoxyaniline, yel ndls (from dil alc), mp 141–42°; prepd by treating 5,6-dinitro-3-acetamino-veratrole with concd sulfuric acid (Ref 1, p307)
4,6-Dinitro-2,3-dimethoxyaniline or 3-Amino-4,6-dinitroveratrole, yel plates (from alc), mp 129°; was obtd by hydrolizing in cold concd sulfuric acid the 4,6-dinitro deriv of

3-toluene-p-sulphonamidoveratrole (Ref 2)
5,6-Dinitro-2,3-dimethoxyaniline, yel-brn ndls (from MeOH); prepd by heating 4,5-dinitro-3-acetamino-veratrole with concd sulfuric acid at 100°(Ref 1, p306)

Other props are given in Refs. No expl props are reported

Refs: 1) Beil **13**, (306, 307) 2) F. Bell & A.S. Millar, JChemSoc, Org **1966**(4), 376 & CA **64**, 9625(1966)

Trinitrodimethoxyaniline, $(H_3CO)C_6(NH_2)(NO_2)_3(OCH_3)$, not found in Beil or in CA thru 1966

Dimethoxyazobenzene and Derivatives. See Azoanisole and Derivatives, including the explosive hexanitro compd in Vol 1, p A646

Dimethoxybenzaldehyde and Derivatives.

Dimethoxybenzaldehyde, $H_3CO.C_6H_3(CHO).OCH_3$, mw 166.17. Two isomers are known: *2,4-Dimethoxybenzaldehyde*, ndls (from aq alc), mp 69–70°, bp 165° at 10mm (Ref 1) and *3,4-Dimethoxybenzaldehyde or Vanillin Methyl Ether*, ndls (from eth), mp 44–7°, bp 280–85° (Ref 2)

Its *Mononitroderivs* are described in Beil (Ref 3) and the *2,6-Dinitro-3,5-dimethoxybenzaldehyde* is found in the literature (Ref 4), but none is reported to be expl

The *Trinitro* deriv, $C_9H_7N_3O_9$, was not found in the literature thru 1966

Refs: 1) Beil **8**, 242 2) Beil **8**, 255 3) Beil **8**, 261, 262, (603, 610), [270, 271, 275, 277, 290] & {1989, 2001, 2002, 2062, 2064, 2065} 4) Beil **8**{2075}

Dimethoxybenzene and Derivatives

Dimethoxybenzenes, $(H_3CO)C_6H_4(OCH_3)$, mw 138.16. Three isomers are known: *ortho- or veratrol*, crysts, mp 22.5°, bp 207.1° (Ref 1); *meta- or Resorcinol dimethyl ether*, liq, mp −52°, bp ca 217° (Ref 2) and *para- or Hydroquinone dimethyl ether*, lfts, mp 56°, bp 212.6° (Ref 3). Their prepns and other props are in Beil

Mononitrodimethoxybenzene, $(H_3CO)C_6H_3(NO_2)(OCH_3)$; mw 183.16, N 7.65%. All possible isomers are found in the literature, but none is reported to be expl (Ref 3)

Dinitrodimethoxybenzene, $(H_3CO)C_6H_2(NO_2)_2(OCH_3)$; mw 228.16, N 12.28%. All isomers are described in the literature, but none is expl (Ref 4)

Trinitrodimethoxybenzene, $(H_3CO)C_6H(NO_2)_3(OCH_3)$; mw 273.16, N 15.38%. These derivs or their salts are probably expl:

3,4,5-Trinitro-1,2-dimethoxybenzene or 3,4,5-Trinitroveratrole, crysts (from benz or acet acid), mp 144–45°; sol in hot alc, eth, toluene or butanol; sl sol in w, heptane & methanol; prepd by nitration of veratrole with concd HNO_3 & H_2SO_4 at 100°(Ref 5)

3,4,6-Trinitrodimethoxybenzene, lt yel ndls (from alc), mp 174°; sl sol in ethyl acetate; diffc sol in chlf, alc & acetic acid; obtd by reaction of 2,4,6-trinitro-brenzcatechin-1-methylether, dimethylsulfate & Na_2CO_3 in hot xylene (Ref 6)

2,4,6-Trinitro-1,3-dimethoxybenzene or 2,4,6-Trinitroresorcinol-dimethylether, almost col pltlts (from alc) or lt-yel ndls (from alc), mp 124–25°; sol in alc & eth; prepd by nitrating 2,4-dinitro- & 4,6-dinitroresorcinol-dimethylether with HNO_3 & concd H_2SO_4 in the cold (Ref 7)

4,5,6-Trinitro-1,3-dimethoxybenzene, col crysts (from alc), mp 193°; obtd by nitrating 5-nitroresorcinol-dimethylether or 4,5-dinitroresorcinol-dimethylether with HNO_3 & concd H_2SO_4 (Ref 8)

Trinitrohydroquinone-dimethylether, yel ndls (from alc), mp 100–01°; diffc sol in hot 80% alc; almost insol in cold alc; prepd by treating cold soln of hydroquinone-dimethylether in acetic acid with a cold mixt of HNO_3 1 part & concd H_2SO_4 2 parts (Ref 9)

Other methods of prepn & props are found in the Refs

Tetranitrodimethoxybenzene. Not found in literature thru 1966

Refs: 1) Beil **6**, 771 2) Beil **6**, 813 3) Beil **6**, 788, 789, 823, 824, 825, 843, 857, (392, 404, 418), [790, 793, 794, 822, 849] & {4263, 4264, 4344, 4347, 4442} 4) Beil **6**, 791, 792, 827, 828, 857, 858, (393, 394, 405, 418), [793, 794, 823, 824] & {4274, 4351, 4352, 4444} 5) Beil **6**, 792, (395), [795] & {4276} 6) Beil **6**, (396) 7) Beil **6**, 832, (406) & [826] 8) Beil **6**, 833 9) Beil **6**, 858

Dimethoxybenzophenone and Derivatives

Dimethoxybenzophenone (4,4') or p-Anisoyl-anisole, $(H_3CO)C_6H_4.CO.C_6H_4(OCH_3)$, mw 242.26, ndls from alc, mp 144–45°. Its prepn and other props are in Ref 1

Forms on nitration tetranitro- and pentanitro compounds:

Dimethoxy-3,3',5,5'-tetranitro-benzophenone, $(H_3CO)C_6H_2(NO_2)_2.CO.C_6H_2(NO_2)_2(OCH_3)$, mw 422.26, N 13.27%, crysts, mp 197° (2nd form on recrystn from acet, mp 182°). Can be prepd by slowly adding concd nitric acid to dimethoxybenzophenone at –15°, allowing to stand at RT and pouring on ice (Refs 2 & 4)

3,3'-Dimethoxy-2,4,6,4',6'-pentanitro-benzophenone,
$(H_3CO)C_6H(NO_2)_3.CO.C_6H_2(NO_2)_2(OCH_3)$; mw 467.27, N 14.99%; crysts (from acet); mp 209°; was obtd by nitrating the tetranitro deriv with a mixt of nitric acid and sulfuric acid (Refs 1a & 4). No expl props of this compd are reported

Refs: 1) Beil **8**, 317, (641), [355] & {2649} 1a) Beil **8** {2648} 2) J. van Alphen, RecTravChim **49**, 153–64 (1930) & CA **24**, 3007 (1930) 3) J.G. Kerkhof, RecTravChim **51**, 739 (1932) & CA **26**, 5557 (1932) 4) C.W. Pohlmann, Rec **55**, 737–52 (1936) & CA **30**, 7110–11 (1936)

o,o'-Dimethoxybenzoyl Peroxide, $H_3CO.C_6H_4.O.O.C_6H_4.OCH_3$; mw 246.25, O 25.99%; crysts, mp 81–82°; was obtd when o-methoxybenzoyl chloride was slowly added over 30 mins to a soln of Na peroxide in w contg a few drops of "Triton" 720 (Rohm & Haas Co trademarked surfactant)

A 1g sample heated 4.5°/min decompd suddenly with a shattering effect & a loud report; a 10g sample at 75° exploded after 30 mins. At 30° the compd liberated 98.2% available O in 3 wks, leaving a dk viscous mass. The Peroxide is shock sensitive (8 out of 10 trials) to an 8-lb impact at 3 inches (Ref 2)

Refs: 1) Beil – not found 2) J.R. Crowdle et al, Rec **73**, 560 (1954) & CA **49**, 8869 (1955) 3) S. Matsumoto et al, JapP 3918 (1956) & CA **51**, 16547 (1957) (Prepn)

Dimethoxy-biphenyl. See Dianisole, p D1151-L

Dimethoxy-bis(phenylmercapto)-benzene and Derivatives

2,4-Dimethoxy-1,5-bis(phenylmercapto)-benzene, may be considered as the parent compd of its hexanitro deriv, although not used to prep it:

Hexanitrodimethoxy-bis(phenylmercapto)-benzene or **2,4-Dimethoxy-1,5-bis(2,4,6-trinitro-phenylmercapto)-benzene,**
$(CH_3.O)_2C_6H_2[S.C_6H_2(NO_2)_3]_2$; mw 624.48, N 13.46%; yel tablets (from acet acid) with 2 moles acet ac, mp 228–30°; readily sol in acet ac; mod sol in benz & eth acet; diffc sol in alc, eth, chlf & petr eth; was obtd by reaction of 2,4-dimethoxy-1,5-dimercapto-benzene & picryl chloride in alc (Refs 2 & 3)

No expl props are reported

Refs: 1) Beil – not found (Parent) 2) Beil **6**, (571) (Hexanitro) 3) J. Pollak & A. Wienerberger, Monatshefte **35**, 1488 (1914) & CA **9**, 911 (1915)

Dimethoxy-methylbenzene. See Dimethoxy-toluene

Di(methoxymethyl)-peroxide or **Bis(methoxymethyl)-Peroxide,** $(H_3COCH_2)_2O_2$; mw 122.12, O 52.41%; crysts; decomp into HCOOH & HCHO by Fe salts; was prepd when ether solns of HCHO & of hydrogen peroxide were brought together and the ether evaporated; this peroxide & H_2O_2 in ether, or anhyd HCHO & H_2O_2 gives the expl, **Hydroxymethyl Hydroperoxide** (Ref 2)

Refs: 1) Beil – not found 2) A. Rieche & R. Meister, Ber **68B**, 1465 (1935) & CA **29**, 6877 (1935)

Dimethoxynaphthalene and Derivatives
Dimethoxynaphthalene, $H_3CO.C_{10}H_6.OCH_3$, mw 188.22. The various derivs are described in Beil (Ref 1)

2,4,6,8-Tetranitro-1,5-dimethoxy-naphthalene, $(H_3CO).C_{10}H_2(NO_2)_4(OCH_3)$, mw 368.22, N 15.22%; orn-colored crysts (from toluene), mp 255°(dec); prepd by nitrating either 2,4-dinitro- or 2,4,8-trinitro-1,5-dimethoxynaphthalene with nitric acid (d 1.5) in AcOH (Refs 2 & 3)
Refs: 1) Beil **6**, 975, 979, 980, 983, 984, 986, (478, 480, 482), [952, 953, 956] & {5261, 5266, 5280, 5287, 5288, 5292} 2) Beil **6**, {5275} 3) E. Race & F.M Rowe, JChemSoc **1947**, 350–53 & CA **41**, 5494(1947)

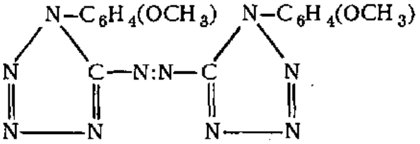
Dimethoxypentamethylenetetramine and Derivatives
1,9-Dimethoxy-1,3;5,7,9-pentamethylene-2,4,6,8-tetramine or *1,9-Dimethoxy-2,4,6,8-tetrazanonane,* $(H_3CO)CH_2NH\,CH_2.NH.CH_2.NH.CH_2.NH.CH_2(OCH_3)$, mw 192.27, N 29.14%. May be considered as the parent compd of its tetranitro deriv although not used to prep it:
2,4,6,8-Tetranitronitramine-1,9-dimethoxy-1,3,5,7-pentamethylene or 2,4,6,8-Tetranitro-1,9-dimethoxy-2,4,6,8-tetrazanonane, $(H_3CO)CH_2.N(NO_2).CH_2.N(NO_2).CH_2.N(NO_2).CH_2.N(NO_2).CH_2(OCH_3)$; mw 372.26, N 30.01%; crysts (from 1:1 dioxane-MeOH); mp 182–83°; sol in 1:1 dioxane-methanol. Was prepd in Canada as one of the products of nitrolysis of hexamine (hexamethylenetetramine): when the nitrate ester of 1,9-Dinitroxy-2,4,6,8-tetranitro-2,4,6,8-tetrazanonane (qv) was boiled in methanol for one hr, the subject compd crystallized out on cooling (Ref 2)
Refs: 1) Beil – not found 2) A.F. McKay et al, CanJRes **27B**, 467(1949)

Dimethoxyphenol and Derivatives
3,5-Dimethoxyphenol or *Pyrogallol Dimethyl Ether*, $(H_3CO)_2C_6H_3OH$, mw 154.16, crysts, mp 55–56°, bp 262.7°. Other props and method of prepn in Beil (Ref 1)

Dimethoxypicric Acid or 2,4,6-Trinitro-3,5-dimethoxyphenol, $(H_3CO)_2C_6H(NO_2)_3OH$, mw 289.16, N 14.53%; crysts; mp 76.5°; was prepd by the following reactions: 3,5-dinitromethoxybenzene with $AlCl_3$ at 130° yielded $(O_2N)_5C_6OH$ which by Blanskma method for the introduction of MeO groups yield the trinitro deriv (Ref 3)
Refs: 1) Beil **6**, 1081 & {6305} 2) Beil – not found (Trinitro) 3) H.H. Schlubach & F. Mergenthaler, Ber **58B**, 2732(1925) & CA **20**, 1395(1926) 4) Not found in later refs thru 1966

2,4-Dimethoxyphenylarsinic Acid and Derivatives
Dimethoxyphenylarsinic Acid, $(CH_3O)_2C_6H_3.AsO(OH)_2$, mw 263.09, ndls (from w), mp 242–43°; sol in MeOH, alc & acet ac; sl sol in w; v sl sol in eth (Ref 1)
x-Dinitro-3,4-dimethoxyphenylarsinic Acid, $(H_3CO)_2\,C_6H(NO_2)_2.AsO(OH)_2$, mw 352.08, N 7.95%; crysts, mp decomp at 215° and explodes at higher temps. Can be prepd by treating 3,4-dimethoxyphenylarsinic acid with abs HNO_3 & concd H_2SO_4 (Refs 2 & 3)
Refs: 1) Beil **16**, (459) (Parent) 2) Beil **16**, [473] (Dinitro) 3) M.P. deLange, Rec **45**, 55(1926) & CA **20**, 1982(1926)

1,1'-Di-o-methoxyphenyl-5,5'-azotetrazole or 5,5'-Azobis-[1-(o-methoxyphenyl)-1,2,3,4-Tetrazole],



mw 378.35, N 37.02%; yel crysts, mp 190° (dec); sol in hot alc; insol in w & eth. Its prepn and other props are not given in abstract (Ref 2). It is described in Ref 3
Refs: 1) Beil – not found 2) R. Stollé et al, JPraktChem **134**, 282–309(1932) & CA **26**, 5565(1932) 3) L.F. Audrieth & J.W. Currier, Univ of Ill Rept, "Derivatives of Aminotetrazole" (1954) 4) Not found in later refs thru 1966

Dimethoxytetramethylenetriamine and Derivatives

1,7-Dimethoxy-1,3,5,7-tetramethylene-2,4,6-triamine or *1,7-Dimethoxy-2,4,6-triazaheptane*, $(H_3CO).CH_2.NH.CH_2.NH.CH_2.NH.CH_2(OCH_3)$, mw 163.22, N 25.75%, may be considered as the parent compd of its trinitro deriv although not used to prep it:

1,7-Dimethoxy-1,3,5,7-tetramethylene-2,4,6-trinitramine or **1,7-Dimethoxy-2,4,6-trinitro-2,4,6-triazaheptane,** $(H_3CO).CH_2.N(NO_2).CH_2.N(NO_2).CH_2.N(NO_2).CH_2(OCH_3)$, mw 298.22, N 28.18%; crysts, mp 100–104°. Was prepd in Canada as one of the products of nitrolysis of hexamine (hexamethylenetetramine). See Ref 3 also

Refs: 1) Beil – not found 2) W.J. Chute et al, CanadJRes **27B**, 510(1949) 3) A.D. Little, Synthesis HE's, 2nd Rept(1951), p 273 (IR data)

Dimethoxytoluene and Derivatives

Dimethoxytoluenes or *Dimethoxy-methylbenzenes*, $H_3C.C_6H_3(OCH_3)$, mw 152.19. The known isomers are found in Beil (Ref 1)

Mononitrodimethoxytoluenes, $H_3C.C_6H_2(NO_2)(OCH_3)_2$. All derivs are described in Beil (Ref 2)

Dinitrodimethoxytoluenes, $H_3C.C_6H_2(NO_2)_2(OCH_3)_2$. These isomers are described in Beil (Ref 3)

Trinitro-dimethoxytoluenes or **Trinitro-dimethoxymethylbenzenes,** $H_3C.C_6(NO_2)_3(OCH_3)_2$, mw 287.19, N 14.63%.

4,5,6-Trinitro-2,3-dimethoxytoluene, ndls (from alc), mp 131–32° (Ref 4, p428)

3,5,6-Trinitro-2,4-dimethoxytoluene, no description given (Ref 4, p861)

2,5,6-Trinitro-3,4-dimethoxytoluene, yel prisms (from alc), mp 100–01° or ndls (from MeOH), mp 73–74° (Ref 4, pp433, 872)

2,4,6-Trinitro-3,5-dimethoxytoluene, crysts, mp 69.5° (Ref 4, p891)

The methods of prepn & other props are found in the Refs. These compds are probably mild expls

Refs: 1) Beil **6**, 872, 879, (426, 428, 432), [859, 866] & {4493, 4496, 4499, 4513, 4516, 4533} 2) Beil **6**, 881, (427, 433), [861, 868, 869] & {4497, 4504, 4522, 4536} 3) Beil **6**, (427, 428, 433) & [859, 870, 871, 872] 4) Beil **6**, 891, (428, 433) & [861, 872]

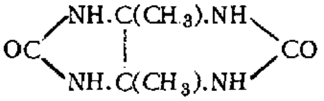
Dimethylacetylene and Derivatives

Dimethylacetylene, Butyne-2 or *Crotonylene*, $H_3C.C\vdots C.CH_3$, mw 54.09, col liq, fr p –32.2°, bp 27°. Other props & prepn are in Beil (Ref 1)

Hexanitrodimethylacetylene, $(O_2N)_3C.C\vdots C.C(NO_2)_3$, mw 324.09, N 25.93% solid, mp – explodes. Was prepd in the US, but Blatt & Whitmore (Ref 2) found that this expl is too expensive for use as a component of military expls

Refs: 1) Beil **1**, 249, (107), [223] & {925} 2) A.H. Blatt & F.C. Whitmore, OSRD **1085** (1942), p43

7.8-Dimethyl-acetylendiurein,



$$OC\begin{matrix} \diagup NH.C(CH_3).NH \diagdown \\ | \\ \diagdown NH.C(CH_3).NH \diagup \end{matrix}CO$$

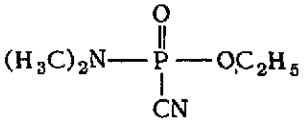
This German name, as well as the following two names, is given in Beil **26**, 445 & (131): *2.2′-Dioxo-4.5-dimethyl-hexahydro-[imidazolo-4′.5′:4.5-imidazol]*, *β.γ* and *β.γ-Diureylen-butan*

This compound can be nitrated to a dinitro deriv. See β.γ-Diurelylen-butan and Derivatives

Dimethylamine and Derivatives

Dimethylamine, $(H_3C)_2NH$, mw 45.08, N 31.07%, col gas at RT and liq at below 7°, sp gr 0.680 at 0°/4, fr p –92.2°; extremely flammable and hazardous; causes burns; sol in w, alc & eth. Can be prepd by passing a mixture of ammonia with vapors of methanol over a catalyst, such as silica gel. Has been used in agricultural herbicides and fungicides, synthetic textiles, fiber solvents, rubber vulcanizing accelerators and in *missile fuels* (Refs 1, 2, 4)

Accdg to Dr H. Walter, formerly at Picatinny Arsenal, dimethylamine was used during WWII in Germany for the production of war gas, *Tabun*, also called *Trilon 83*,



$$(H_3C)_2N-\underset{\underset{CN}{|}}{\overset{\overset{O}{\|}}{P}}-OC_2H_5$$

(Ref 3), and also for manuf of Di-Salz, described here as Diammonium Nitrate. Some of its salts are expl (Ref 1)
Refs: 1) Beil **4**, 39, (320), [550] & {93} 2) Kirk & Othmer **1**(1947), p706 2a) Anon, "The Methylamines", Rohm & Haas, Philadelphia, Pa (1954), 86pp & CA **49**, 3236 (1955) 3) PATR **2510**(1958), p Ger 204-L 4) CondChemDict (1961), p392-L 5) Kirk & Othmer **2**(1963), p118

Dimethylamine Chlorite, $(H_3C)_2.NH.HClO_2$; mw 113.54, N 12.34%; was prepd by Levi by treating 1 mol of concd aq soln of dimethylamine (previously neutralized with 2N sulfuric acid) with 1 mol of Ba chlorite and evaporating the liquid mixture in vacuum. The resulting compd was very unstable and decomposed explosively on heating
Refs: 1) Beil **4**,[552] 2) G.R. Levi, Gazz **52**(I), 209(1922) & CA **16**, 2474(1922)

N-Nitrodimethylamine or **Dimethylnitramine,** $(H_3C)_2N.NO_2$; mw 90.08, N 31.10%, OB to CO_2 −88.8%; col ndls, sp gr 1.090 at 72.3°, mp 57–58°, bp 187° at 758mm, n_D 1.4462 at 72.3°; sol in w, alc & eth. Can be prepd by treating 10 parts of dimethylammonium nitrate with 20 parts of acetic anhydride (Ref 1)

It was investigated in Germany by Römer (Refs 2 & 3) as a possible addn to R-Salz in order to render it castable at temps below 100°. It was found that incorporation of as low as 10% of DMNA was sufficient. R-Salz is Ger designation for Cyclotrimethylenetrinitrosamine described in Vol 3 of Encycl, p C630-R
Refs: 1) Beil **4**, 85, (342) & {167} 2) G. Römer, "Report on Explosives", PBL Rept 85160(1946), pp3–15 3) Fedoroff et al, PATR **2510**(1958), p Ger 36-R (Dimethylnitramine) and Ger 170 (R-Salz) 4) K. Suryanarayanan & S. Bulusu, JChemPhys **76**, 496–500(1972)

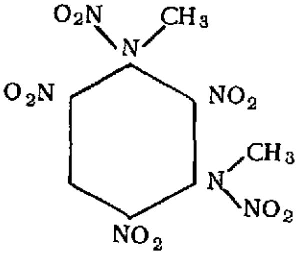
N-Nitrosodimethylamine or **Dimethylnitrosamine,** $(H_3C)_2N(NO)$, mw 74.08, N 37.82%, yel oil, bp 154°, d 1.0061 at 20°, n_D 1.4368 at 20°- heat of combstn at Cv detd by Swientoslavski 394.3cal/g; prepd by heating dimethylammonium nitrite at 60°, or by heating trimethylamine with Na nitrite & concd HCl (Ref 1). Microcolorimetric detn of DMNsA is described by LeDuigou (Ref 6) and polarographic detn by Monard & Garrigues (Ref 7)
Refs: 1) Beil **4**, 84, (341), [585] & {166} 2) V. Swientoslawski, JRussPhysChemSoc **41**, 925(1909) & CA **5**, 1415(1911) 3) M. Levine & R.M. Isham, USP 2703154(1955) & CA **49**, 7153(1955) 4) G. Desseigne & A. Audiffren, MP **40**, 337–41(1958) (Prepn of DMNsA) 5) J. Issoire, FrP 1165675 (1958) & MP **41**, 465–66(1959) (Procedure for extraction) 6) Yves LeDuigou, MP **41**, 319–25(1959) 7) C. Monard & C. Garrigues, MP **41**, 364–68(1959)

1,1'-Dinitramino-N-nitro-dimethylamine (called Trinitrodiaminodimethylamine by DiCerrione), $O_2N.N(CH_2NHNO_2)_2$, mw 310.12, N 26.93%. A secondary product formed in the nitration of hexamethylenetetramine to trimethylenetrinitramine. Description of this compd is not given in the abstract (Ref 2)
Refs: 1) Beil – not found 2) A. DiCerrione, AnnChimApplicata **38**, 255(1948) & CA **43**, 4633(1949) 3) Not found in later refs thru 1966

Dimethylaminoaniline and Derivatives

N,N-Dimethylaminoaniline, p-Aminodimethylaniline or *Dimethyl-p-phenylenediamine,* $(H_3C)_2N.(C_6H_4).NH_2$, mw 136.19, N 20.57%; col ndls, sp gr 1.041 at 15°/15, mp 41°, bp 262.3°. Other props and method of prepn in Refs 1 & 2

Pentanitrodimethylaminoaniline, N,N',2,4,6-Pentanitro-N,N'-dimethyl-m-phenylenediamine,



mw 361.19, N 27.15%. This compd is properly described under N,N'-Dimethyl-m-phenylenediamine and Derivatives in this Vol
Refs: 1) Beil **13**, 72, (21) & [39] 2) CondChemDict (1961), p60-R (p-Aminodimethylaniline)

Dimethylamino-anthraquinone. See Bis(methylamino)-anthraquinone in Vol 2 of Encycl, p B148-R

Dimethylaminoazobenzene and Derivatives
p-Dimethylaminoazobenzene,
$(H_3C)_2N.C_6H_4.N:N.C_6H_5$, mw 225.28, N 18.65%; yel lfts, mp 116–17°, bp decomp. Other props & prepn are in Refs 1 & 4

4,6,4'-Trinitro-3-dimethylaminoazobenzene,
$O_2N.C_6H_4.N:N.C_6H_2(NO_2)_2.N(CH_3)_2$, mw 360.28, N 23.33%; orn-red powd (from glac acet ac), mp 214–16° (sinters at 190°); prepd by boiling quinone oxime-2,4-dinitro-5-dimethylaminophenylhydrazone with AcOH & concd nitric acid (Ref 2 & 3)
Refs: 1) Beil **16**, 312, (310) & [151] 2) Beil **16**, [149] 3) W. Borsche, Ber **54B**, 675(1921) & CA **15**, 2844(1921) 4) CondChemDict (1961), p382-L

Dimethylaminobenzaldehyde and Derivatives
p-Dimethylaminobenzaldehyde,
$(H_3C)_2N.C_6H_4.CHO$, mw 149.19, N 9.39%; lfts, mp 74–75°, bp 176–77° at 17mm; sol in hot w and in alc, eth & AcOH. Can be prepd by mixing dimethylaniline, anhydrous chloral and phenol and allowing the mixt to stand. The phenol is removed by shaking with dil caustic soda and the residue dissolved in w & HCl and crystallized (Ref 1)

It was used in some military applications and its US Military specification and tests were described in Ref 3, which is now cancelled
Refs: 1) Beil **14**, 31, (360) & [360] 2) CondChemDict (1961), p392-R 3) US Spec MIL-D-11359 (Nov 1951) (Cancelled)

Mononitrodimethylaminobenzaldehyde,
$(H_3C)_2N.C_6H_3(NO_2).CHO$, mw 239.19, N 17.57%. Two isomers are found in Beil:
5-Nitro-2-dimethylaminobenzaldehyde, yel ndls (from petr eth), mp 105° (Ref 1)
3-Nitro-4-dimethylaminobenzaldehyde, yel ndls (from alc), mp 103–05° (Ref 2)

Neither compd is indicated to be expl
Refs: 1) Beil **14**, 28 2) Beil **14**, 39 & (364)
NOTE: No higher nitrated derivs of Dimethylaminobenzaldehyde were found in the literature thru 1966

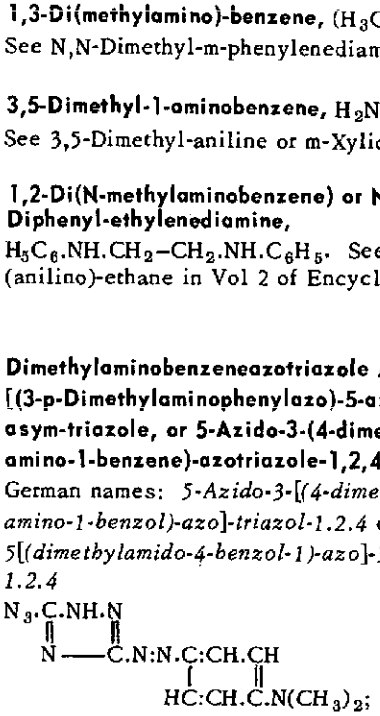
N,N-Dimethyl-aminobenzene, $(H_3C)_2N.C_6H_5$. See N,N-Dimethylaniline

1,3-Di(methylamino)-benzene, $(H_3C.HN)_2C_6H_4$. See N,N-Dimethyl-m-phenylenediamine

3,5-Dimethyl-1-aminobenzene, $H_2N.C_6H_3(CH_3)_2$. See 3,5-Dimethyl-aniline or m-Xylidine

1,2-Di(N-methylaminobenzene) or N,N'-Diphenyl-ethylenediamine,
$H_5C_6.NH.CH_2{-}CH_2.NH.C_6H_5$. See Bis(anilino)-ethane in Vol 2 of Encycl, p B131-L

Dimethylaminobenzeneazotriazole Azide, [(3-p-Dimethylaminophenylazo)-5-azido]-asym-triazole, or 5-Azido-3-(4-dimethylamino-1-benzene)-azotriazole-1,2,4.
German names: *5-Azido-3-[(4-dimethylamino-1-benzol)-azo]-triazol-1.2.4 or Azido-5[(dimethylamido-4-benzol-1)-azo]-3-triazol-1.2.4*



mw 257.26, N 49.00%; bordeaux-red pdr; mp def gr ca 185° when heated in a capillary tube; sol in w & alc; nearly insol in eth; insol in eth acet. Was prepd by Stollé & Dietrich on treating an intimate mixt of Na nitrite and dihydrochloride of 3-amino-5-hydrazino-1,2,4-triazole with concd HCl, followed by addn of dimethylaniline in concd HCl
Refs: 1) Beil – not found 2) R. Stollé & W. Dietrich, JPraktChem **139**, 202(1934) & CA **28**, 2714(1934)

Dimethyl-aminobenzene-4-diazonium Hydroxide. Same as N,N-Dimethyl-aniline-4-diazonium Hydroxide

1,1-Dimethyl-aminobenzene-4-diazonium Perchlorate. Same as N,N-Dimethyl-aniline-diazonium Perchlorate

Di(methylamino)-benzil. See Bis(methylamino)-benzil in Vol 2 of Encycl, p B149-L

Dimethylaminobenzoic Acid and Derivatives

Dimethylaminobenzoic Acid, $HO_2C.C_6H_4.N(CH_3)_2$, mw 165.19, N 8.48%. Exists in three isomeric forms:
Dimethylamino-o-benzoic Acid, crysts, mp 72° (Ref 1)
Dimethylamino-m-benzoic Acid, ndls, mp 151° (Ref 2)
Dimethylamino-p-benzoic Acid, ndls, mp 238–43° (Ref 3)

3,5-Dinitro-2-dimethylaminobenzoic Acid, $HO_2C.C_6H_2(NO_2)_2.N(CH_3)_2$; mw 255.19, N 16.47%; orn-yel ndls, mp 121° (Ref 4, p380)
3,5-Dinitro-4-dimethylaminobenzoic Acid, orn-yel ndls, mp 246°; sol in hot alc; sl sol in glac acet ac; insol in cold w & benz (Ref 4, p445)

No expl props are reported

3,5-Dinitro-4-dimethylaminobenzoyl Azide, $N_3OC.C_6H_2.(NO_2)_2.N(CH_3)_2$, mw 280.20, N 30.00%; crysts (from AcOH), mp 125°; obtd by reacting 3,5-dinitro-4-dimethylaminobenzoyl chloride (mp 118–19°) in AcOH with NaN_3 (Ref 6)

Its expl props are not reported

3,x,x-Trinitrodimethylamino-p-benzoic Acid, $HO_2C.C_6H(NO_2)_3.N(CH_3)_2$; mw 300.19, N 18.67%; yel pltlts, mp 193°; was prepd by nitrating 3-nitro-4-dimethylaminobenzoic acid with concd H_2SO_4 & HNO_3 (Ref 5). No expl props were reported

NOTE: No higher nitrated derivs were found in the literature thru 1966

Refs: 1) Beil 14, 325, (532) & [213] 2) Beil 14, 392 3) Beil 14, 426, (571) & [259] 4) Beil 14, 380, 445 5) Beil 14, 447 6) K.A. Jensen et al, ActaChemScand 4, 392(1950) & CA 44, 9377(1950)

Di(methylamino)-benzophenone. See Bis-(methylamino)-benzophenone in Vol 2, p B149-L

Dimethylamino-benzophenoneimide. Same as Auramine described in Vol 1, p A507-R

N,N-Dimethylaminobiphenyl and Derivatives

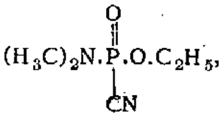
N,N-Dimethyl-p-aminobiphenyl; N,N-Dimethylxenylamine; N,N-Dimethyl-p-biphenylamine; N,N-Dimethyl-p-phenylaniline or N,N-Dimethyl-diphenylylamine, $C_6H_5.C_6H_4.N(CH_3)_2$, mw 197.27, N 7.10%; pltlts (from alc or eth), mp 123–26°. Its salt, $C_{14}H_{17}N_3O_5S$, in warm dil HCl gives colorless ndls product which is expl (Ref 1)

3,4'-Dinitro-4-dimethylaminobiphenyl, $O_2N.C_6H_4.C_6H_3(NO_2).N(CH_3)_2$; mw 287.27, N 14.63%; orn-red pltlts (from alc), mp 137–38°; prepd by nitrating either 3-nitro- or 4'-nitro-4-dimethylaminobiphenyl (Ref 2)

2,6,2',4'-Tetranitro-N,N-dimethyl-p-aminobiphenyl or N,N-Dimethyl-2,6,2',4'-tetranitro-xenylamine, $(O_2N)_2C_6H_3.C_6H_2(NO_2)_2.N(CH_3)_2$, mw 377.27, N 18.56%; large ndls (from alc), mp 122°; was obtd by nitrating either 4-dimethylaminobiphenyl or 3-nitro-4-dimethylaminobiphenyl with HNO_3 & AcOH (Ref 3). Its expl props were not reported

Refs: 1) Beil 12, [754] 2) Beil 12, [763] 3) A.G. Banús & J.F. Tomás, AnalesSoc-EspañFísQuím 19, 293(1921) & CA 16, 2135(1922)

Dimethylaminocyanophosphoric Acid, Monomethylester of; Dimethylaminoethoxycyanophosphine Oxide or Ethylphosphorodimethylamino-cyanadote,



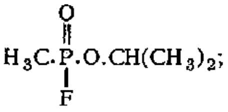
$$(H_3C)_2N.\underset{\displaystyle CN}{\overset{\displaystyle O}{\overset{\|}{P}}}.O.C_2H_5,$$

invented and known in Germany before WWII as **Tabun Trilon 83, T83 or T100**; adopted by the US Army after WWII under the code name **GA**; pure product is colorless and odorless oil, while technical material is dk brown with fishy odor, sp gr 1.077 at 20°. It was prepd by treating the dichloride of dimethylaminophosphoric acid (an irritating agent called **Product 39**) with NaCN, ethanol and chlorobenzene

Tabun was planned to be used in chem bombs and rockets, but as it proved to be unstable and of high vapor pressure when used alone, chlorobenzene was incorporated, 5% at first **(Tabun A)** and later 20% **(Tabun B)** (Refs 1, 2, 3 & 4)

Tabun is one of the three compounds known in Germany as **Trilons** and in the US as **Chemical Warfare "G" Agents**

Other members of Trilon group are:
Sarin (GB) or Trilon 46. It is **Methylisopropoxyfluoro-phosphine Oxide** or **Monoisopropylester of methylfluorophosphoric Acid** (called **Nerve Gas** by G. Schrader)

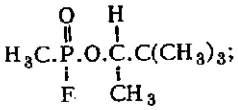


$$H_3C.\underset{\underset{F}{|}}{\overset{\overset{O}{\|}}{P}}.O.CH(CH_3)_2;$$

col, odorless, very volatile liq, 3 times as toxic as Tabun. It was adopted in the US under the code name **GB** and quite a number of rockets, mines, etc were loaded with Sarin. As these munitions started to leak, 12450 rockets contg 68 tons of "nerve gas", plus one land mine contg 10.5 lb of even deadlier **VX** gas were drowned (notwithstanding numerous protests), loaded on an old Liberty ship, under 16000 feet of water 282 miles east of Cape Kennedy, Florida (Refs 1, 2, 3, 4 and newspapers)

Note: VX gas in an extremely poisonous gas of secret formula. A small quantity accidentally released during tests conducted March 1968 in Utah instantly killed a herd of sheep (ca 6500)

The 3rd member of the Trilon group was **Soman.** It is **Methylpinacolyloxyfluorophosphine Oxide, Monopinacolicester of methylfluorophosphoric Acid or Pinacolylmethylphosphonofluoridate,**

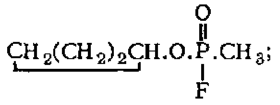


$$H_3C.\underset{\underset{F}{|}}{\overset{\overset{O}{\|}}{P}}.O.\underset{\underset{CH_3}{|}}{\overset{\overset{H}{|}}{C}}.C(CH_3)_3;$$

col liq with camphor-like odor; it is less volatile than Sarin but even more toxic

Soman was adopted in the US under code name **GD** (Refs 1, 2, 3 & 4)

Another member of "G" Agents, which seems to be developed in the USA is **CF**, which is **Cyclohexylmethylphonofluoridate** or **Cyclohexylic-ester of methylfluorophosphoric Acid,**



$$\underbrace{CH_2(CH_2)_2CH}.O.\underset{\underset{F}{|}}{\overset{\overset{O}{\|}}{P}}.CH_3;$$

(Ref 4, p C168)

Refs: 1) Captain Collomp, Revue Mensuel de l'Armée de l'Air, **No 37**, October 1949 2) R.D. McLeod, ChemEngrgNews **32**, 8 (1954) 3) Fedoroff et al, PATR **2510** (1958), p Ger 204-L 4) Fedoroff & Sheffield, PATR **2700**, Vol 2 (1962), pp C167-R, C168-L & C169-L 5) Anon, "Military Chemistry and Chemical Agents", **TM 3-215/AFM 355-7,** US Depts of the Army and the Air Force, Washington, DC (Dec 1963), pp 14–16, 18 & Table 1

Di(methylamino)-diazido-copper (II) or **Di(methylamino-copper (II) Diazide,** $[Cu(CH_3NH_2)_2](N_3)_2$; mw 209.76, N 53.42%; crysts smelling like methylamine; sinters at 84°; mp 126–28°; bp expl at 180–90° on a preheated block; does not detonate by impact; burns in flame with crepitation; unstable in air but can be stored in a closed container; insol in neutral solvents, but dissolves in some basic liquids, such as pyridine; hydrolized by water. Can be prepd by dissolving $Cu(N_3)_2$ in dimethylamine and addg an acid to ppt the salt

Refs: 1) Beil 4 {98} 2) A. Cirulis & M. Straumanis, ZAnorgChem **251**, 345 (1943) & CA **37**, 6574 (1943)

Dimethylaminodiphenyl. Same as Dimethylaminobiphenyl described in this Vol

Dimethylaminodiphenylamine and Derivatives
4-Dimethylaminodiphenylamine,

$(H_3C)_2N.C_6H_4.NH.C_6H_5$, mw 212.28, N 13.20%; ndls (from petr eth), mp 130°(Ref 1)

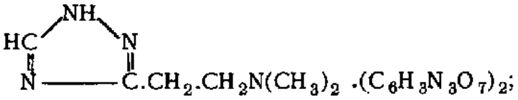
2,4,2′,4′,6′-Pentanitro-3-dimethylaminodiphenylamine,
$(H_3C)_2N.C_6H_2(NO_2)_2.NH.C_6H_2(NO_2)_3$, mw 437.28, N 22.42%; brn-yel crysts (from glac acetic ac), mp 149°; was prepd by reacting dimethylamine & 2,4,6,2′(?),3′,4′-hexanitrodiphenylamine in MeOH (Refs 2 & 3). No expl props are reported
Refs: 1) Beil **13**, 79, (23) & [41] 2) Beil **13**, (16) 3) C.F. van Duin & B.C.R. van Lennep, Rec **38**, 362(1919) & CA **14**, 2471 (1920)

Di-methylamino)-diphenylmethane. See Bis-(methylamino)-diphenylmethane in Vol 2 of Encycl, p B149-R

2-Dimethylaminoethanol and Derivatives
2-Dimethylaminoethanol; Deanol or *2-Dimethylethanolamine,* $(H_3C)_2N.CH_2.CH_2.OH$, mw 89.14, N 15.71%, col liq, sp gr 0.887 at 20°/4, fr p −59°, bp 135° at 758mm, flash p (open cup) 105°F. Can be prepd from ethylene oxide and dimethylamine. Used as chemical intermediate, pharmaceutical and corrosion inhibitors (Refs 1 & 3)

2-Dimethylaminoethanol Nitrate,
$(H_3C)_2N.CH_2.CH_2.ONO_2$, mw 134.14, N 20.89%; prisms (from alc), mp 63°; was prepd by reaction of 97% HNO_3 on 2-dimethylaminoethanol nitrate, $C_4H_{10}N_2O_3.HNO_3$, at −5° (Refs 2 & 4). It forms a salt, $2C_4H_{10}N_2O_3+H_2PtCl_6+2H_2O$, orn-red prisms, mp 204–05° (dec) (Refs 2 & 4)
Refs: 1) Beil **4**, 276, (425), [719] & {647} 2) Beil **4**, [719] & {651} 3) CondChemDict (1961), 392-R 4) D. Bovet et al, CRSocBiol **140**, No 9–10, 292(1946) & CA **41**, 3868 (1947) (Hypotensive & vasodilator activities of nitric esters of amino alcohols)

3β-Dimethylaminoethyl-1,2,4-triazole Dipicrate,


$HC{-}NH{-}N{=}C.CH_2.CH_2N(CH_3)_2\;.(C_6H_3N_3O_7)_2$;

mw 598.40, N 23.41%; ndls (from w), mp 181–82°, expl at higher temp. Was prepd by refluxing for 2 days a mixt of α-(1,2,4-triazolyl-3)-N,N-dimethylacetamide with $LiAlH_4$ and anhydrous tetrahydrofurane, followed by treatment with Picric Acid
Refs: 1) Beil – not found 2) C. Ainsworth & R.G. Jones, JACS **76**, 5653(1954) & CA **49**, 13979(1955)

Dimethylaminohydroxylaminoazobenzene and Derivatives
Dimethylaminohydroxylaminoazobenzene,
$(H_3C)_2C_6H_4.N{:}N.C_6H_4.NHOH$; mw 202.29, N 20.77%; may be considered as the parent compd of its dinitro deriv although not used to prep it (Ref 1)

4,6-Dinitro-3-dimethylamino-4′-hydroxylaminoazobenzene,
$(H_3C)_2C_6H_2(NO_2)_2.N{:}N.C_6H_4NHOH$ or $(H_3C)_2C_6H_2.NH.N{:}C_6H_4{:}N.OH$; mw 332.29, N 21.73%; red-brn crysts, mp 227°; sol in NaOH with violet-red color; obtd by reaction of 4,6-dinitro-3-dimethylamino-phenylhydrazine & benzoquinone-monoxime in alc HCl (Refs 2 & 3)
Refs: 1) Beil **16** – not found 2) Beil **16**, [261] 3) W. Borsche, Ber **54**, 675(1921) & CA **15**, 2844(1921)

Dimethylaminomethylaminotoluene and Derivatives
Dimethylaminomethylaminotoluene,
$(H_3C)_2N.C_6H_3(NHCH_3).CH_3$; mw 164.24, N 17.06%. May be considered as the parent compd of its nitrated derivs:

3,5-Dinitro-2-dimethylamino-4-methylaminotoluene (Ref 1),
$(H_3C)_2N.C_6H(NO_2)_2(NHCH_3).CH_3$; mw 254.24, N 22.04%; yel ndls (from alc), mp 115°; prepd from 2,3,5-trinitro-4-methyl-

aminotoluene in hot alc by treating it with a 33% alc soln of dimethylamine (Refs 2 & 4, p565)

3,5-Dinitro-2-dimethylamino-4-methylnitraminotoluene, $(H_3C)_2N.C_6H(NO_2)_2N(NO_2)(CH_3).CH_3$; mw 299.24, N 23.41%; brn-yel ndls (from alc), mp 126–27°; prepd by heating 2,3,5-trinitro-4-methylnitraminotoluene in alc with a 33% alc soln of dimethylamine in a sealed tube on the water bath for 20 mins (Refs 3 & 4, p528)
Refs: 1) Beil **13**, not found 2) Beil **13**, 142 3) Beil **13**, 143 4) F. Sommer, JPraktChem [2] **67**, 528, 565 (1903)

Di(methylaminomethyl)-methylamine. See Bis(methylaminomethyl)-methylamine in Vol 2 of Encycl, p B149-R

Dimethylaminophenol and Derivatives

Dimethylaminophenol, $(H_3C)_2N.C_6H_4.OH$, mw 137.18, N 10.21%; three forms: ortho, prisms, mp 45°; meta, mp 85°; and para, mp 78° are listed in Beil (Refs 1, 2 & 3)

Dinitrodimethylaminophenol, $(H_3C)_2N.C_6H_2(NO_2)_2.OH$, mw 227.18, N 18.50%. The following isomers are described in the literature:
4,6-Dinitro-2-dimethylaminophenol, yel powd (from glac acet ac), mp 218–20° (Ref 4, p124)
2,4-Dinitro-3-dimethylaminophenol, no description given (Ref 4, p137)
2,6-Dinitro-3-dimethylaminophenol; yel crysts, mp 191–95°; forms expl salts: Ammonium, $NH_4C_8H_8N_3O_5$, gold-yel pltlts, mp 195°; Barium, $Ba(C_8H_8N_3O_5)_2 + 1\frac{1}{2}H_2O$, ppt of orn-red crysts; Potassium, $KC_8H_8N_3O_5$, orn-red ndls; and Silver, $AgC_8H_8N_3O_5$, lt-red crysts, mp—expl violently at ca 140° (Refs 5 & 6)
4,6-Dinitro-3-dimethylaminophenol, crysts, mp 231° (Ref 4, p138 & Ref 7)
2,6-Dinitro-4-dimethylaminophenol, purple-colored ndls having a copper luster (from alc), mp 182–83° (Ref 4, p190)

Lipmann & Fleissner (Ref 6) prepd in 1885 a silver salt of dinitrocompd and found that it exploded violently at ca 140°
Refs: 1) Beil **13**, 362 & [168] 2) Beil **13**, 405, (130) & [211] 3) Beil **13**, 442, (149) & [229] 4) Beil **13**, (124, 137, 138, 190) 5) Beil **13**, 424, (137) & [216] 6) E. Lipmann & F. Fleissner, Monatsh **6**, 807–17 (1885) & JCS **50**, 235 (1886) 7) G. Leandri, AnnChim (Rome) **40**, 620 (1950) & CA **46**, 928 (1952)
NOTE: No higher nitrated derivs of Dimethylaminophenol were found in the literature thru 1966

[(3-p-Dimethylaminophenylazo)-5-azido]-asym-triazole. See Dimethylaminobenzeneazotriazole Azide

Dimethylaminophenyl-methylamine and Derivatives

Dimethylaminophenyl-methylamine $(H_3C)_2N.C_6H_4.NH(CH_3)$; mw 150.22, N 18.65% (Ref 1)

2,4,6-Trinitro-3-dimethylaminophenyl-methylnitrosamine, $(H_3C)_2N.C_6H(NO_2)_3.N(NO).CH_3$; mw 314.22, N 26.75%; yel crysts, mp 132°; prepd by reacting a warm glac acet acid soln of N,N,N',N'-tetranitro-m-phenylenediamine with nitric acid (Refs 2 & 3)

Its expl props are not reported
Refs: 1) Beil **13**, not found 2) Beil **13**, 61 3) Wurster & Morley, Ber **12**, 1815 (1879) 4) No later refs found thru 1966

Dimethylaminopropane and Derivatives

2,2-Dimethylaminopropane or *Dimethylisopropylamine*, $CH_3.C(CH_3)_2.CH_2.NH_2$; mw 87.16, N 16.07%. Blomquist attempted to prep the following compd:

2,2-Dimethyl-1,3-dinitroaminopropane, $(H_3C)_2C(CH_2NHNO_2)_2$; mw 192.18, N 29.16%. Synthesis was undertaken but not completed because of diffc in obtg 2,2-dimethyl-1,3-diaminopropane in good yields
Refs: 1) Beil – not found 2) A.T. Blomquist, "Certain Aliphatic Nitramines and Related Compounds", OSRD **4134** (1944), 21

Dimethylaminopyridine and Derivatives

Dimethylaminopyridine, $(H_3C)_2N.C_5H_4N$, mw 122.17, N 22.93%. The 2-Dimethylamino-, liq, fr p 15°, bp 200–01° (Ref 1) and 4-Dimethylamino- pltlts (from eth), mp 114° (Ref 2) derivs are known. They both form crystalline addn salts

3,5-Dinitro-2-dimethylaminopyridine, $(H_3C)_2N.C_5H_2(NO)_2.N$; mw 212.17, N 26.41%; yel ndls (from dil acet ac), mp 125–26°; sol in hot alc & 80% acet ac; v sl sol in cold w; almost insol in eth, benz & petr eth; prepd by nitrating 2-dimethylaminopyridine with 2 Moles HNO_3 & concd H_2SO_4 at low temp (Refs 3 & 4)

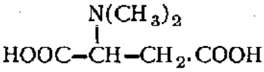
No expl props are reported

Refs: 1) Beil **22**, (629) & [325] 2) Beil **22**, [341] 3) Beil **22**, [338] 4) A.E. Chichibabin (Tschitschibabin) & I.L. Knunianz (Knunjanz), Ber **61B**, 431 (1928) & CA **22**, 1975 (1928)

NOTE: No higher nitrated derivs are found in the literature

Dimethylaminosuccinic Acid and Derivatives

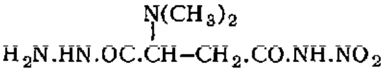
Dimethylaminosuccinic Acid (Dimethylamino-bernsteinsäure, in Ger),



$$HOOC-\overset{\overset{\displaystyle N(CH_3)_2}{|}}{C}H-CH_2.COOH$$

; mw 161.16, N 8.69%; monoclinic prisms, mp 185°; sol in dil acids & alkalies; v sl sol in cold w (Ref 1)

Dimethylaminosuccinic Acid Dihydrazide,



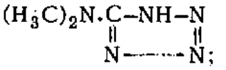
$$H_2N.HN.OC.\overset{\overset{\displaystyle N(CH_3)_2}{|}}{C}H-CH_2.CO.NH.NO_2$$

; mw 189.22, N 37.01%; ndls (from alc), mp 147°; sol in w & alc; insol in eth; obtd by warming inactive dimethylaminosuccinic acid dimethyl ester & hydrazine hydrate in alc. Forms a Hydrochloride salt, $C_6H_{15}N_5O_2+HCl$, deliquescent powd (Refs 2 & 3)

Refs: 1) Beil **4**, 486 (Parent) 2) Beil **4**, (535) (Dihydrazide) 3) T. Curtius, JPraktChem [2] **95**, 346 (1917)

1,5-Di(methylamino)-tetranitro-anthraquinone. See Tetranitro-bis(methylamino)-anthraquinone in Vol 2, p B149-L

5-Dimethylamino-α-tetrazole or **5-Dimethylamino-1-H-tetrazole,**



$$\begin{array}{l}(H_3C)_2N.C-NH-N\\ \qquad\quad\;\, \| \qquad\quad \|\\ \qquad\quad\; N\text{-----}N\end{array}$$

; mw 113.13, N 61.91%; very hygro pltlts (from ethyl acet), mp – dec ca 80°; mod sol in alc & ethyl acet; v sl sol in eth; prepd by heating 5-aminotetrazole with an excess of methyl iodide in a tube at 160–65°; not stable as a free base. It forms salts, such as

$C_3H_7N_5+HCl$, ndls (from alc), mp 241° (dec); mod sol in w;

$C_3H_7N_5+HCl+AuCl_3$, yel pltlts (from w), mp 164°;

$2C_3H_7N_5+2HCl+PtCl_4$, yel prisms (from w), mp 200°; mod sol in w; sl sol in alc; and

Picrate, $C_3H_7N_5+C_3H_6N_3O_7$, ndls, mp 203° (dec); v sl sol in cold w

Refs: 1) Beil **26**, 404 2) L.A. Burkardt & D.W. Moore, AnalChem **24**, 1583 (1952) & CA **47**, 2010 (1953) (X-ray diffraction pattern) 3) J.H. Bryden, ActaCryst **6**, 669 (1953) & CA **47**, 10947 (1953) (Unit cell dimensions & space group) 4) W.G. Finnegan et al, JOrgChem **18**, 788 (1953) & CA **48**, 7007 (1954) (Prepn & props) 5) W.L. Garbrecht & R.M. Herbst, JOrgChem **18**, 1010 (1953) & CA **48**, 8225 (1954) (Synthesis)

Di(methylamino)-toluene. See Bis (methylamino)-toluene in Vol 2 of Encycl, p B150-L

Dimethylammonium-diazidochlorocuprate, $(H_3C)_2NH_2[Cu(N_3)_2Cl]$, mw 229.14, N 42.79%, crysts, mp 141°, detonates weakly; method of prepn not given in CA

Refs: 1) Beil – not found 2) M. Straumanis & A. Cirulis, ZAnorgChem **252**, 9 (1943) & CA **38**, 3564 (1944)

Dimethylammonium-diazidonitratocuprate, $(H_3C)_2NH_2[Cu(N_3)_2NO_3]$; mw 255.68, N 43.83%, crysts, mp – explodes at 215°; method of prepn not given in CA

Refs: 1) Beil. – not found 2) M. Straumanis & A. Cirulis, ZAnorgChem **252**, 9(1943) & CA **38**, 3564(1944)

Dimethylammonium Nitrate (Code named in Germany as **DI-Salz**), $(H_3C)_2NH_2.NO_3$; mw 108.10, N 25.92%; crysts, mp – decomp explosively above 120°; unstable at temps above 100°. Was prepd during WWII in Germany by treating aqueous dimethylamine (qv) with nitric acid of sp gr 1.42, followed by vacuum distillation. Dissolves in water and is hydrolized on heating. It was produced with the intention of using it as a "substitute explosive" (Ersatzsprengstoffe) in order to combat the acute shortage of TNT and of other HE's. No military application of this salt in the US was reported
Refs: 1) Beil – not found 2) H. Walter et al, "German Development in High-Explosives", FIAT Final Rept 1035, p7 (PB Rept 78271) (1947) 3) Fedoroff et al, PATR **2510**(1958), p Ger 37-R

Dimethylammonium-pentazidocuprate, $(H_3C)_2NH_2[(N_3)_2CuN_3Cu(N_3)_2]$; mw 383.29, N 58.47%; crysts, mp – explodes at 201°; method of prepn not given in CA
Refs: 1) Beil – not found 2) M. Straumanis & A. Cirulis, ZAnorgChem **252**, 9(1943) & CA **38**, 3564(1944)

Dimethylammonium-tetrazidocuprate , $[(H_3C)_2NH_2]_2Cu(N_3)_4$, mw 323.82, N 60.56%; crysts, mp – explodes at 210°; method of prepn not given in CA
Refs: 1) Beil – not found 2) M. Straumanis & A. Cirulis, ZAnorgChem **252**, 9(1943) & CA **38**, 3564(1944)

DIMETHYLANILINES AND DERIVATIVES

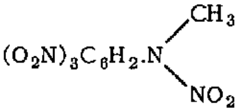
Dimethylanilines, $C_8H_{11}N$, mw 121.18, N 11.56%

Two types of these compds are known: *N,N-Dimethylaniline* or *N,N-Dimethylphenylamine*, $C_6H_5.N(CH_3)_2$. In this type, two methyl groups are attached to N of amino group

2,3-, 2,4-, 2,5-Dimethylanilines or *o-, m-, p-Xylidines*, $(H_3C)_2C_6H_3.NH_2$. In these compds two methyl groups are attached at various positions in the benzene ring

Following are the descriptions of these compds and of their derivatives:

N,N-Dimethylaniline; N,N-Dimethylphenylamine; or N,N-Dimethylaminobenzene, $C_6H_5.N(CH_3)_2$. This compd is of importance because it serves for prepn (by nitration with mixed nitric-sulfuric acid, as described, for example, in Davis, p177) of 2,4,6-Trinitrophenyl-N-methylnitramine,



$$(O_2N)_3C_6H_2.N\begin{matrix}\nearrow CH_3\\ \searrow NO_2\end{matrix}$$

commonly known as Tetryl

Properties of N,N-Dimethylaniline (DMeAn) are as follows: mw 121.18, N 11.56%; yellowish to brownish oily liquid; sp gr 0.956 at 20/4°; n_D 1.5582, fr p 2.5° bp 194.2°, fl p 61° C; insol in w; sol in alc, eth & chlf. Can be prepd by heating in an autoclave a mixture of aniline, aniline hydrochloride and methanol (free of acetone), followed by distillation and rectification (Ref 1). Laboratory prepn of pure product can be made by treating methylaniline with methyl iodide (Refs 24 & 28)

Detailed investigation of nitric acid action on DMeAn under various conditions was conducted in France, after WWII, by Lang (Ref 25). He also listed previous works on nitration, beginning with P. van Romburgh's papers in Rec **2**, 40 & 103(1883); **6**, 253(1887) and **8**, 248(1889)

Action of nitrous acid was described in Ref 19

US Armed Forces requirements for N,N-Dimethylaniline (DMeAn) intended for use in manuf of Tetryl are covered by Specification MIL-D-20305 (21 Nov 1951)

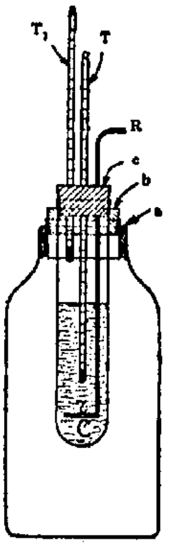
They are as follows:

1) *Color* – yellow
2) *Freezing Point* – minimum 1.9°
3) *Residue* – maximum 0.1%
4) *Solubility* – 100%
5) *Alkalinity or Acidity* – maximum 0.005%
6) *Aniline* – maximum 0.1%

Specification Tests for N,N-Dimethylaniline

1) *Color.* Determine by visual inspection

2) *Freezing Point.* Transfer a 50-ml sample to a low beaker, add 1 ml of abs alc, and heat the soln for 1 hr in an oven 95 to 100° to remove any moisture. Cool in a vacuum desiccator over sulfuric acid and transfer to 1x6 inch test tube. Close it with previously arranged three-hole cork-stopper assembly c as shown in Fig. Thru the center hole of the cork insert a "standardized" total immersion thermometer (T), graduated in 0.2° or 0.1° subdivisions and extending over the range minus 10° to plus 10°. Thru the second hole insert a "correction" thermometer (T_1) with the bulb approx level with the center of the exposed mercury column of "standardized" thermometer when the DMeAn freezes. Thru the third hole insert a stirrer (R), which consists of an aluminum or nichrome wire, ca 1.5mm diam, bent at its lower end at 90° in the shape of a ring (r). After closing the test tube with the cork assembly, as shown in Fig, adjust the "standardized" thermometer so that its bulb is at least half an inch from the bottom of the tube. Insert the assembled tube thru the stopper (b), held in place by tape (a), into wide mouth liter bottle nearly filled with ice-water slurry, so that the level of DMeAn in the tube is at least 1 inch below the level of ice-water. Stir the sample thruout the test, making 120 complete up and down motions of (R) per minute. Watch the thermometer closely (using special reading lens) to determine when the temp of the sample ceases falling and begins to rise due to heat of crystallization. Then note the temp every 15 seconds until the maximum is reached and record this as the "uncorrected freezing point", (t). Record at the same time the reading (t_1) of the "correction thermometer" and consider this value as the average temp of the exposed mercury column. Observe the number of degrees (n) of exposed mercury column of thermometer (T) and calculate the correction for emergent stem as follows:



EmStemCorrection = $0.000159\,(t - t_1)n$, where 0.000159 is the coefficient of expansion of mercury in glass

Calculate the "corrected freezing point" of the sample by adding the value calcd from the above formula to the "uncorrected freezing point" (t)

Note: For samples contg more than 1% of impurities, add salt to ice-water to cause lowering of its temp, because impure DMeAn might freeze at below 0°

3) *Residue.* Evaporate rapidly to dryness on a hot plate a 100g sample weighed in a tared evaporating dish. Cool the dish with residue in a desiccator and reweigh. Record the increase in weight of the dish and, since the sample was 100g, the increase is equal to percentage of residue

4) *Solubility.* Add a 1-ml portion of the sample to 10ml of 94–96% sulfuric acid soln, shake and observe if the oil appears completely dissolved. Pour the soln into 50ml of cold distd w and note if there is any separation of an oily or solid matter, indicating incomplete solubility

5) *Alkalinity or Acidity.* Shake thoroughly for 2 mins in a 100-ml separatory funnel a 25-g sample with 20ml neutral distd w. After allowing to stand for a few mins, draw off the lower aqueous layer into a beaker and pipette 10ml into a 100-ml Erlenmeyer. Add 3 drops of rosolic acid indicator (0.1% in 90% alc) and if the soln turns alkaline (yel),

titrate with N/10 HCl soln; if it turns acidic (red), titrate with N/10 NaOH soln

$$\% \text{ Alkalinity, as NaOH} = \frac{8.001 \times AB}{C}$$

$$\% \text{ Acidity, as HCl} = \frac{7.293 \times AB}{C}$$

where: A = mls of KOH or HCl solns used
B = their normality
C = wt of sample

6) *Aniline*. Prepare Ca oxychloride soln by shaking not less than 35g of solid (contg not less than 30% of available chlorine) with200ml distd w until no further soln takes place. After allowing the mixt to settle, filter and use the filtrate as indicated below: Pipet 5-ml DMA samples into each of the two 25-ml glass tubes (such as used in colorimeters) and a 5-ml portion of pure DMA contg 0.1% aniline (serving as a blank) into the 3rd tube. Add to each tube 15ml of ether and 5ml of the above oxychloride soln. Stopper the tubes, shake thoroughly and allow to stand for 30–35 mins. Compare the colors produced and , if the colors in tubes of each of the two samples are not darker than that in the blank, the sample is approved

4-Nitroso-N,N-dimethylaniline, $(H_3C)_2N.C_6H_4.NO$, mw 150.18, N 18.65%; grn pltlts (from eth or petr eth + benzol), dk-grn pltlts + 1 C_6H_6 (from benzol), mp 85°; d 1.145 at 20°; can be prepd by reaction of nitrosoguanidine & dimethylaniline in concd HCl, and by other methods

It forms many salts and addn compds, some of which decomp on heating; for example, one salt, $3C_8H_{10}N_2O + 2SiF_4$, yel amorph powd, deflagrates or explodes mildly on heating above 150° (Refs 14 & 18)

Mononitro-N,N-dimethylanilines, $(H_3C)_2N.C_6H_4.NO_2$, mw 166.18, N 16.86%. Three isomers are known, none of them is explosive:

2-Nitro-N,N-dimethylaniline, yel-orn oil, sp gr 1.794 at 20°/4, mp minus 20°, bp 151–53° at 30mm. Prepn & other props are in Ref 2

3-Nitro-N,N-dimethylaniline, red crysts, sp gr 1.313 at 17°, mp 60–61°, bp 280–85°. Prepn & other props are in Ref 3

4-Nitro-N,N-dimethylaniline, yel ndls, mp 163–65°. Prepn & other props are in Ref 4

Dinitro-N,N-dimethylanilines, $(H_3C)_2N.C_6H_3(NO_2)_2$, mw 211.18, N 19.90%. Four isomers are known and they may be considered as weak expls:

2,4-Dinitro-N,N-dimethylaniline, yel crysts, sp gr 1.476 at 17°; mp 87°. Can be prepd by condensation of N,N-dimethylaniline with 2,4-dinitrochlorobenzene or by nitration of dimethylaniline in glac acet acid. Its heat of combstn at C_v & 18° is 1053.4kcal/mole and heats of formation 6.4 at C_v and 10.75 at C_p (Refs 5 & 27)

2,5-Dinitro-N,N-dimethylaniline, yel crysts, sp gr 1.395 at 18°, mp 112°. Can be prepd by nitrating 3-nitrodimethylaniline with $NaNO_2$ & dil HCl, and by several other methods (Ref 6)

2,6-Dinitro-N,N-dimethylaniline, orn-yel ndls (from dil alc), mp 78°. Can be prepd by reacting 2-chloro-1,3-dinitrobenzene with dimethylaniline in dil alc (Ref 7)

3,4-Dinitro-N,N-dimethylaniline, crysts, sp gr 1.423, mp 174–76°. Can be prepd by nitrating 3-nitrodimethylaniline with 20% HNO_3 (Ref 8)

Trinitro-N,N-dimethylanilines, $(H_3C)_2N.C_6H_2(NO_2)_3$, mw 256.18, N 21.87%, N (of NO_2) 16.44%. Three isomers are known, of which 2,4,6-trinitro was examined as a possible military expl:

2,3,4-Trinitro-N,N-dimethylaniline, brownish crysts, sp gr 1.552 at 17°; mp 154°. Can be prepd along with the 2,4,5-trinitro deriv by warming of 3-nitrodimethylaniline with 70% nitric acid at 40°, or by nitrating 3,4-dinitro deriv with nitric acid (d 1.37 to 1.40)(Ref 9)

2,4,5-Trinitro-N,N-dimethylaniline, orn-red crysts (from acet or benz), mp 198°, sp gr 1.585 at 17°. Can be prepd by heating N,N-dimethyl-N'-phenyl-4,6-dinitro-1,3-phenylenediamine with aniline & alc in a tube at 110–20°, or by other methods (Ref 10)

2,4,6-Trinitro-N,N-dimethylaniline; 2,4,6-Trinitro-N,N-dimethylphenylamine; or **N,N-Dimethylpicramide,** yel tablets, mp 138°; expl at higher temps; power and brisance – less than picric acid; impact sensitivity – 2kg positive at 95cm (comparable to TNT). Can be prepd by interaction of picryl chloride with dimethylaniline (Refs 11, 15, 21 & 23)

2,3,4,6(or 2,4,5,6)-Tetranitro-N,N-dimethylaniline or **Tetralita No 1,** $(H_3C)_2N.C_6H(NO_2)_4$, mw 301.18, N 23.26%, N (of NO_2) 18.60%, mp 125–28°; expl on heating; mp 153° (Ref 17). No other props are reported in the refs. Can be prepd by dissolving N,N-dimethylaniline in concd sulfuric acid and pouring the cooled soln slowly into concd nitric acid at 30°. It has been suggested in Spain as a military expl. The name *Tetralita No 1* is given in the book of Vivas, Feigenspan and Ladreda (Ref 24, Vol 2, p294) to Tetryl, which they also call *Tetranitrometilanilina* (Refs 12, 16, 17 & 22, p T3)

Pentanitro-N,N-dimethylaniline, Pentanitro-N,N-dimethylphenylamine, Pentralita or **Tetralita No 2,** $(H_3C)_2N.C_6(NO_2)_5$, mw 346.18, N 24.28%; crysts, mp 125–29°, expl violently at higher temp. It was prepd by dissolving N,N-dimethylaniline in concd sulfuric acid and pouring the liquid into concd nitric acid, preheated to 120°; the reaction was accompanied by a copious evolution of nitrogen oxides. The resulting crysts in the slurry were separated from the waste acid by centrifuging (Refs 20 & 24)
Note: Accdg to Vivas et al (Ref 24, p298), some authorities consider that it is doubtful that all five hydrogens in the ring could be replaced by NO_2 groups, because of difficulty of direct nitration of the group in meta position

Pentanitrocompd was used in Spain as a military explosive, under the name of *Pentralita* or *Tetralita No 2* (Refs 20 & 22,p P4)

References for N,N-Dimethylaniline and Derivitives: 1) Beil **12**, 141, (151) & [82] 2) Beil **12**, 690, (340) & [369] 3) Beil **12**, 701, (345) & [372] 4) Beil **12**, 714, (350) & [386] 5) Beil **12**, 749, (361) & [406] 6) Beil **12**, 757, (365) & [413] 7) Beil **12**, (365) 8) Beil **12**, 758, (365) & [414] 9) Beil **12**, 763 & [419] 10)Beil **12**, 763, (367) & [420] 11) Beil **12**, 764, (368) & [420] 12) Beil **12**, [428] 13) Beil **12** – not found (Pentanitro-N,N-dimethylaniline) 14) Beil **12**, 677, (337) & [364] 15) W. Will, SS **1**, 209(1906) (Prepn and props of 2,4,6-Trinitro-N,N-dimethylaniline) 16) Anon, SS **10**, 295(1915) & CA **10**, 390 (1916) (Manuf in Spain of tetra- and pentanitrodimethylanilines and their uses as military expls under the names of Tetralita No 1 and Tetralita No 2 or Pentralita) 17) A. Forster & W. Coulson, JChemSoc **121**, 1988(1922) & CA **17**, 76(1923) (N,N-Dimethyl-2,3,4,6-tetranitroaniline) 18) S. Secareanu, Ber **64B**, 834(1931) & CA **25**, 4127(1931) (Nitroso-N,N-dimethylaniline) 19) J.C. Earl & A.W. Mackney, JProcRoySoc NS Wales **67**, 419(1934) & CA **28**, 4043(1934) (Action of nitrous acid on N,N-dimethylaniline) 20) Sancho (1941), 170–71 (Prepn of Pentanitrodimethylaniline) 21) A.H. Blatt & F.C. Whitmore, OSRD **1085**, 105(1942) (2,4,6-Trinitro-N,N-dimethylaniline) 22) Clift & Fedoroff, Vol **2**(1943), p P4 (Pentanitrodimethylaniline); p T3 (Tetranitrodimethylaniline) 23) A.H. Blatt, OSRD **2014**(1944) (2,4,6-Trinitrodimethylaniline) 24) Vivas, Feigenspan & Ladreda, Vol **2**(1946), 297–98 (Pentralita) 25) F.M. Lang, MP **30**, 373–92(1948) (Action of nitric acid on N,N-dimethylaniline) 26) L. Horner & C. Betzel, Ann **579**, 175–92 (1953) & CA **48**, 2625(1954) (N,N-Dimethyl-2,4,6-trinitroaniline) 27) L. Médard & M. Thomas, MP **38**, 49(1956) (Prepn and props of N,N-Dimethyl-2,4-dinitroaniline) 28) CondChemDict (1961), 393-R (N,N-Dimethylaniline) 29) No later refs on Tetranitro- and Pentanitro derivs were found in the literature thru 1966

Dimethylanilines or Xylidines, also called *Aminoxylenes, Aminodimethylbenzenes* or *Dimethylphenylamines,*
$(H_3C)_2C_6H_3.NH_2$, mw 121.18, N 11.56%

Following six isomers exist:

2,3-Dimethylaniline or *(vic)o-Xylidine,* liq, sp gr 0.991 at 15°, mp < minus 15°, bp 223–224°; sl sol in w; sol in alc & eth (Refs 1 & 14)

3,4-Dimethylaniline or *(uns)o-Xylidine,* crysts, sp gr 1.076 at 17.5°, mp 49–50°, bp 224–226°; v sl sol in w; sol in petr eth (Ref 2) (Not in Ref 14)

2,6-Dimethylaniline or *(vic)m-Xylidine,* liq, sp gr 0.980 at 15°, fr p 10–12°, bp 216–217°; insol in w; sl sol in alc & eth (Refs 3 & 14)

2,4-Dimethylaniline or *(uns)m-Xylidine*, liq, sp gr 0.974 at 20°; bp 213–214°; v sl sol in w; sol in alc & eth (Refs 4 & 14)
3,5-Dimethylaniline or *(sym)m-Xylidine*, oil, sp gr 0.972 at 20/4°, bp 221–222°; solubilities not given (Ref 5) (Not in Ref 14)
2,5-Dimethylaniline or *p-Xylidine*, oil, sp gr 0.980 at 15°; fr p 15.5°; bp 215° at 739mm; v sl sol in w; sl sol in alc; sol in eth (Refs 6 & 14)

Azidodimethylaniline. See N,N-Dimethylaniline-p-Azide which follows

Dinitrodimethylaniline, $(H_3C)_2C_6H(NO_2)_2.NH_2$; mw211.18, N 19.90%. The following isomers are known:
4,5-Dinitro-2,3-dimethylaniline, yel ndls (from alc), mp 172°; sol in acet & ethyl acetate (Ref 7, p480)
4,6-Dinitro-2,3-dimethylaniline, yel ndls (from alc), mp 161°; readily sol in cold acet, warm eth acet, chlf & benz (Ref 7, p479)
5,6-Dinitro-2,3-dimethylaniline, orn ndls (from alc), mp 143°; sol in acet, eth acet, warm benz & warm chlf (Ref 7, p479)
2,6-Dinitro-3,4-dimethylaniline, orn-red ndls (from alc), mp 143°; sol in chlf, benz & eth acet (Ref 7, p481)
5,6-Dinitro-3,4-dimethylaniline, dk, orn-red ndls (from acet), mp 212°; v sl sol in alc, benz & chlf (Ref 7, p482)
3,5-Dinitro-2,6-dimethylaniline, yel pltlts (from alc), mp 177° (Ref 4)
3,5-Dinitro-2,4-dimethylaniline, yel ndls or yel prisms (from dil acet ac), mp 191–93°; sl sol in alc, eth & chlf (Ref 7, pp1130 & 613)
3,6-Dinitro-2,4-dimethylaniline, crysts (from alc + H_2O), mp 145° (Ref 7, p1130)
5,6-Dinitro-2,4-dimethylaniline, yel ndls, mp 115°; sl sol in alc (Ref 7, p1130)
2,4-Dinitro-3,5-dimethylaniline, crysts, mp 94° (Ref 7, p1132)
2,6-Dinitro-3,5-dimethylaniline, yel crysts, mp 101° (Ref 7, p1132)
3,4-Dinitro-2,5-dimethylaniline, crysts, mp 172° (Ref 7, p490)
3,6-Dinitro-2,5-dimethylaniline, crysts, mp 140° (Ref 7, p490)
4,6-Dinitro-2,5-dimethylaniline, yel ndls (from acet ac), mp 202–06° (Ref 7, pp1141 & 490)

Other props & methods of prepn are given in the Refs

2,4-Dinitro-3,6-dimethyl-nitraniline; 4,6-Dinitro-2,5-dimethylphenyl-nitramine, $(H_3C)_2C_6H(NO_2)_2.NH(NO_2)$; mw 256.18, N 21.87%; ndls (from alc + water), mp 130° (dec); mod sol in alc, acet acid & eth; sl sol in w; was obtd by reaction of 5-amino-p-xylol-2-sulfonic acid with nitric acid at 5°, in addn to a product 3,5-dinitro-p-xylol-2-diazonium nitrate

The compd forms a *Potassium salt*, $KC_8H_7N_4O_6$, ndls (from alc); very sl sol in w; and a *Silver salt*, $AgC_8H_7N_4O_6$, ndls (from hot w) (Ref 8)

2,4,6-Trinitro-3,5-dimethyl-1-aniline or **2,4,6-Trinitro-m-xylidene,** $(H_3C)_2C_6(NO_2)_3.NH_2$, mw 256.18, N 21.87%; yel crysts, mp 206°; can be prepd by heating 2,4,6-trinitro-5-methoxy-1,3-dimethylbenzene with alcoholic NH_3 in a tube, or from 5-bromo-2,4,6-trinitro-m-xylidine with alc NH_3 at 230° (Ref 9)
Tetranitrodimethylaniline or *Tetranitro-m-xylidene*, $C_8H_7N_5O_8$, not found in the literature thru 1966

Pentanitro-m-xylidene; 2,4,6-Trinitro-3,5-dimethyl-1-dinitroaniline or **2,4,6-Trinitro-3,5-dimethyl-1-dinitroaminobenzene,** $(H_3C)_2C_6(NO_2)_3.N(NO_2)_2$, mw 346.18, N 24.28%; crysts, mp – explodes at high temp

This compd is listed by Duin & Brackmann (Ref 11) as a brisant nitro expl, but its method of prepn is not given in the abstract. It was considered as not having all of the desirable props for use in ammunition

Refs for Xylidines: 1) Beil **12**, 1101 2) Beil **12**, 1103 3) Beil **12**, 1107 4) Beil **12**, 1111 5) Beil **12**, 1131 6) Beil **12**, 1135 7) Beil **12**, 1130, 1132, 1141, (479, 480, 482, 490) & [613] 8) Beil **16**, 675 9) Beil **12**, 1133 (Trinitro) 10) Beil **12** – not found (Pentanitro) 11) C.F. vanDuin & K. Brackmann, ChemWeekblad **16**, 501–09 (1919) & CA **13**, 1638 (1919) 12) C.F. vanDuin & B.C. vanLennep, Rec **39**, 145–77 (1920) & CA **14**, 2708 (1920) 13) Clift & Fedoroff, Vol **2** (1943), p T12 14) CondChemDict (1961), 1234-R & 1235-L (Xylidines)

N,N-Dimethylaniline-p-Azide, $(H_3C)_2N.C_6H_4.N_3$; mw 162.19, N 34.55%; yel leaflets, mp 40°, explodes at higher temp. Horner & Gross (Ref 2) report its prepn: N,N-Dimethyl-p-aniline (13.6g) in 2N sulfuric acid is diazotized with 7g $NaNO_2$ at 0–5°, treated with 2g Cu powd and then with 7g NaN_3 in 50cc H_2O, the mixt kept 2 hrs, excess 2N NaOH added, kept 5 hrs at 0°, steam distd, the distillate extd with acet, and the extract dried & evapd, giving the product. Ugi et al (Ref 3) report the prepn as follows: N,N-Dimethylaniline HCl salt (0.050 mole) in 50cc 2N HCl dild with 20–30g crushed ice is treated while stirring with 3.70g $NaNO_3$ in 15cc water, neutralized with $CaCO_3$, treated with 4.0g NaN_3 in 15cc water, extd with acet, the ext evapd, and the residue crystd or distd to give the product. Ugi also studied its rate of reduction with alk arsenite

Refs: 1) Beil – not found 2) L. Horner & A. Gross, Ann **591**, 133–34(1955) & CA **50**, 2496(1956) 3) I. Ugi et al, ChemBer **91**, 2330–36(1958) & CA **53**, 6123(1959) 4) C.N.R. Rao et al, JSciIndRes(India) **17B**, 504–05(1958) & CA **53**, 13774(1959) (UV spectrum)

N,N-Dimethylaniline-p-azido-picrate, $(H_3C)_2N.C_6H_4.N_3 + C_2H_2N_3O_7$; mw 391.30, N 25.06%; yel ndls (from MeOH), mp 128°, probably explodes at higher temp. Prepn: to the residue from evapn (in darkness) of an alc-chlf soln of N,N-Dimethylaniline-p-Azide, a few cc of MeOH and excess Picric Acid in MeOH are added, the picrate is dissolved in acet, treated with animal C, the soln freed of PA by aq Na_2CO_3, and the acet soln is dried & evapd. Yield 78%

Refs: 1) Beil – not found 2) S. Maffei & A.M. Rivolta, Gazz **84**, 750–52(1954) & CA **49**, 13925(1955) 3) L. Horner & A. Gross, Ann **591**, 134(1955) & CA **50**, 2496 (1956)

N,N-Dimethylaniline-4-diazonium Hydroxide and Derivatives

N,N-Dimethylaniline-4-diazonium Hydroxide or *N,N-Dimethylaminobenzene-4-diazonium Hydroxide,* $(H_3C)_2N.C_6H_4.N(\vdots N).OH$. A hypothetical compd known in the form of its salts, some of which are expl:

Chloride, $(H_3C)_2N.C_6H_4.N(\vdots N).Cl$, mw 183.64, N 22.88%; pale-yel crysts (from alc + eth), mp – puffs off at ca 130°; sol in cold w & in alc; insol in eth; decompd by hot w. Hantzsch (Refs 1 & 2) prepd it by the low-temp diazotization of N,N-dimethyl-p-phenylenediamine with Na nitrite, while Stolle (Ref 4) treated N,N-dimethyl-p-phenylenediamine hydrochloride with amyl nitrite in alcoholic medium

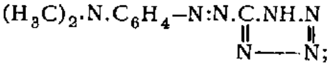
Perchlorate, $(H_3C)_2N.C_6H_4.N(\vdots N).ClO_4$, mw 247.64, N 16.98%; crysts, mp – expl on heating; insol in w and ordinary org solvents. Was prepd by Hofmann et al (Ref 3) on treating N,N-dimethyl-p-phenylenediamine hydrochloride with Na nitrite and perchloric acid of 20% strength

Refs: 1) Beil **16**, 602, (371) 2) A. Hantzsch, Ber **35**, 896(1902) 3) K.A. Hofmann et al, Ber **43**, 2628(1910) 4) R. Stolle, Ber **45**, 2681(1912)

N,N-Dimethylaniline-pentazido-dicuprate, $(H_3C)_2N.C_6H_4.NH[(N_3)_2CuN_3Cu(N_3)_2]$, mw 472.31, N 50.42%; crysts, explodes at 174°; dec even in alc, but is stable in the presence of Dimethylaniline Azide or in NH_3; was obtd by dissolving $Cu(N_3)_2$ in N,N-Dimethylaniline Azide (Ref 2)

Refs: 1) Beil – not found 2) A. Cirulis & M. Straumanis, Ber **76B**, 825(1943) & CA **38**, 1972(1944)

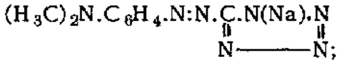
[(p-N,N-Dimethylanilino)-(tetrazolyl-5)]-diazine, called by Benson *Tetrazole-5-azo-4-N,N-dimethylaniline,*



$$\begin{array}{llll}(H_3C)_2.N.C_6H_4{-}N{:}N. & C & .NH. & N \\ & \| & & \| \\ & N & \text{———} & N;\end{array}$$

mw 217.23, N 45.14%, pale reddish lflts (from alc), mp – begins to decomp ca 150°, expl ca 155°; diff sol in w & in eth; sl more sol in alc; possesses sl basic props. Was prepd by mixing dil solns of equimolecular quantities of 5-diazotetrazole (qv) and N,N-dimethylaniline, followed by addn of Na acetate (Refs 1, 2 & 3)

It is an expl which forms explosive salts with strong bases, such as with NaOH, *Sodium Salt,*



$$\begin{array}{l}(H_3C)_2N.C_6H_4.N{:}N.\overset{}{C}.N(Na).N\\ \hspace{11em}\| \hspace{5.5em} \| \\ \hspace{10.8em}N\text{———————}N;\end{array}$$

mw 239.22, N 40.99%; carmine red shiny crysts, explodes on heating (Ref 2)

Refs: 1) Beil **26**, 593 2) J. Thiele, Ann **270**, 61(1892) & **303**, 74(1898) 3) F.R. Benson, ChemRevs **41**, 8(1947)

Dimethylanisole and Derivatives

Dimethylanisole (called Methoxyxylol or Dimethylanisol in Ger), $H_3CO.C_6H_3(CH_2)_2$; mw 136.19. Six isomers are found in the literature:

2,3-Dimethylanisole, crysts, mp 29°, bp 199° & 85° at 18mm press (Ref 1)
2,4-Dimethylanisole, liq, bp 192°, d 0.974 at 16°, n_D 1.5190 at 16° (Ref 2)
2,5-Dimethylanisole, oil liq, bp 186–88°, d 0.9693 at 12.8°, n_D 1.5182 at 12.8° (Ref 3)
2,6-Dimethylanisole, col oil, bp 181–83°, d 0.9619 at 13.6°, n_D 1.5053 at 13.6° (Ref 4)
3,4-Dimethylanisole, liq, bp 200–01°, d 0.9744 at 13.7°, n_D 1.5198 at 13.7° (Ref 5)
3,5-Dimethylanisole, col oil, bp 194°, d 0.9569 at 20°, n_D 1.5110 at 20° (Ref 6)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **6**, 480, [453] & {1723} 2) Beil **6**, 486, (241), [459] & {1744} 3) Beil **6**, 494, (245),[466] & {1772} 4) Beil **6**, 485, [457] & {1737} 5) Beil **6**, 481, [455] & {1727} 6) Beil **6**, 493, (244), [462] & {1756}

4-Nitroso-2,6-dimethylanisole, $H_3CO.C_6H_2(NO)(CH_3)_2$; mw 165.19, N 8.48%; yel pltlts, mp 51°; was prepd by reaction of 4-nitroso-2,6-dimethylphenol with diazomethane in eth (Refs 1 & 2)

Refs: 1) Beil **6**, {1740} 2) F. Fichter & R. Gunst, Helv **22**, 267, 274(1939)

Mononitrodimethylanisole, $H_3CO.C_6H_2(NO_2)(CH_3)_2$; mw 181.19, N 7.73%. The following isomers are known:

5-Nitro-2,4-dimethoxyanisole, ndls (from alc), mp 56–57°; sl sol in alc & eth (Ref 1)
6-Nitro-2,4-dimethoxyanisole, prisms (from eth), bp 268–69° (Ref 2)
3-Nitro-2,5-dimethoxyanisole, ndls (from MeOH), mp 62–62.5°; sol in w (Ref 3)
4-Nitro-2,6-dimethylanisole, lt yel ndls (from alc), mp 91–92° (Ref 4)
6-Nitro-3,4-dimethoxyanisole, yel crysts (from alc), mp 79° (Ref 5)
2-Nitro-3,5-dimethoxyanisole, exists as tablets (from MeOH), mp 43–46° & as ndls (from dil MeOH), mp 35–35.5° (Ref 6)
4-Nitro-3,5-dimethoxyanisole, ndls (from dil MeOH), mp 50–53° (Ref 6)

Other props & methods of prepn are given in the Refs. No expl props are reported

Refs: 1) Beil **6**, 491 2) Beil **6**, 490 & [461] 3) Beil **6**, (246) 4) Beil **6**, {1740} 5) Beil **6**, (240) & {1731} 6) Beil **6**, {1765}

Dinitrodimethylanisole, $H_3CO.C_6H(NO_2)_2(CH_3)_2$; mw 226.19, N 12.39% Two isomers are known:

2,4-Dinitro-3,5-dimethylanisole, crysts (from alc), mp 172–75°; was prepd by heating 2,4-dinitro-3,5-dimethylphenol with methyl iodide & aq NaOH (Ref 1)
2,4-Dinitro-3,6-dimethylanisole or *4,6-Dinitro-2,5-dimethylanisole,* almost colorless ndls (from alc), mp 60°; was obtd by reaction of trinitro-p-xylol with Na methylate in MeOH (Ref 2)

Other props are found in the Refs. No expl props are reported

Refs: 1) Beil **6**, {1767} 2) Beil **6**, 497

Trinitrodimethylanisole, $H_3CO.C_6(NO_2)_3(CH_3)_2$; mw 271.19, N 15.50%. Two isomers are known:

3,4,6-Trinitro-2,5-dimethylanisole, crysts, mp 146°; was prepd by heating 2,4-dinitro-3,6-dimethylanisole with HNO_3 & H_2SO_4 (Ref 1)
2,4,6-Trinitro-3,5-dimethylanisole, ndls (from alc), mp 124–27°; obtd, with 2,4-dinitro-3,5-dimethylanisole, by nitrating 3,5-dinitro-3,5-dimethylanisole with a mix of concd HNO_3 & H_2SO_4 at –5° (Refs 2 & 3)

Their expl props are not reported in the Refs

Refs: 1) Beil **6**, 497 2) Beil **6**, 493 & {1767} 3) F.M. Rowe et al, JSocChemInd **49**, 469T, 473T(1930) & CA **25**, 930(1931)

Dimethylazobenzene. See Azotoluene in Vol 1, p A660-L

1,1'-Dimethyl-5,5'-azotetrazole or Di(methyltetrazolyl)-diazene. See Bis(1-methyl-1,2,3,4-tetrazolyl-5)-diazene in Vol 2 of Encycl, p B151-L and: M.M. Williams et al, JPhysChem **61**, 263(1957). They give heats of combstn 770.5kcal/mole for trans compd and 769.8 for cis compd. Heats of formation minus 189.3 for trans- and 188.6 for cis compd

Williams et al also examined: **2,2'-Dimethyl-5,5'-azotetrazole** and determined heat of combstn as 761.5 and heat of formation minus 180.3

Dimethylazoxybenzene. See Azoxytoluene in Vol 1, p672-L

Dimethylazoxyethane. See Azoxypropane in Vol 1, p A671-R

Dimethylbenzaldehyde and Derivatives

Dimethylbenzaldehyde (called 1'-Oxo-trimethyl-benzol, Dimethylbenzaldehyd or Dimethyl-1-formyl-benzol in Ger), $(H_3C)_2C_6H_3.CHO$; mw 134.17. The following isomers are known:

2,3-Dimethylbenzaldehyde, isolated as Oxime, crysts, mp 80–82° & as Semicarbozone, crysts, mp 222° (Ref 1)

2,4-Dimethylbenzaldehyde, liq, fr p 9°, bp 212–15° at 710mm press (Ref 2)

2,5-Dimethylbenzaldehyde, liq, bp 220° (Ref 3)

2,6-Dimethylbenzaldehyde, liq, fr p 30–31°, bp 226–28° at 742mm press (Ref 1)

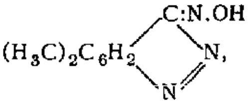
3,4-Dimethylbenzaldehyde, liq, bp 223–25° (Ref 4)

3,5-Dimethylbenzaldehyde, liq, fr p 9°, bp 220–22° (Ref 4)

Other props & methods of prepn are found in Beil

Refs: 1) Beil **7**, {1071} 2) Beil **7**, 310, (165), [240] & {1072} 3) Beil **7**, 311, (166) & {1072} 4) Beil **7**, 312, [240] & {1073}

2-Azido-3,5-dimethylbenzaldehyde, $(H_3C)_2C_6H_2(N_3).CHO$; mw 175.19, N 23.99%; ndls (from petr eth), mp 33.5–34°; volatile on steam bath; sol in org solvents; v sl sol in w; obtd from the oxime of 3-oxo-5,7-dimethylindiazene,



with NaOH and distilling on a steam bath. The Azide on oxidation with $KMnO_4$ yields 2-Azido-3,5-dimethylbenzoic Acid. Expl props of the Azide are not reported (Refs)

Refs: 1) Beil **7**, 313 2) E. Bamberger & Demuth, Ber **34**, 1317(1901) 3) No later refs found thru 1966

Mononitrodimethylbenzaldehyde, $(H_3C)_2C_6H_2(NO_2).CHO$; mw 179.17, N 7.82%. Four isomers are found in the literature:

5-Nitro-2,4-dimethylbenzaldehyde, yel ndls (from petr eth), mp 81°; obtd by nitrating the 2,4-isomer with HNO_3 & concd H_2SO_4 (Ref 1, p311)

6-Nitro-2,5-dimethylbenzaldehyde, yel ndls or pltlts (from alc), mp 120°; prepd from reaction of the 2,5-isomer with KNO_3 & concd H_2SO_4 in the cold (Ref 2)

2-Nitro-3,5-dimethylbenzaldehyde, ndls, mp 102–03°; sol in alc & petr eth; obtd by nitrating the 3,5-dimethyl isomer with KNO_3 & concd H_2SO_4 in the cold (Ref 1, p313 & Ref 3)

4-Nitro-3,5-dimethylbenzaldehyde, col crysts (from petr eth), mp 42–44°. Its prepn is described by Steiner & Sorkin (Ref 3): To 16g nitromesitylene, $(H_3C)_2C_6H_2(NO_2).CH_3$, in 48g CS_2 30g CrO_2Cl_2 in 40ml CS_2 is added with cooling & stirring; the condensation product filtered after 5 days, dried, dissolved in one liter water, extracted with ether, and the concd ether soln is treated with $NaHSO_3$ giving 8.8g crysts hydrolized with 10% H_2SO_4 & 3.4g of a mixt of the 2-Nitro & 4-Nitro derivs. Recrystallization of the mixt from petr eth gave 0.8g of the 2-Nitro deriv, and from the mother liquors 1.9g of the 4-Nitro deriv. See also Ref 4

No expl props of the Mononitro derivs are reported

Refs: 1) Beil **7**, 311, 313 2) Beil **7**, (166) 3) K.U. Steiner & E. Sorkin, Helv **35**, 2490–91(1952) & CA **47**, 12303(1953) 4) R.F. Stockel, ChemInd(London) **1963**, 613–14 & CA **58**, 13834(1963) (Novel reduction of 4-Nitro-3,5-dimethylbenzaldehyde)

Dinitrodimethylbenzaldehyde, $(H_3C)_2C_6H(NO_2)_2.CHO$; not found in Beil nor in CA thru 1966

Trinitrodimethylbenzaldehyde, $(H_3C)_2C_6(NO_2)_3.CHO$; not found in Beil nor in CA thru 1966

Dimethylbenzene and Derivatives

Dimethylbenzene or Xylene, $C_6H_4(CH_3)_2$, mw 106.16. Three isomers are known:
1,2(or o-)Dimethyl-benzene, col liq, sp gr 0.880 at 20/4°, fr p minus 25.2°, bp 144.4°; insol in w; sol in alc & eth (Ref 1)
1,3(or m-)Dimethylbenzene, col liq, sp gr 0.864 at 20/4°, fr p minus 47.9°, bp 139.1°; insol in w; sol in alc & eth (Ref 2)
1,4(or p-)Dimethylbenzene, plts, sp gr 0.861 at 20/4°, mp 13.3°, bp 138.4°; insol in w; sol in alc; v sol in eth (Ref 3)

Other props & methods of prepn are found in Beil
Refs: 1) Beil **5**, 362, (179), [281] & {807} 2) Beil **5**, 370, (182), [287] & {823} 3) Beil **5**, 382, (185), [296] & {845}

Azidodimethylbenzene, $N_3.C_6H_3(CH_3)_2$; mw147.18, N 28.55%. Four isomers are known:
2-Azido-1,3-dimethylbenzene or *4-Azido-m-xylene,* compd prepd by Smolinsky (Ref 6) by a procedure which converts the amine to the corresponding azide, but no details of its props are given
4-Azido-1,3-dimethylbenzene, yel-brn aromatic smelling oil, bp 87–88°; was obtd from 4-amino-1,3-dimethylbenzene by diazotizing in HCl soln, followed by treating the diazonium chloride salt with concd aq NH_3 (Refs 1, 4 & 5). No expl props are reported
1'-Azido-1,4-dimethylbenzene or p-Xylyl Azide, aromatic liq, bp 94° at 12mm press, deflgr on rapid heating; was prepd by warming 1'-Nitrosohydrazine-1,4-dimethylbenzene, $CH_3.C_6H_4.CH_2.N(NO_2).NH_2$, with 10% H_2SO_4 on a water bath and distilling with steam (Ref 2)
2-Azido-1,4-dimethylbenzene, yel oil, bp 90° at 15mm press, volatile with steam; was obtd by reacting 2,5-dimethylphenylhydrazine with $NaNO_2$ & dil HCl in the cold (Refs 3 5). No expl props are reported
Refs: 1) Beil **5**, [296] 2) Beil **5**, 389 & (188) 3) Beil **5**, [303] 4) E. Bamberger, Ann **424**, 233(1921) & CA **15**, 3986–87(1921) 5) E. Bamberger, Ann **443**, 192(1925) & CA **19**, 2341(1925) 6) G. Smolinsky, JOrgChem **26**, 4110(1961) & CA **56**, 8606–07(1962)

Mononitroso-dimethylbenzene, $ON.C_6H_3(CH_3)_2$; mw 135.16, N 10.36%. Six isomers are known:
3-Nitroso-1,2-dimethylbenzene, wh ndls, mp 91–91.5°; sol in benz & warm alc; sl sol in eth & chlf; v sl sol in petr eth (Ref 1)
4-Nitroso-1,2-dimethylbenzene, yel-brn ndls (from alc), mp 44–45°; mod sol in petr eth; sl sol in acet & warm alc (Ref 1)
2-Nitroso-1,3-dimethylbenzene, col ndls, mp 141.5°(dec); sol in benz & chlf; sl sol in warm alc & acet; v sl sol in eth; diffc sol in petr eth (Ref 2)
4-Nitroso-1,3-dimethylbenzene, wh prisms (from alc), mp 42.5°; sol in petr eth & eth; sl sol in acet & chlf; vol in steam (Ref 3)
5-Nitroso-1,3-dimethylbenzene, crysts, mp 59°(Ref 4)
2-Nitroso-1,4-dimethylbenzene, wh ndls (from alc), mp 101.5°; readily sol in eth & petr eth (Ref 5)

Other props & methods of prepn are found in the Refs
Refs: 1) Beil **5**, 367 2) Beil **5**, 377 & [294] 3) Beil **5**, 377 & {840} 4) Beil **5**, {841} 5) Beil **5**, 387 & {861}

Mononitrodimethylbenzene, $O_2N.C_6H_3(CH_3)_2$; mw 151.16, N 9.27%. Six isomers are known:
2-Nitro-1,2-dimethylbenzene, almost colorless ndls (from alc), mp 15°, bp 240°, d 1.4020 at 20°, n_D 1.5441 at 20°(Ref 1)
4-Nitro-1,2-dimethylbenzene, yel prisms (from alc), mp 28.5–30°, bp (decomp); sol in common org solvs (Ref 2)

2-Nitro-1,3-dimethylbenzene, liq, crysts on cooling below RT, bp 221–22°, d 1.12 at 15° (Ref 3)

4-Nitro-1,3-dimethylbenzene, liq, fr p 9°, bp 239–44°, d 1.135 at 15° (Ref 3)

5-Nitro-1,3-dimethylbenzene, col ndls (from alc), mp 71–75°, bp 273° at 739mm press (Ref 4)

2-Nitro-1,4-dimethylbenzene, lt yel liq, bp 239–41°, d 1.132 at 15°, n_D 1.5415 at 20° (Ref 5)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **5**, 367, (181), [286] & {821} 2) Beil **5**, 368, (181), [286] & {821} 3) Beil **5**, 378, (184), [294] & {841} 4) Beil **5**, 378, [295] & {841} 5) Beil **5**, 387, (187), [302] & {862}

Nitrosonitrodimethylbenzene,
$O_2N.C_6H_2(NO)(CH_3)_2$; mw 180.16, N 15.55%

Four isomers are known:

5-Nitroso-4-nitro-1,3-dimethylbenzene, no description given in Beil; was prepd by reaction of H_2SO_5 on 4-nitro-5-amino-1,3-dimethylbenzene (Ref 1, p843 & Ref 3)

6-Nitroso-4-nitro-1,3-dimethylbenzene, crysts (from alc), mp 108°; vol in steam; was prepd by treating 6-nitro-4-hydroxylamino-1,3-dimethylbenzene with $FeCl_3$ in alc (Ref 1, p 844 & Ref 2)

4-Nitroso-5-nitro-1,3-dimethylbenzene, grn-yel ndls (from alc), mp 134°(dec); was prepd by reacting an aq Na_2SO_5 soln with 5-nitro-4-amino-1,3-dimethylbenzene at 30° (Ref 1, p844 & Ref 3)

6-Nitroso-2-nitro-1,4-dimethylbenzene, crysts (from alc), mp 134–35°, giving a green melt; was prepd by reacting 6-nitro-2-hydroxylamino-1,4-dimethylbenzene with $FeCl_3$ in aq alc in the cold (Ref 1, p863 & Ref 2)

Their expl props are not reported

Refs: 1) Beil **5**, {843, 844 & 863} 2) K. Brand & J. Mahr, JPrChem [2] **142**, 153, 165 (1935) & CA **29**, 2931 (1935) 3) R. Kuhn et al, Ber **70**, 1293, 1296, 1314 (1935) & CA **31**, 6211, 6239 (1937)

Azidonitrodimethylbenzene,
$O_2N.C_6H_2(CH_3)_2.N_3$; mw 196.16, N 14.28%

Two isomers are known:

4-Azido-5-nitro-1,3-dimethylbenzene, wh ndls (from alc), mp 66°; sol in common org solvs; was obtd by reaction of NH_3 on the diazoperbromide of 5-nitro-4-amino-1,3-dimethylbenzene; by heating under vacuum at 130°, the 4,5-Dinitroso-1,3-dimethylbenzene deriv is formed (Ref 1, p381 & Ref 3)

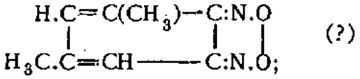
4-Azido-6-nitro-1,3-dimethylbenzene, ndls (from dil alc), mp 75°; readily sol in alc; vol in steam; was obtd from the 6-nitro-4-amino deriv (Ref 1, p382 & Ref 2)

Their expl props are not reported

Refs: 1) Beil **5**, 381, 382 2) E. Grandmougin & (?) Michel, Ber **25**, 3342 (1892) 3) T. Zincke & (?) Schwarz, Ann **307**, 47 (1899) 4) No later refs found

Dinitrosodimethylbenzene, $C_8H_8N_2O_2$; mw 164.16, N 17.07%. Two isomers are known:

4,5-Dinitroso-1,3-dimethylbenzene,



$$\begin{array}{l} H.C{=}C(CH_3){-}C{:}N.O \\ \quad | \qquad\qquad\quad | \quad\; | \qquad (?) \\ H_3C.C{=}CH{-\!-\!-}C{:}N.O; \end{array}$$

pltlts (from benzin), mp 108–09°; vol in steam; readily sol in common org solvs; less sol in benzin & water; was obtd by heating 4-azido-5-nitro-1,3-dimethylbenzene in a vacuum at 130° (Ref 1, p657 & Ref 3)

2,5-Dinitroso-1,4-dimethylbenzene,
$\overline{O.N:C_6H_2(CH_3)_2:N.O}$ (?); mp 250°; diffc sol; prepd by heating a soln of 1,4-dimethylbenzoquinone-dioxime in NaOH with K ferric cyanide soln (Ref 1, p659 & Ref 2)

No expl props are reported

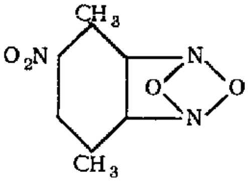
Refs: 1) Beil **7**, 657, 659 2) Pflug, Ann **255**, 176 (1889) 3) T. Zincke & (?) Schwarz, Ann **307**, 47 (1899)

4,5-Dinitroso-2-nitro-1,3-dimethylbenzene or **5,6-Dinitroso-4-nitro-1,3-dimethylbenzene,**

$\overline{O.N:C_6H(NO_2)(CH_3)_2:N.O}$; mw 209.16, N 20.09%; yel ndls (from abs alc), mp 116°; mod sol in benz & glac acet ac; sl sol in alc; was obtd by heating 2,5- or 5,6-dinitro-4-azido-1,3-dimethylbenzene at 110°, or by nitrating 4,5-dinitroso-1,3-dimethylbenzene with nitric acid (d 1.51) in the cold (Refs 1 & 2). Its expl props are not reported

Refs: 1) Beil **7**, 657 2) (?) Drost, Ann **313**, 313(1900)

5,6-Dinitroso-2-nitro-1,4-dimethylbenzene or **5-Nitro-4,7-dimethylbenzfurazan-1,3-oxyd** or **5-Nitro-4,7-dimethylbenzfuroxan,**



yel crysts (from benzin), mp 81°; mod sol in alc & benz; decompd by alkalies; was prepd by heating 3,5-dinitro-2-azido-1,4-dimethylbenzene at 105–30° (Refs 1 & 2). Its expl props are not reported

Refs: 1) Beil **27**, (623–24) 2) K. Fries & K. Noll, Ann **389**, 374(1912) & CA **6**, 2416(1912)

Dinitrodimethylbenzene,$(O_2N)_2C_6H_2(CH_3)_2$; mw 196.16, N 14.28%. Fourteen isomers are known:

3,4-Dinitro-1,2-dimethylbenzene, ndls (from alc), mp 82°, bp–explodes on heating in a tube at 413°; sol in org solvs; obtd by nitrating o-xylene (Ref 1, p369; Ref 2, p181; Ref 3, p287 & Ref 4, p822)

3,5-Dinitro-1,2-dimethylbenzene, crysts (from alc), yel ndls (from alc, or chlf + petr eth), mp 75–76°, sinters at 71°, bp–explodes on heating in a tube at 438°; sol in benz, acet, chlf & eth acet; prepd by nitrating 4-nitro deriv with HNO_3/H_2SO_4 mixed acid (Ref 1, p369; Ref 2, p181 & Ref 4, p823)

3,6-Dinitro-1,2-dimethylbenzene, ndls (from alc), mp 56–60° & 89–90°; obtd with other products on nitrating o-xylene or 3-nitro-o-xylene with concd nitric acid (Ref 1, p369 & Ref 2, p181)

4,5-Dinitro-1,2-dimethylbenzene, ndls (from benz or acet acid), mp 116–18°; sl sol in hot w, cold alc & petr eth; mod sol in other org solvs; prepd by nitrating 4-nitro deriv with mixed acid on a water bath for 10 mins and by other methods (Ref 1, p369; Ref 2, p181 & Ref 4, p823)

a,a-Dinitro-1,2-dimethylbenzene, $(O_2N)_2HC.C_6H_4.CH_3$; yel prisms (from petr eth), mp 42°, bp–dec at 115°; obtd with other products by action of NO_2 & $CuSO_4$ on o-xylene at 15–20°; forms a Potassium salt, yel ndls (from aq $KHCO_3$ soln)(Ref 4, p823)

2,4-Dinitro-1,3-dimethylbenzene, yel crysts (from alc), mp 84–86°; prepd by reacting 4-nitro-2-amino-o-xylol with K persulfate & H_2SO_4 in the cold, and HNO_3 (d 1.48) at 40–45° (Ref 1, p379; Ref 3, p295 & Ref 4, p844)

2,5-Dinitro-1,3-dimethylbenzene, col crysts, becoming yel in the light (from alc), mp 101°; prepd by diazotizing 2,5-dinitro-2-amino-1,3-dimethylbenzene and heating the diazonium salt in alc (Ref 1, p380 & Ref 3, p295)

4,5-Dinitro-1,3-dimethylbenzene, yel crysts (from alc), mp 132°; prepd by warming 4-nitroso-5-nitro-1,3-dimethylbenzene with aq H_2O_2 & HNO_3 in glac acet acid on a w bath (Ref 1, p380; Ref 3, p295 & Ref 4, p844)

4,6-Dinitro-1,3-dimethylbenzene, col prisms (from alc), mp 92–94°, bp–explodes; prepd by nitrating m-xylene with concd nitric acid at RT (Ref 1, p380; Ref 2, p184; Ref 3, p295 & Ref 4, p844)

a,a-Dinitro-1,3-dimethylbenzene, $(O_2N)_2HC.C_6H_4.CH_3$; prisms (from alc or benz), mp 53–54.5°; obtd by reacting an excess of NO_2 on m-xylene in the presence of $CuSO_4$ at 15–20°; forms a Potassium salt, $KC_8H_7N_2O_4$, crysts (from w), mp 125° (dec) (Ref 4, p844)

2,3-Dinitro-1,4-dimethylbenzene, monoclinic prisms (from alc or benz), mp 90–93°; obtd with other products on nitrating p-xylene with concd nitric acid or with mixed acid (Ref 1, p387; Ref 2, p188; Ref 3, p302 & Ref 4, p863)

2,5-Dinitro-1,4-dimethylbenzene, yel ndls (from alc), mp 142° & 147–48°; diffc sol in cold alc & cold eth; more sol in warm solvs; prepd by heating the diazotized product of 5,6-dinitro-2-amino-p-xylene with alc (Ref 1, p388 & Ref 2, p188)

2,6-Dinitro-1,4-dimethylbenzene, ndls (from alc), mp 123–24°; prepd by heating diazotized 3,5-dinitro-2-amino-p-xylene with alc (Ref 1, p388; Ref 2, p188; Ref 3, p302 & Ref 4, p863) *a,a-Dinitro-1,4-dimethylbenzene,* $(O_2N)_2HC.C_6H_4.CH_3$; wh pltlts (from petr eth), mp 77–79°, bp–dec at 130°; sl sol in cold petr eth; prepd from p-toluylaldehyde or p-toluylaldoxime by nitrating with HNO_3 (d 1.5) in acetic acid (Ref 1, p388 & Ref 4, p864)

Refs: 1) Beil **5**, 369, 379, 380, 387, 388 2) Beil **5**, (181, 184, 188) 3) Beil **5**, [287, 295, 302] 4) Beil **5**, {822, 823, 844, 863, 864}

Azidodinitrodimethylbenzene, $N_3.C_6H(NO_2)_2(CH_3)_2$; mw 237.18, N 29.53%. Two isomers are known:

4-Azido-2,5 (or 5,6)-dinitro-1,3-dimethylbenzene, wh pltlts (from alc), becoming yel in the light, mp 82°; mod sol in alc; formed by nitrating 4-azido-5-nitro-1,3-dimethylbenzene with nitric acid (d 1.51); on heating to 110°, it forms 5,6-dinitroso-2(or 4)-nitro-1,3-dimethylbenzene (qv). Its expl props are not reported (Refs 1 & 3)

2-Azido-3,5-dinitro-1,4-dimethylbenzene, pltlts (from dil alc), mp 71–73°; mod sol in benz & acet ac; sl sol in alc; v sl sol in benzine; prepd from 3,5-dinitro-p-xylol-diazonium-2-perbromide thru reaction with aq NH_3; on heating to 105–30°, it forms 5,6-Dinitroso-2-nitro-1,4-dimethylbenzene(qv). Its expl props are not reported (Refs 2 & 4)

Refs: 1) Beil **5**, 382 2) Beil **5**, (188) 3) Dorst, Ann **313**, 312(1900) 4) K. Fries & K. Noll, Ann **389**, 373(1912) & CA **6**, 2416(1912)

Trinitrodimethylbenzene or **Trinitroxylene** (abbr as **TNX**), $(O_2N)_3C_6H(CH_3)_2$; mw 241.16, N 17.43%. Six isomers are known:

3,4,5-Trinitro-1,2-dimethylbenzene, col ndls (from alc), mp 115°; mod sol in chlf, benz, eth acet, acet & alc; was obtd with other isomers on nitrating 1,1-dimethylcyclohexane with a mixt of 1 vol HNO_3 & 2 vols H_2SO_4 on a w bath (Ref 1). Marqueyrol & Loriette (Ref 5) detd its press when fired in a 1.125cc bomb with 1 g priming chge to be 1762kg/cm^2 at d 0.2 & 3202 at d 0.3

3,4,6-Trinitro-1,2-dimethylbenzene, yel ndls (from alc), mp 72°; becomes more yel in the light; sol in common org solvs; prepn – see above (Ref 1). Its pressures, as above, were 1661 & 3080, respectively (Ref 5)

2,4,5-Trinitro-1,3-dimethylbenzene, wine-yel crysts (from abs alc), mp 90°, d 1.553 at 14°; v sl sol in alc at 20°; was obtd by nitrating 5-nitro-1,3-dimethylbenzene with mixed acid (Ref 2)

2,4,6-Trinitro-1,3-dimethylbenzene or **2,4,6-Trinitro-m-xylene,** ndls (from benz + hexane or dioxane), mp 181–84°; bp–explodes on heating in a tube at 410–12°, d 1.69; was prepd by electrolysis of 2,4,6-trinitrotoluene with Na acetate in acet ac using a Pt electrode at 35–45° (Ref 3)

4,5,6-Trinitro-1,3-dimethylbenzene, prisms (from alc), d 1.494 at 19°; v sl sol in alc; can be prepd by nitrating 5-nitro-1,3-dimethylbenzene, 4,5-dinitro-1,3-dimethylbenzene or 4-iodo-5-nitro-1,3-dimethylbenzene with mixed acid (Ref 2)

2,3,5-Trinitro-1,3-dimethylbenzene, col ndls (from alc), becomes yel in the light; mp 137–40°, explodes on heating in a tube at 410°; can be prepd by the step-wise nitration of p-xylene (Ref 4). Marqueyrol & Loriette (Ref 5) detd its press when fired in a 1.125cc bomb with 1 g priming chge to be 1670kg/cm^2 & 3080 at d 0.3

Refs: 1) Beil **5**, 370, (181), [287]& {823} 2) Beil **5**, 381 3) Beil **5**, 381, (185), [295] & {845} 4) Beil **5**, 389, (188), [303] & {864} 5) M. Marqueyrol & P. Loriette, BullFr [4] **27**, 424(1920) & CA **14**, 2709(1920)

TRINITROXYLENE, Commercial (TNX) or Xylyl, known in Russia as **Kcilil** or **Ksilil**, consists chiefly of 2,4,6-Trinitro-meta-xylene (m-TNX) with some ortho and para isomers (o-TNX and p-TNX). TNX is a grayish-yellow substance prepd by nitration of commercial xylene with mixed nitric-sulfuric acid. Accdg to Pepin Lehalleur (Ref 1), Solonina developed during WWI one-stage nitration of xylene with a yield of 75%. TNX is insol in water, is slightly more sensitive to initiation than TNT and about as sensitive to impact and bullet tests as the latter. A small amount of TNX added to TNT is claimed to make the latter expl more sensitive to initiation. This is due, according to Shilling (Ref 2, p99), to the exceptionally small crystalline structure of cast mixts of TNT & TNX

TNX has been used in composite expls as a partial substitute for TNT. For example, Ya.I. Leitman proposed during WWII a mixt of TNT 95 & TNX 5%, under the name of **L-Splav** (L-Fuse or L-Mixture), for cast-loading anti-tank mines and in demolition blocks. The L-Splav has a d of loading 1.54–1.56g/cc and mp 74°. It is more sensitive to initiation than TNT, requiring a smaller booster chge (such as Tetryl) and can be initiated by a very strong detonator alone. TNX has also been used in industrial expls

Refs: 1) Pepin Lehalleur (1935), 188 2) Shilling (1946), 99 & 101 3) Blinov, Vol 1 (1948), 19 & 16 4) Fedoroff et al, PATR **2145**(1955), p Russ 10 5) Gorst (1957), 98

α,α,α',α'-Tetranitro-1,3-dimethylbenzene or **α,α,α',α'-Tetranitro-m-xylene,** $(O_2N)_2HC.C_6H_4CH(NO_2)_2$; mw 286.16, N 19.58%; compd listed in CA 7th Collective Index (1962-66), p770F but no info is found in abstract (Ref 2)

Refs: 1) Beil. – not found 2) V.I. Slovetskii et al, KhimGeterotsiklSoedin, AkadNaukLatv SSR **1965** (6), 835–45 (Russ) & CA **64**, 15705 (1966)

Dimethylbenzidine and Derivatives

Dimethylbenzidine or Tolidine, $C_{14}H_{16}N_2$; mw 212.28, N 13.20%. Eleven isomers exist:

N,N'-Dimethylbenzidine, $[-C_6H_4NH.CH_3]_2$, ndls (from alc), mp 74–76° & 81–82°; sl sol in alc, eth, chlf & benz; v sl sol in cold petr eth; forms salts (Ref 1, p220 & Ref 3, p97)

N,N-Dimethylbenzidine, $H_2N.C_6H_4-C_6H_4.N(CH_3)_2$, pltlts (from alc or w), mp 146°; becomes green & brn on warming in the light; sol in dil HCl & H_2SO_4; forms salts (Ref 3, p97)

4,4'-Diamino-2,2'-dimethyldiphenyl; 2,2'-Dimethylbenzidine or *m-Tolidine,* $H_2N(H_3C)C_6H_3-C_6H_3(CH_3)NH_2$, prisms (from hot w), mp 106–109°; v sol in alc & eth; forms salts (Ref 1, p255 & Ref 2, p79)

4,4'-Diamino-2,6-dimethyldiphenyl or *2,6-Dimethylbenzidine,* $H_2N.C_6H_4-C_6H_2(CH_3)_2.NH_2$, ndls (from benz + petr eth), mp 124°; sol in alc, eth & benz; sl sol in petr eth & hot w; forms salts (Ref 3, p115)

5,5 -Diamino-2,2'-dimethyldiphenyl or *5,5'-Diamino-2,2'-ditolyl,* $H_2N(H_3C)C_6H_3-C_6H_3(CH_3).NH_2$, crysts, mp 96–98°, bp 225–28° at 13mm press; v sl sol in chlf; sol in eth, benz, hot alc & hot petr eth; forms salts (Ref 3, p115)

6,6'-Diamino-2,2'-dimethyldiphenyl or *6,6'-Diamino-2,2'-ditolyl,*

Inactive or dl-form: ndls (from alc), mp 136°

Right rotation or d-form: crysts (from alc), mp 156°

Left rotation or l-form: crysts (from alc), mp 156°

All form salts, such as Diacetyl derivs (Ref 13, pp115–16)

4,4 -Diamino-2,3'-dimethyldiphenyl or *2,3'-Dimethylbenzidine,* amorph mass, bp 243–46° (Ref 1, p256 & Ref 3, p116)

2,4'-Diamino-3,3'-dimethyldiphenyl or 3,3 -Dimethyldiphenylene, ppt; forms a Hydrochloride salt, $C_{14}H_{16}N_2+2HCl$, ndls, v sol in w (Ref 1, p256)

4,4'-Diamino-3,3'-dimethyldiphenyl; 3,3'-Dimethylbenzidine or *o-Tolidine,* pltlts, mp 126–29°; sol in alc & eth; sl sol in w; forms numerous cryst salts with inorg & org compds (Ref 1, p256, Ref 2, p79 & Ref 3, p116)

6,6'-Diamino-3,3'-dimethyldiphenyl, crysts, mp 80–83° (cited in Ref 5)

2,2'-Diamino-2,2'-dimethyldiphenyl, long ndls (from alc), mp 120°; sol in common org solvs, sl sol in w (Ref 1, p261)

Other props & methods of prepn are given in Refs

Refs: 1) Beil **13**, 220, 255, 256 & 261 2) Beil **13**, (79) 3) Beil **13**, [97, 115, 116] 4) Beil **20**, 449 (line 4) & **27**, (676)

Dinitrodimethylbenzidines, $C_{14}H_{14}N_4O_4$, are found in Beil **13**, 260, 261, (80, 81) & [120]

Trinitrodimethylbenzidine, $C_{14}H_{13}N_5O_6$, not found in the literature thru 1966

3,5,3',5'-Tetranitro-N,N'-dimethylbenzidine, $[-C_6H_2(NO_2)_2.NH.CH_3]_2$; mw 392.28, N 21.43%; red ndls (from phenol), mp–dec at 282°; sol in hot phenol; insol in glac acet ac & hot alc; was prepd by heating 3,5,3',5'-tetranitro-4,4'-dimethoxydiphenyl or the tetranitrodiethoxy deriv with methylamine in alc in a tube at 100°; can be nitrated to the hexanitro deriv (Refs 1 & 2)

Refs: 1) Beil **13**, 237 & [109] 2) P. van Romburgh, Rec **41**, 41 (1922)

Hexanitro-N,N'-Dimethylbenzidine, $C_{14}H_{10}N_8O_{12}$; mw 482.28, N 23.24%.

N,N',3,5,3',5'-Hexanitro-N,N'-diaminodimethyldiphenyl or N,N',3,5,3',5'-Hexanitro-N,N'-dimethylbenzidine, $[-C_6H_2(NO_2)_2.N(NO_2).CH_3]_2$; lt yel ndls (from concd nitric acid & pptd with w), mp–explodes above 220°; sol in phenol & aniline; insol in alc; was prepd by nitrating the tetranitro deriv with concd nitric acid (Refs 1 & 2)

Refs: 1) Beil **13**, 237 & [109] 2) P. van Romburgh, Rec 41, 41 (1922)

N,N',4,4',6,6'-Hexanitro-N,N'-dimethylbenzidine; N,N',4,4',6,6'-Hexanitro-N,N'-dimethyl-3,3'-biphenyldiamine (CA nomenclature); or 4,4',6,6'-Tetranitro-3,3'-bis(methylnitramino)biphenyl (called by van Alphen), $[-C_6H_2(NO_2)_2N(NO_2).CH_3]_2$; crysts, mp 210°; sol only in acet; was prepd by nitrating the corresponding tetranitro-dimethylamino deriv with HNO_3–H_2SO_4 on a w bath (Ref 2). Its expl props are not reported

Refs: 1) Beil – not found 2) J. van Alphen, Rec **51**, 361 (1932) & CA **26**, 2447 (1932)

Dimethylbenzoic Acid and Derivatives

Dimethylbenzoic, Xylic or Methyltoluic Acid, $(H_3C)_2C_6H_3.COOH$, mw 150.17, O 21.31%. Six isomers exist:

2,3-Dimethylbenzoic Acid, crysts (from alc or cyclohexane), mp 144–46°, vol in steam; v sl sol in hot w; forms metallic salts (Ref 1)

2,4-Dimethylbenzoic Acid or m-Xylic Acid, crysts (from cyclohexane by sublimation), mp 125–26°, bp 267° at 727mm press; sol in toluene, xylene, benzene, chlf & acet; almost insol in cold w (Ref 2)

2,5-Dimethylbenzoic Acid, crysts (from cyclohexane), mp 132–34°, bp 268°, vol in steam; sol in alc; v sl sol in cold w (Ref 3)

2,6-Dimethylbenzoic Acid, crysts (from w or cyclohexane), mp 115.8–17°; sol in eth; v sl sol in cold petr eth (Ref 4)

3,4-Dimethylbenzoic Acid, crysts (from cyclohexane or xylene), mp 165–68°; mod sol in alc, v sl sol in hot w; almost insol in cold w; forms salts (Ref 5)

3,5-Dinitrobenzoic Acid, crysts (from alc or cyclohexane), mp 168–72°, vol in steam; sol in cold alc; v sl sol in hot w; forms metallic salts (Ref 6)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **9**, 531 ,(209) & {2434} 2) Beil **9**, 531 (209), [350] & {2436} 3) Beil **9**, 534, (210) & {2439} 4) Beil **9**, 531, [350] & {2435} 5) Beil **9**, 535, (210), [353] & {2441} 6) Beil **9**, 536, (210), [354] & {2444}

2-Azido-3,5-dimethylbenzoic Acid, $(H_3C)_2C_6H_2(N_3).COOH$; mw 191.19, N 21.98%; ndls (from petr eth), mp 156° (dec); sol in alc & benz; sl sol in w & cold petr eth; was prepd by oxidation of 2-azido-3,5-dimethylbenzaldehyde with $KMnO_4$ at 100°, or by diazotizing 2-amino-3,5-dimethylbenzoic acid and treating the diazoniumperbromide salt with concd NH_3 (Refs 1 & 2)

Refs: 1) Beil **9**, 538 2) E. Bamberger & Demuth, Ber **34**, 1320 (1901) 3) No later refs found thru 1966

X,X-Dinitroso-2,4-dimethylbenzoic Acid (?), $(H_3C)_2C_6H(NO)_2.COOH$; mw 208.17, N 13.46%; crysts, mp 166°; prepd by reacting x,x-dinitroso-2,4-dimethylphenylglyoxylic acid with $K_2Cr_2O_7$ & H_2SO_4 (Refs 1 & 2). Its expl props are not reported

Refs: 1) Beil **9**, 534 2) Claus, JPraktChem [2] **41**, 490 (1890)

Mononitrodimethylbenzoic Acid, $(H_3C)_2C_6H_2(NO_2).COOH$; mw 195.17, N 7.18%. Several isomers are known:

5-Nitro-2,4-dimethylbenzoic Acid, crysts (from alc or benzine + benzene), mp 195–98°; sol

in alc, eth, chlf & CS_2; sl sol in hot w; v sl in cold w; forms cryst metallic salts (Ref 1)
6-Nitro-2,4-dimethylbenzoic Acid, yel ndls, mp 180°; mod sol in alc; sl sol in w (Ref 2)
4-Nitro-2,5-dimethylbenzoic Acid, crysts (from benzene), mp 165.5–166.5° (Ref 3)

2-Nitro-3,5-dimethylbenzoic Acid, ndls (from hot w), mp 210–11°; sl sol in cold w (Ref 4)
4-Nitro-3,5-dimethylbenzoic Acid, lt-yel crysts (from dil alc), mp 225.5–227°, crysts (from w) + 1 mol water, mp 174–76° (Ref 5)

Other props & methods of prepn are given in Refs
Refs: 1) Beil **9**, 534, [353] & {2438} 2) Beil **9**, (210) 3) Beil **9**, {2440} 4) Beil **9**, 537 5) Beil **9**, {2446}

x-Nitroso-x-nitro-2,4-dimethylbenzoic Acid (?) $(H_3C)_2C_6H(NO)(NO_2).COOH$, mw 224.17, N 12.50%; ndls (from hot w), mp 256°; sol in hot w; sl sol in acet ac; almost insol in CS_2 & benz; was prepd by heating 2,4-dimethylphenylglyoxylic acid with an excess of concd HNO_3(d 1.5) (Refs 1 & 2). Its expl props are are not reported
Refs: 1) Beil **9**, 534 2) Claus, JPraktChem [2], 41, 489, 491 (1890)

Dinitrodimethylbenzoic Acid,
$(H_3C)_2C_6H(NO_2)_2.COOH$; mw 240.17, N 11.67%. Only three isomers are known:
3,5-Dinitro-2,4-dimethylbenzoic Acid, crysts (acetic acid), mp 197-203°; mod sol in alc, eth & chlf; sl sol in hot w; can be prepd by nitrating 2,4-dimethylbenzoic acid with mixed acid, or by oxidizing 3,5-dinitro-2,4-dimethylacetophenone with $KMnO_4$ (Refs 1 & 4). Its expl props are not reported
2,4-Dinitro-3,5-dimethylbenzoic Acid, ndls (from w or xylene), mp 210.5–211°; mod sol in alc & hot xylene; v sl sol in w & petr eth; was obtd with the 2,6-dinitro deriv when 3,5-dimethylbenzoic acid was treated with nitric acid (d 1.525) at 40–50° (Refs 2 & 3). Its expl props are not reported
2,6-Dinitro-3,5-dimethylbenzoic Acid, col prisms, mp 215.5–216°; mod sol in alc & eth; sl sol in hot xylene; v sl sol in chlf & w; prepn (See above) (Refs 2 & 3). Its expl props are not reported
Refs: 1) Beil **9**, 534, (210) & {2439} 2) Beil **9**, 538 3) E. Bamberger & Demuth, Ber **34**, 29, 32 (1901) 4) R.C. Fuson et al, JACS **61**, 1937, 1939 (1939)
NOTE: No higher nitrated derivs of Dimethylbenzoic Acid were found in the literature thru 1966

Dimethylbenzophenone and Derivatives
Dimethylbenzophenone, $C_{15}H_{14}O$, mw 210.26. Several isomers are known, but only one has been nitrated to form a tetranitro deriv:
2,4-Dimethylbenzophenone, $(H_3C)_2C_6H_3.CO.C_6H_5$, oil, bp 362° & 178–80° at 12mm press; was prepd by treating m-xylene with benzoyl chloride & $AlCl_3$ in CS_2 and by other methods (Ref)
Ref: Beil **7**, 449, (239), [386], {2176}

Azido, $C_{15}H_{13}N_3O$, and *Diazido*, $C_{15}H_{12}N_6O$, derivs, not found in the literature thru 1966

Mononitro, $C_{15}H_{13}NO_3$, and **Dinitro**, $C_{15}H_{12}N_2O_5$, derivs. Several are described in the literature, but none is expl

Trinitrodimethylbenzophenone, $C_{15}H_{11}N_3O_7$, mw 345.26, N 12.17%. Two isomers are known:
3,5,3'-Trinitro-2,4-dimethylbenzophenone, $(H_3C)_2(O_2N)_2C_6H.CO.C_6H_4.NO_2$; pltlts (from alc, glac acet ac or benz), mp 139–40°; mod sol in glac acet ac, benz & acetone; sl sol in hot alc; v sl sol in eth, petr eth & CS_2; insol in w; can be prepd by nitrating 2,4-dimethylbenzophenone; 3'-nitro-2,4-dimethyl- or 3,5-dinitro-2,4-dimethylbenzophenone with mixed acid (Refs 1 & 3)
3',X,X-Trinitro-2,4-dimethylbenzophenone, $C_{15}H_{11}(NO_2)_3$; yel ndls (from alc), mp 138–39°; sol in benz; sl sol in acet; v sl sol in eth, chlf, hot alc; insol in cold alc; prepd by nitrating 3'-nitro-2,4-dimethylbenzophenone with mixed acid (Ref 2 & 3)

Their expl props are not reported
Refs: 1) Beil **7**, [387] 2) Beil **7**, 450 & [387] 3) L. Chardonnens, Helv **12**, 652, 651 (1929) & CA **23**, 4687 (1929)

3,5,3',5'-Tetranitro-2,4-dimethylbenzophenone, $(H_3C)_2(O_2N)_2C_6H.CO.C_6H_3(NO_2)_2$; mw

390.26, N 14.36%; yel prisms (from glac acet ac), mp 187–88°; sl sol in eth, alc & chlf; insol in w; was prepd by nitrating 3',5'-dinitro-2,4-dimethylbenzophenone with mixed acid at 0° then at higher temp (Refs 1 & 2). Its expl props are not reported

Refs: 1) Beil **7**, [387] 2) L. Chardonnens, Helv **12**, 662(1929) & CA **23**, 4687(1929)

NOTE: No higher nitrated derivs of Dimethylbenzophenone were found in the literature thru 1966

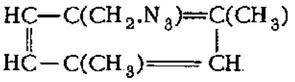
Dimethylbenzylamine and Derivatives

3,5-Dimethylbenzylamine,
$(H_3C)_2C_6H_3.CH_2.NH_2$; mw 135.20, N 10.36%, liq, bp 217–18°, d 0.9500 at 20°, n_D 1.53046 at 20.5°; forms a Picrate, $C_9H_{13}N+C_6H_3N_3O_7$, yel crysts which dec at 225° (Ref 1). This is the only isomer which has a dinitro deriv:

2,4- or 4,6-Dinitro-3,5-dimethylbenzylamine,
$(H_3C)_2C_6H(NO_2)_2.CH_2.NH_2$; mw 225.20, N 18.66%; liq; obtd by heating the acetyl deriv, $(H_3C)_2C_6H(NO_2)_2.CH.NH.CO.CH_3$, with HCl (d 1.1) in a tube at 150°; forms a *Hydrochloride* salt, $C_9H_{11}N_3O_4+HCl$, pltlts, dec at 245°; and a *Picrate*, $C_9H_{11}N_3O_4+C_6H_3N_3O_7$, yel prisms (from w), dec at 220° (Refs 2 & 3)

Refs: 1) Beil **12**, 1163 2) Beil **12**, 1164 3) Landau, Ber **25**, 3015(1892)

2,4-Dimethylbenzylazide; 1'-Azido-pseudocumene or 1'-Azido-1,2,4-trimethylbenzene,



$$\begin{array}{l} HC{-}C(CH_2.N_3){=}C(CH_3) \\ \| \qquad\qquad\qquad\quad | \\ HC{-}C(CH_3){=\!=}CH \end{array}$$

mw 161.20, N 26.07%, liquid, bp 114° at 15mm press; expl at higher temps. Insol in w; easily sol in eth, alc, benz or ligroin. Was prepd by Mayer by treating the nitrosohydrazine, $C_9H_{13}N_3O$, with 10% sulfuric acid (Refs 1 & 2)

Refs: 1) Beil **5**, 405 & (199) 2) F. Mayer, "Dissertation", Heidelberg (1902), p41
3) No later refs found in the literature thru 1966

α,α-Dimethylbenzyl Hydroperoxide (DMBH) or Cumene Hydroperoxide. Its properties are described in Vol 3, pp C574-R & C575-L, but method of prepn, such as described by Drs E. Hedaya & S. Winstein in JACS **89**, 1661(1967) was not included because Vol 3 of Encycl was published one year earlier (1966). The same authors stated in C&EN, Oct 9, 1967, p73 that in their description of prepn from dimethylbenzylcarbinol, 90% H_2O_2 and concd H_2SO_4 was a "subtle and unfortunate error". Dr W. Adam of the Univ of Puerto Rico, who tried to prep DMBH by the "erroneous" method stated that "a damaging and violent explosion occurred when the concentrated sulfuric acid was added to a mixture of alcohol and 90% hydrogen peroxide. (Here the word "alcohol" means dimethylbenzylcarbinol)

Drs H & W stated that the text in their JACS paper should read: "Dimethylbenzylcarbinol (6 grams) was combined with 30ml of 90% hydrogen peroxide acidified with 12 drops of concentrated sulfuric acid"

A still safer procedure is to add the acidified 90% hydrogen peroxide to the alcohol. Even so, Drs H & W recommend that this reaction and similar prepns involving 90% hydrogen peroxide be carried out on a small scale (no more than 5g 90% H_2O_2)

Dimethylbiguanides

1,1-Dimethylbiguanide or N,N-Dimethyl N-guanylguanidine,
$(H_3C)_2N.C(:NH).NH.C(NH_2):NH$, mw 129.17, N 54.22%; known as salts, such as, *Hydrochloride*, $C_4H_{11}N_5+HCl$, prisms (from w), mp 235°; and *Picrate*, mp 219° (Refs 1 & 3)

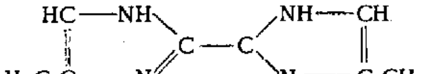
1,2-Dimethylbiguanide or N,N'-Dimethyl-N-guanylguanidine,
$H_3C.N:C(NH.CH_3).NH.C(NH_2):NH$; known as the *Hydrobromide* salt, $C_4H_{11}N_5+HBr$, crysts (from w), dec at 240–45° (Ref 2, p579)

1,5-Dimethylbiguanide,
$H_3C.NH.C(:NH).NH.C(:NH).NH.CH_3$; *Sulfate* salt, $C_4H_{11}N_5+H_2SO_4$, dec at 200° (Ref 2, p572)

Refs: 1) Beil. 4, [575] & {146} 2) Beil. 4, [572, 579] 3) J.H. MacGregor, USP 2448448 (1948) & CA **43**, 868(1949)

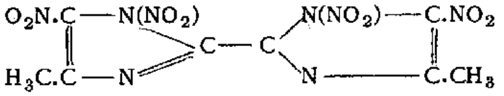
Dimethyl-biimidazole and Derivatives

4,4'-Dimethyl-2,2'-biimidazole,



mw162.19, N 34.55%; isolated as the *Dipicrate,* $C_8H_{10}N_4+2C_6H_3N_3O_7$, ndls, dec 230°(Ref 4)

4,4'-Dimethyl-1,1'-5,5'-tetranitro-2,2'-biimidazole,



mw 342.20, N 32.75%; lt yel crysts, mp – dec 239–40°; was obtd when the anhyd Na salt of 4-methyl-1,5,1',5'-tetranitro-2,2-biimidazole & $MeSO_4$ were warmed on a w bath. The Dimethyl-tetranitro deriv & NaOH in acetone gives a brick-red salt, $C_8H_6N_6O_6Na_2$, which in hot MeOH-Me ONa yields 1,1'-dimethoxy-4,4'-dimethyl-5,5'-dinitro-2,2'-biimidazole, dec at 242°(Ref 3)

Refs: 1) Beil, not found (Parent) 2) Beil, not found (Tetranitro) 3) K. Lehmstedt, Ann **507**, 213(1933) & CA **28**, 765(1934) 4) R. Kuhn & W. Blau, Ann **605**, 32(1957) & CA **51**, 16437(1957)

Dimethyl-biphenyl; Dimethyl-diphenyl or **Ditolyl.** See Bitolyl in Vol 2 of Encycl, p B163

N,N-Dimethyl-p-biphenylamine. See N,N-Dimethylaminobiphenyl

N,N'-Dimethyl-N,N'-bis [phenyl]-urea; N,N'-Dimethyl-carbanilide or **N,N'-Dimethyl-diphenyl-urea.** See Centralite 2 in Vol 2, pp C137ff

N,N'-Dimethyl-N,N'-bis[2,4-dinitrophenyl]-urea. See Tetranitrocentralite, under Centralite 2, in Vol 2, p C139-L and Beil. **12**, [411]

N,N'-Dimethyl-N,N'-bis[x,x,x-trinitrophenyl]-urea. See Hexanitrocentralite, under Centralite 2, in Vol 2, p C139-L and Beil. **12**, [411]

Dimethylbiuret and Derivatives

1,5-Dimethylbiuret or *N,N'-Dimethylallophanamide,* $HN(CO.NH.CH_3)_2$, mw 131.14, N 32.05%; prism (from alc), mp 162–63°; sol in benz & eth acetate; sl sol in alc & benz; v sl sol in w & chlf; almost insol in eth & petr eth (Ref 1)

1,5-Dinitroso-1,5-dimethylbiuret, $HN[CO.N(NO).CH_3]_2$; mw 189.14, N 37.03%; yel prisms (from eth), mp – dec at 94°; sol in w & eth; sl sol in ethyl acet; v sl sol in cold w & petr eth; was obtd when 1,5-dimethylbiuret reacted with N_2O_3 in an ice-cold aq soln. It decomp on heating in water at 50°, or on heating in alc (Refs 2 & 3)

Refs: 1) Beil. 4, [569] (Dimethylbiuret) 2) Beil. 4, [585] (Dinitrosodimethylbiuret) 3) H. Biltz & A. Jeltsch, Ber **56B**, 1921 (1923) & CA **18**, 530 (1924)

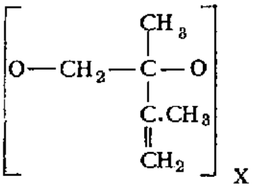
Dimethylborylmethyl Peroxide, $(H_3C)_2BOOCH_3$- mw 87.92; col liq of negligible vap pressure at –118°; compd is expl: two occurred during transfer in vacuo & one occurred during prepn; was prepd by reaction of O & trimethylborane in a flow system at RT & 10–15mm press and a 2–3 min contact time (Ref 2). It forms addn compds with NH_3 and pyridine

Refs: 1) Beil, not found 2) R.C. Petry & F.H. Verhock, JACS **78**, 6416–17(1956) & CA **51**, 4936(1957)

Dimethylbutadiene and Derivatives

2,3-Dimethyl-butadiene-1,3 or *Di-iso-propenyl,* $[H_2C:C(CH_3)]_2$; mw 82.14; liq, sp gr 0.727 at 20°/20, fr p –76°, bp 68.5° (Refs 1 & 4)

2,3-Dimethylbutadiene Peroxide, Polymeric,



mw (114.14)x, O 28.04%; yel, honeylike substance, exploding violently at ca 140°

Was prepd by oxidation of 2,3-dimethylbutadiene with molecular oxygen (Refs 3, 5 & 6)

It is not as violent an expl as Butadiene Peroxide described in Vol 2 of Encycl, p B366-R

Refs: 1) Beil **1**, 256, (118), [232] & {991} (Dimethylbutadiene) 2) Beil (Peroxide Polymer – not found) 3) K. Bodendorf, ArchPharm **271**, 1(1933) 4) P.N. Kogerman, SitzberNaturforschGesUnivTartu **41**, No 3–4, 62pp(1934) & CA **29**, 3297(1935) 5) W. Kern et al, Makromol Chem **3**, 223(1949) & **7**, 199(1951) 6) Tobolsky & Mesrobian (1954), 31 & 178

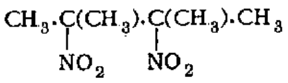
Dimethylbutane and Derivatives

2,2-Dimethylbutane or *Neohexane*, $H_3C.C(CH_3)_2.CH_2.CH_3$; mw 86.17, liq, sp gr 0.649 at 20°/20, fr p −99.7°, bp 49.7°, fl p −32°C. Can be prepd from refinery gases resulting from the cracking of petroleum. Used as a component of motor and aviation gasolines with very high octane ratings (Refs 1 & 9)

2,3-Dimethylbutane or *Diisopropyl*, $CH_3.CH(CH_3).CH(CH_3).CH_3$, liq, sp gr 0.662 at 20°/4, fr p −128.4°, bp 58.0°, flash p 29°C. Can be prepd by alkylation of ethylene with isobutane using Al chloride catalyst. Used as a component of high octane fuel (Refs 2 & 8)

2,2-Dimethyl-1,3-dinitrobutane, $(O_2N)H_2C.C(CH_3)_2.CH(NO_2).CH_3$; mw 176.17, N 15.90%, OB to CO_2 minus 127%; liq, bp 128–32° at 9mm press; was prepd when a mixt of nitro-tert-butyl alc, nitroethane & piperidine was kept at RT for 3 months; after neutralization & fractionation, the product was obtd (Refs 3 & 7)

2,3-Dimethyl-2,3-dinitrobutane or **sym-Tetramethyl-dinitroethane,**



$$CH_3.\underset{NO_2}{C}(CH_3).\underset{NO_2}{C}(CH_3).CH_3$$

mw, N & OB to CO_2 – same as above; lfts (from alc), sp gr 1.25; mp decomp at 210–14°; explodes above 360°; sublimes at 120–50° at 15mm pressure; insol in w; sl sol in ligroin, petroleum eth; sol in alc, toluene, acet ester, hot eth, acetone, chlf and benzene; can be prepd by shaking 2-brom-2-nitropropane with a large amt of pulverized silver in presence of ether (Ref 4) or by the action of NaOH on 2-nitropropane and 2-chloro-2-nitropropane (Ref 6)

It is an explosive comparable in sensitivity to impact to TNT, but difficult to detonate completely; heat of combstn 898.8 kcal/mole; sl hygroscopic but fairly stable to heat. Its power was not determined on account of incomplete detonation (Refs 4 & 6)

2,2-Dimethyl-3,3-dinitrobutane, $(H_3C)_3C.C(NO_2)_2.CH_3$, crysts, having a strong camphor odor, very volatile, mp 173–74°; easily sol in ether; obtd in a small quantity from pinacoline oxime & N_2O_4, in addn to pinacoline nitrimine (Ref 5)

NOTE: No higher nitrated derivs of Dimethylbutane were found in the literature thru 1966

Refs: 1) Beil **1**, 150, (54), [113] & {405} (2,2-Dimethylbutane) 2) Beil **1**, 151, (55), [113] & {410} (2,3-Dimethylbutane) 3) Beil **1**, {410} (1,3-Dinitro-2,2-dimethylbutane) 4) Beil **1**, 153, [114] & {415} (2,3-Dinitro-2,3-dimethylbutane) 5) Beil **1**, 151 (3,3-Dinitro-2,2-dimethylbutane) 6) Blatt, OSRD **2014**(1944) 7) A. Lambert & A. Lowe, JCSoc **1947**, 1519 & CA **42**, 4917 (1948) 8) CondChemDict(1961), 394-L (2,3-Dimethylbutane) 9) Ibid, 788-L (Neohexane)

Dimethylbutene and Derivatives

Dimethylbutene, C_6H_{12}; mw 84.16. Three isomers are known:

2,2-Dimethyl-butene-(3) or *tert-Butylethylene*, $H_2C:CH.C(CH_3)$, liq, bp 41.2°, d 0.6549 at 18°/4, n_D 1.37667 (Ref 1)

2,3-Dimethyl-butene-(1) or *1-Methyl-1-isopropylethylene*, $(H_3C)_2CH.C(CH_3):CH_2$, liq, fr p 157.3°, bp 55.67°, d 0.6731 at 25°/4, n_D 1.3874 at 25° (Ref 2)

2,3-Dimethyl-butene-(2) or *Tetramethylethylene*, $(H_3C)_2C:C(CH_3)_2$, liq, fr p 74.2°, bp 73.2°, d 0.7075 at 20°/20 (Ref 3)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **1**, 217, (91) & {814}

2) Beil **1**, 218, (91), [195] & {816}
3) Beil **1**, 218, (91), [195] & {817}

4-Nitroso-1-nitro-2,3-dimethyl-butene-(2), $ON.CH_2.C(CH_3):C(CH_3).CH_2.NO_2$; mw 158.16, N 17.71%; ndls (from eth acet), bp 104–05°; sl sol in cold benz; obtd with other products by reaction of N_2O_3 on 2,3-dimethyl-1,3-butadiene in ether in the cold (Refs 1 & 2). Its expl props are not reported

Refs: 1) Beil **1**, {820} 2) A.A. Ivanov, JGenChem(Russ) **16**, 648(1946) & CA **41**, 1202(1947)

1,4-Dinitro-2,3-dimethyl-butene-(2) $O_2N.CH_2.C(CH_3):C(CH_3).CH_2.NO_2$; mw 174.16, N 16.09%; ndls (from alc), mp 71.5–72°; mod sol in eth acet, chlf & hot alc; sl sol in eth; was obtd with other products when 2,3-dimethyl-1,3-butadiene in ether was treated with ice-cold N_2O_3 and the liq N_2O_3 adduct was steam distilled and crystd on standing over sulfuric acid (Refs 1 & 2). Its expl props are not reported

Refs: 1) Beil **1**, {820} 2) A.A. Ivanov, JGenChem(Russ) **16**, 653(1946) & CA **41**, 1202(1947)

Di(methyl)-butene-1 and Derivatives

3,3-Di(methyl)-butene-1; 3,3-Di(methyl)-a-butylene or *Bis(methyl)-butene-1,* $H_2C:CH.C(CH_3)_2CH_3$; mw 84.16; may be considered as the parent compd of its nitrated deriv although not used to prep it (Ref 1):

3,3-Di(nitratomethyl)-butene-1 or 3,3-Bis(nitratomethyl)-butene-1, $H_2C:CH.C(CH_2ONO_2)_2CH_3$; mw 206.16, N 13.59%; oxygen-rich monomer; no props are reported except IR spectrum; was prepd by reaction of ethyl methyl ketone with formaldehyde, hydrogenation of the product with Cu chromite catalyst, acetylation to the triacetate, pyrolysis, deacetylation, and nitration of 3,3-bis(hydroxymethyl) -butene-1. The subject compd was synthesized as a binder constituent, which might be polymerized, of propellants for rockets having a favorable oxygen balance (Ref 3)

Refs: 1) Beil **1**, 217, (191) & {814}
2) Beil, not found (Dinitrato) 3) L. P. Kuhn & A.C. Duckworth, "Preparation of 3,3,3-Tris(nitratomethyl)-propene-1 and 3,3-Bis(nitratomethyl) -butene-1", BRL(Ballistic Research Laboratory), Memorandum Report No **1230**(1959), Project No 503-02-001 and TB 3-0110

1,3-Dimethyl-5-tert-butyl-benzene and Derivatives

1,3-Dimethyl-5-t-butyl-benzene, $(H_3C)_2C_6H_3.C(CH_3)_3$, mw 162.26, liq, fr p 21.5°, bp 205.5–206.5°, d 0.8619 at 30°, n_D 1.4935 at 25°(Ref 1)

2-Azido-4,6-dinitro-1,3-dimethyl-5-tert-butylbenzene, $(H_3C)_2C_6(NO_2)_2(N_3).C(CH_3)_3$, mw 293.28, N 23.88%; wh pltlts (from alc), mp 89° & 146°; sol in org solvs; insol in w; was prepd by reaction of NH_3 on the 4,6-dinitro-2-amino deriv, diazotizing and converting with K perbromide soln (Ref 2)

2,4,6-Trinitro-1,3-dimethyl-5-tert-butylbenzene, $(H_3C)_2C_6(NO_2)_3.C(CH_3)_3$; ndls (from alc), exist in a RT stable form, mp 107°; form I, mp 114°; and a form III, converted from form I; was prepd by nitrating the parent compd with nitric acid (d 1.5) & concd sulfuric acid (Refs 3 & 4). Its expl props are not reported

Refs: 1) Beil **5**, 447, (213), [339] & {1032}
2) Beil **5**, 448 & [340] 3) Beil **5**, 448, (213), [340] & {1043} 4) D.V. Nightingale & L.I. Smith, JACS **61**, 101, 103(1939) & CA **33**, 1681(1939)

Di(3-methylbutynyl)-3-peroxide. See Bis(1,1-dimethyl-2-propynyl) -peroxide in Vol 1 of Encycl, p A66-R, under item C) Dialkynyl peroxides

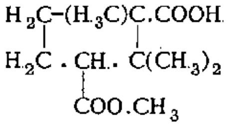
Dimethylbutyric Acid and Derivatives

2,2-Dimethylbutyric Acid, $H_3C.CH_2.C(CH_3)_2.COOH$; mw 116.16, liq, fr p 14°, bp 187°, d 0.9276 at 20°/4°; n_D 0.9276 at 20°; forms several metallic salts (Ref 1)

3,3-Dinitro-2,2-dimethylbutyric Acid, $H_3C.C(NO_2)_2.C(CH_3)_2.COOH$; mw 206.16, N 13.59%; monoclinic crysts (from alc), volatile at 100°, mp 215° (dec); sol in alc- sl sol in hot w; v sl sol in cold w; was prepd by heating camphor with nitric acid; forms several cryst metallic salts, some of which are unstable on heating, for example, *Barium salt*, $Ba(C_6H_9N_2O_6)_2+3H_2O$ or $5H_2O$, deflgr on heating above 120° (Refs 2 & 3)
Refs: 1) Beil **2**, 335, (143), [293] & {756}
2) Beil **2**, 337 (Dinitrodimethylbutyric Acid)
3) Kullheim, Ann **163**, 231 (1872) & Kachler, Ann **191**, 143, 156 (1878)

Dimethyl-Cadmium or **Cadmium Dimethyl,** $(CH_3)_2Cd$, mw 142.48, col liq, sp gr 1.985 at 17°/4, fr p –4.5°, bp 105.5° at 756mm; explodes at higher temp; sol in ether; decomp by w; can be prepd by reaction of methyl Mg bromide & $CdCl_2$, or from methyl Mg iodide & $CdBr_2$ in ether (Refs 1 & 2). At 70° vapor explodes in air
Refs: 1) Beil **4**, 677, (611), [1047] & {2002}
2) R.D. Anderson & H.A. Taylor, JPhysChem **56**, 161–62 (1952) & CA **46**, 7387 (1952) (Prepn & some physical constants)

Dimethylcamphorate (Camphorate de methyle in Fr),



$$\begin{array}{l} H_2C-(H_3C)C.COOH \\ \;\;|\qquad\qquad\;\; | \\ H_2C\;.\;CH.\;C(CH_3)_2 \\ \qquad\;\; | \\ \qquad COO.CH_3 \end{array}$$

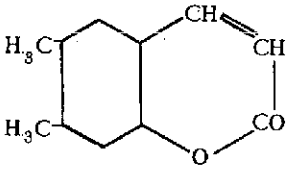
mw 182.25; liquid which can be distilled under reduced pressure; was prepd in France by esterifying disodium camphorate with dimethyl sulfate and tried as a possible substitute of Centralite in solventless, smokeless propellants [poudres SD (sans dissolvant)]. It was found, however, to be unsatisfactory as a stabilizer
Refs: 1) Beil, not found 2) R. Dalbert & H. Ficheroulle, MP **30**, 283–300 (1948)

Dimethylcarbamoyl Azide, $(H_3C)_2N.CON_3$, mw 114.11, N 49.10%; liq, bp 59° at 15mm press; explodes violently when heated in a free flame; was prepd by long heating of dimethylcarbamide chloride with NaN_3 in acetone (Refs 1 & 2)
Refs: 1) Beil **4**, [575] 2) R. Stollé et al, JPraktChem [2] **117**, 201 (1927) & CA **22**, 422 (1928) 3) F.L. Scott, Chem & Ind **1954**, 959 & CA **49**, 15 (1955) (Carbamyl migration)

N,N-Dimethyl-carbanilide. See Centralite 2 in Vol 2 of Encycl, pp C137ff

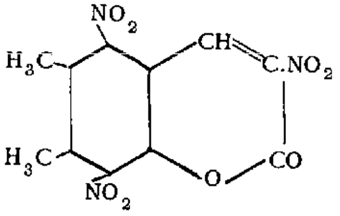
Dimethylcoumarin and Derivatives

6,7-Dimethylcoumarin or *Dimethylcumarin,*



mw 174.19; ndls (from alc), mp 148–49°; was prepd by heating o-xylenol, malic acid & concd sulfuric acid (Ref 1)

3,5,8-Trinitro-6,7-dimethylcoumarin,



mw 309.19, N 13.59%; yel prisms (from glac acet ac), mp 213–16° (dec); was prepd by nitrating the parent compd with an excess of mixed acid in the cold (Refs 2 & 3). No expl props are reported
Refs: 1) Beil **17**, 342 & (180) (Dimethylcumarin) 2) Beil **17**, (181) (Trinitrodimethylcoumarin) 3) A. Clayton, JChemSoc **97**, 1399 (1910)

Dimethylcumene and Derivatives

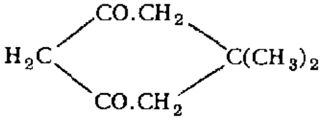
3,5-Dimethylcumene or *Dimethylpropylbenzene,* $(H_3C)_2C_6H_3.CH_2.C_2H_5$, mw 148.24; liq, bp 206–210°; prepd with other products by mixing 4 vols acetone, 2 vols methylpropylketone & 3 vols sulfuric acid (Ref 1)

2,4,6-Trinitro-3,5-dimethylcumene, $(H_3C)_2C_6(NO_2)_3.CH_2.C_2H_5$, mw 283.24, N 14.84%; crysts, mp 98.5–99°; was prepd by nitrating the parent compd (Ref 3). Its expl props are not reported

Refs: 1) Beil **5**, 440 (Dimethylcumene) 2) Beil **5**, not found (Trinitrodimethylcumene) 3) D.V. Nightingale & J.M. Shackelford, JACS **78**, 1226(1956) & CA **50**, 12856(1956)

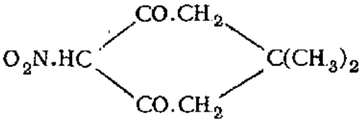
Dimethylcyclohexanedione and Derivatives

5,5-Dimethyl-1,3-cyclohexanedione; Dimedone or *1,1-Dimethyl-3,5-diketocyclohexane,*



mw 140.18; long, thick citron-yel ndls (from w), col prisms (from w) or lt-yel crysts (from dil acet); mp 145–50°, begins to decomp above 130°; volatile in steam; sol in chlf, benz & eth acet; sl sol in eth; v sl sol in petr eth; can be prepd by refluxing diethyl malonate & mesityl oxide with Na ethylate soln, adding NaOH soln and refluxing again, and addg dil HCl to the reaction product. The compd forms numerous salts which decomp on heating (Refs 1 & 4)

2-Nitro-5,5-dimethyl-1,3-cyclohexanedione or **4-Nitro-1,1-dimethyl-3,5-cyclohexanedione,**

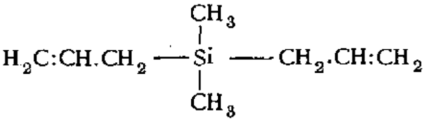


mw 185.18, N 7.56%; col pltlts (from eth + petr eth), mp 102–105°; sl sol in cold w; v sl sol in alc, eth & benz; was prepd by nitrating Dimedone with fuming nitric acid (d 1.5) while cooling with NaCl-ice mixt, and by other methods (Refs 2 & 3). The product & Na_2CO_3 gave a *Sodium salt,* $C_8H_{10}NO_4Na$, yellow crysts; with Ag_2O a *Silver salt,* $C_8H_{10}NO_4Ag$, yellow; with PbO in alc a yel *Lead salt,* $(C_8H_{10}NO_4)_2Pb$; with a hot suspension of CuO a green *Copper salt,* $(C_8H_{10}NO_4)_2Cu$; similarly a *Cobalt salt,* $(C_8H_{10}NO_4)_2Co$; and refluxing with FeS in alc gave violet *Iron salt,* $(C_8H_{10}NO_4)_2Fe$ which on heating decomposed with a flame, characteristic of all the above salts, *Silver* being the least stable (Ref 3)

Refs: 1) Beil **7**, 559, (313), [531] & {3225} 2) Beil **7**, 563 3) E. Gudriniece et al, ZhObshKhim **24**, 1863–66(1954) & CA **49**, 13128(1955) 4) CondChemDict (1961), 390

3,8-Dimethyl-4,6-decdiyn-3,8-dihydroperoxide. See under Diacethylenic Dihydroperoxides in this Vol, p D1120-R

Dimethyldiallylsilane,



$$H_2C{:}CH.CH_2-\underset{\displaystyle CH_3}{\overset{\displaystyle CH_3}{Si}}-CH_2.CH{:}CH_2$$

mw 140.27; liq, bp 124–25° at 760mm, n_D 1.4411 at 20°; was prepd by Schaffner from allylchloridedimethyldichlorosilane and Mg turnings suspended in ether as described in Refs 3 & 4. This procedure is essentially the same as that described by Levi & Scaife (Ref 2). It ignited in nitrogen tetroxide

Reaction with nitrogen tetroxide produced very unstable nitro-nitrates. On two occasions violent explosions took place in USRubberCo Laboratory at Passaic, NJ

Note: In view of remarkable instability of nitrated derivs of silanes and obvious force of detonation, it is suggested to use mono-, di-, tri-, and tetra-allylic silanes and nitrogen tetroxide in liquid rocket propellants. It is also likely that *vinylsilanes* will exhibit similar behavior

Refs: 1) Beil – not found 2) M.N. Levy & C.W. Scaife, JCS **1946**, 1086 3) I.J. Schaffner, "Quarterly Report No 2", Jan 30–Apr 30, 1948; Contract Nord 10129, USRubberCo, Passaic, NJ, pp 4, 8–9 4) Ibid, Rept No 3, Apr 30–July 31, 1948, pp 17 & 28

N,N-Dimethyl-diaminobenzene, Same as 1,3-Di(methylamino)-benzene, described here as N,N-Dimethyl-m-phenylenediamine

Dimethyldiaminoethane. Same as Dimethylethylenediamine

Dimethyldiaminopropane. Same as Dimethylpropylenediamine

Dimethyl-diaminotartaric Acid and Derivatives

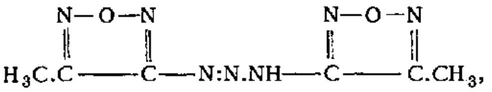
N,N'-Dimethyl-diaminotartaric Acid or *N,N'-Dimethyl-succinamide,* $H_3C.HN.CO.CH_2.CH_2.CO.NH.CH_3$; mw 144.17, N 19.43%; crysts (from acet + eth), mp 174–75°; readily sol in alc & w; sl sol in acet & warm eth acet; v sl sol in CCl_4, eth & benz (Ref 1). Ref 3 gives mp 189° & Ref 4, mp 213–14°

N,N'-Dinitro-N,N'-dimethyl-diaminotartaric Acid Dinitrate or **N,N'-Dinitro-N,N'-dimethyltartardiamide Dinitrate,** $(H_3C)(O_2N)N.CO.CH(ONO_2).CH(ONO_2).CO.-N(NO_2)(CH_3)$; mw 356.17, N 23.60%, OB to CO_2 minus 18%; crysts, (from CCl_4), mp 114°, bp expl at 117° (when heated at the rate of 20° per min); sol in alc, acet,benz, chlf and CCl_4. It can be prepd by dissolving N,N'-dimethyldiaminotartaric acid in acetone at –5 to 0°, adding slowly concd nitric acid and then acetic anhydride

It is a very powerful expl with sensitivity to shock between those of MF and LA, but stable below its mp. It was proposed for use in primary mixtures (Refs 2 & 4)

Refs: 1) Beil **4**, 62, (330) & {132} 2) Beil. **4**, {169} (Nitrated) 3) P. Frankland & A. Slator, JCS **83**, 1360(1903) 4) T. Urbański, RocznikiChem **16**, 334(1936) & CA **31**, 1770 (1937)

4,4'-Dimethyl-3,3'-diazoaminofurazan,

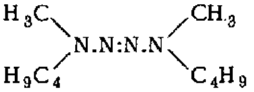


mw 269.17, N 46.88%; hydrated pltlts (from dil alc), dec in light, mp 114° & decomp at higher temp; anhyd crysts sol in cold alc, eth, acet, hot petr eth, chlf & benz; forms a **Silver salt,** $AgC_6H_6N_7O_2$, powd, which explodes violently on heating. Parent compd prepd by treating 4-amino-3-methylfurazan with $NaNO_2$ in aq HCl or sulfuric acid (Refs 1 & 2)

Refs: 1) Beil **27**, [868] 2) G. Ponzio & G. Ruggeri, Gaz **53**, 305(1923)

2,5-Dimethyl-2,5-di(t-butylperoxy)-3-hexyne. See Vol, 1 of Encycl, p A66-R under Acetylene Hydroperoxides and Peroxides

N,N'-Dimethyl-N,N'-dibutyl-tetrazene(1) or **[1,IV-Dimethyl-1,IV-dibutyl]-tetrazene(2)**

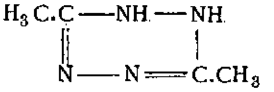


mw 200.32, N 27.97%, oily liq, sp gr 0.8798 at 15°, fr p – did not solidify at minus 20°; bp 119–20° at 18mm pressure; deflagrates on rapid heating; very sl sol in w; was prepd by Franchimont & van Erp by treating cooled ethereal soln of N-methyl-N-butyl hydrazine, $(H_3C)C_4H_9.N.NH_2$, with yel mercuric oxide

Refs: 1) Beil. **4**, 579 2) A.P.N. Franchimont & H. van Erp, Rec **14**, 321(1895) & JCS **70** (Abs 1), 275(1896)

2,5-Dimethyl-2,5-dihydroperoxy-3-hexyne. See 1,1,4,4-Tetramethyl-2-butynylene-dihydroperoxide in Vol 1, p A66-R under Acetylene Hydroperoxides or Peroxides

3,6-Dimethyl-1,2-dihydro-1,2,4,5-tetrazine,



mw 112.14, N 49.97%; cryst + $1H_2O$, mp – deliq at 100°; readily sol in water; insol in eth; prepd by heating acetonitrile with anhyd hydrazine (Refs 1 & 2). No expl props are reported

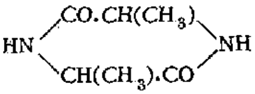
NOTE: No *Azido,* $C_4H_7N_7$, *Mononitro,* $C_4H_7N_5O_2$, or *Dinitro,* $C_4H_6N_6O_4$, derivs were found in the literature thru 1966

Refs: 1) Beil **26**, (111) 2) T. Curtius et al, Ber **48**, 1632(1915)

1,1-Dimethyl-3,5-diketo-cyclohexane. Same as 5,5-Dimethyl-1,3-cyclohexanedione or Dimedone

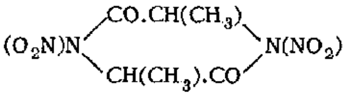
Dimethyldiketopiperazine and Derivatives

3,6-Dimethyl-2,5-diketopiperazine or *3,6-Dimethyl-2,5-dioxopiperazine,*



mw 142.16; N 19.71%; crysts, mp 280°; was first prepd by Franchimont & Friedmann (Ref 3). Shibata & Asahina prepd it (Ref 4) by heating 132g of dl-alanine(2-aminopropanoic acid) with 246g of glycerol in an oil bath at 160–80° for 6 hours. After allowing to cool to RT, the semi-solid residue was mixed with 30ml of 95% alcohol and filtered. The crude product was dissolved in hot w, the soln decolorized with activated charcoal, filtered while still hot, cooled and resulting ppt removed and dried. The yield was 75g or 66.8%

1,4-Dinitro-3,6-dimethyl-2,5-diketopiperazine or **1,4-Dinitro-3,6-dimethyl-2,5-dioxopiperazine,**



mw 232.16, N 24.14%, OB to CO_2 minus 68.9%; wh ndls (from hot chlf), mp 137° (decomp), bp – puffs off at above mp catching fire occasionally but does not explode even at 360°; nearly insol in cold w; sol in eth and hot chlf; was first prepd by Franchimont & Friedmann(Refs 1 & 2). Shibata & Asahina prepd it by adding 15g of 3,6-dimethyl-2,5-diketopiperazine to a mixture of 37.5ml acetic anhydride and 50ml white nitric acid. The resulting soln was warmed to 40–50° for 10 mins and, after allowing to cool to RT was poured with vigorous stirring onto cracked ice. The solid was collected on filter, washed with cold w, dried and recrystallized from hot chlf. Other method is given in Refs 1 & 2

Dinitro compd is an expl comparable in sensitivity to impact to TNT; it is very hygroscopic and hydrolizable by hot w; does not possess good thermal stability (decomposes when heated at 75° for 48 hrs); heat of combustion 759.1 kcal/mole (Refs 2,3,4 & 5)

Refs: 1) Beil **24**, 297, (308) & [159, 160] 2) Beil **24**, 299 3) A.P.L. Franchimont & H. Friedmann, Rec **27**, 192 (1908) 4) Y. Shibata & T. Asahina, BullChemSoc(Japan) **1**, 71 (1926) & CA **20**, 2502 (1926) 5) R. Adams & C.S. Marvel, OSRD **86**(1941), pp 10, 29–30 & 56

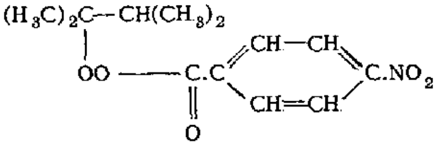
Dimethyl-dimethylene-triamine and Derivatives

1,5-Dimethyl-2,4-dimethylene-1,3,5-triamine or *2,4,6-Triazaheptane,* $H_3C.NH.CH_2.NH.CH_2.NH.CH_3$, mw 103.17, N 40.73%; may be considered the parent compd of its trinitro deriv, although not used to prep it:

1,5-Dimethyl-2,4-dimethylene-1,3,5-trinitramine or **2,4,6-Trinitro-2,4,6-triazaheptane,** $CH_3.N(NO_2).CH_2.N(NO_2).CH_2.N(NO_2).CH_3$ mw 262.19, N 32.06%; crysts (from alc), mp 169.5–170°; was prepd by adding $(O_2NN.CH_3CH_2)_2NH.HCl$ at 0° to HNO_3, allowing the temp to rise to 20° and pouring the mixt into ice-water (Refs 2 & 3). No expl props are reported

Refs: 1) Beil – not found 2) J. Majer & J. Denkstein, CollCzechChemCommun **31** (6), 2547 (1966) (Ger) & CA **65**, 7042 (1966) 3) J. Denkstein & V. Kaderbek, Ibid **31** (7), 2904 (1966) (Ger) & CA **65**, 10483 (1966)

1,1-Dimethyl-2,2-dimethyl-ethane-1-hydroperoxide-nitrobenzoate,



mw 267.27, N 5.24%; colorless pltlts, mp 57–58°, exploding in a flame; the hydroperoxide is prepd by treating hydrogen peroxide at 0° with small amts of sulfuric acid and the mixt treated cautiously with dimethylisopropyl carbinol and allowed to stand overnight in a refrigerator. It was converted to the nitrobenzoate by interaction of the hydro-

peroxide with $4\text{-}O_2N.C_6H_4COCl$, after treatment with sulfuric acid and acet extraction (Ref 2)

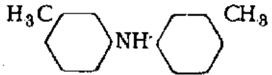
Refs: 1) Beil – not found 2) R. Criegee & H. Dietrich, Ann **560**, 139(1948) & CA **43**, 6189(1949)

Di[N(methyldinitrophenyl)-nitramino]-propane. See 1,3-Bis[N-(4'-methyl-2',6'-dinitrophenyl)-nitramino]-propane in Vol 2, p B151-L

3,7-Dimethyl-3,7-dinitro-1,3,5,7-tetrazacyclooctane. See under Dimethyl-tetrazacyclooctane and Derivatives

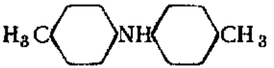
Dimethyldiphenylamine and Derivatives

3,3'-Dimethyldiphenylamine or *Bis(3-methylphenyl)-amine,*



mw 197.27, N 7.10%; liq, fr p 12°. Prepn and other props are in Ref 1

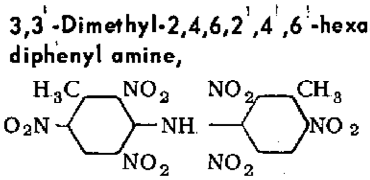
4,4'-Dimethyl-diphenylamine or *Ditolylamine,*



wh crysts, mp 79°; v sol in cold acet & benz and in hot alc & chlf; darkens easily on exposure to light; can be prepd as described in Beil (Ref 2) or by the method of Tavernier & Lamouroux (Ref 7)

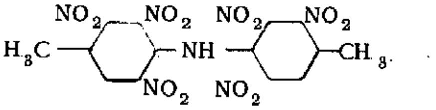
Nitro-, Dinitro- and Trinitro-diphenylamines are known and are listed in Vol 1 of Encycl, p A443 under Anilinoxylenes and Derivatives. None of these compds seems to be explosive

3,3'-Dimethyl-2,4,6,2',4',6'-hexanitrodiphenyl amine,



mw 467.27, N 20.98%; brn tablets (from alc), mp 60°. Can be prepd by warming 4,6-dinitro-3,3'-dimethyldiphenylamine with concd nitric acid on a water bath. Its expl props were not investigated (Refs 3 & 6)

4,4'-Dimethyl-2,3,5,2',3',5'-hexanitrodiphenylamine,

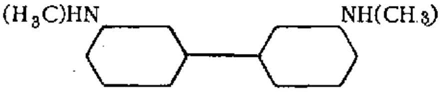


yel crysts, mp 258°; very diff sol in org solvents; can be prepd by treating with fuming nitric acid, either p,p -ditolylamine or nitroso-p,p -ditolylamine. Its expl props were not investigated (Refs 4 & 5)

Refs: 1) Beil **12**, 858 & [467] 2) Beil **12**, 907, (415) & [494] 3) Beil **12**, [482] (Hexanitro-3,3'-dimethyl-diphenylamine 4) Beil **12**, 1013 (Hexanitro-4,4'-dimethyl-diphenylamine) 5) A. Lehne, Ber **13**, 1545(1880) (Hexanitro-4,4 -dimethyl-diphenylamine) 6) L. Brady et al, JCS **125**, 2404(1924) (Hexanitro-3,3'-dimethyldiphenylamine) 7) P. Tavernier & M. Lamouroux, MP **38**, 75 & 84(1956) (4,4 -Dimethyldiphenylamine)

Dimethyldiphenyldiamine and Derivatives

N,N'-Dimethyl-3,3'-diphenyldiamine or *3,3'-Di(methylamino)-diphenyl,*



mw 212.28, N 13.20%, may be considered as the parent compd of its nitrated derivs, although not used to prep them:

4,6,4',6'-Tetranitro-N,N -Dimethyl-3,3'-di(methylamino)-diphenyl, $(O_2N)_2C_6H_2(NH.CH_3)-C_6H_2(NH.CH_3)(NO_2)_2$; mw 392.28, N 21.43%; yel powd, mp – explodes when heated above 360°; turns reddish-brn when exposed to light; was obtd when tetranitro-3,3 -dichloro-diphenyl was heated with methylamine & alc for 5 hrs at 100° (Ref 3)

N,N',4,4',6,6'-Hexanitro-N,N'-dimethyl-3,3'-diphenyldiamine or **4,4',6,6'-Tetranitro-3,3'-di(methylnitramino)-dephenyl,** $(O_2N)_2C_6H_2(N.NO_2CH_3)-C_6H_2(N.NO_2.CH_3)-(NO_2)_2$; mw 482.28, N 23.24%; yel powd, mp 210°(dec) & explodes at 225°; sol only in acet; was prepd from the tetranitro deriv by dissolving it in abs nitric acid at –10° & adding cold water (Ref 3)

Refs: 1) Beil – not found (Dimethyldiphenyldiamine) 2) Beil – not found (Nitrated derivs) 3) J. van Alphen, Rec **51**, 364(1932) & CA **26**, 2447(1932)

Dimethyldiphenylether and Derivatives

Dimethyldiphenylether or *Dimethylphenyloxide*, $(H_3C)C_6H_4.O.C_6H_4(CH_3)$, mw 194.22. Three derivs are known:
2,2'-Dimethyldiphenylether, liq, bp 274°, d 1.047 at 24.3°(Ref 1)
3,3'-Dimethyldiphenylether, liq, bp 284°, d 1.0323 at 21°(Ref 2)
4,4'-Dimethyldiphenylether, prisms (from MeOH), mp 50°, bp 285°(Ref 3)

Other props & methods of prepn are given in the Refs

Tetranitrodimethyldiphenylether, $(H_3C)C_6H_2(NO_2)_2.O.C_6H_2(NO_2)_2.CH_3$; mw 378.25, N 14.81%. Five isomers are known:
4,6,4',6'(?)-Tetranitro-2,2'-dimethyldiphenylether, powd, mp 115°; was prepd by nitrating the 2,2'-dimethyl deriv with concd nitric and sulfuric acids (Ref 4, p180)
2,6,2',6'(?)-Tetranitro-3,3'-dimethyldiphenylether, hexag pltlts, mp 147°; sol in hot alc; obtd with other isomers on nitrating the 3,3-dimethyl deriv with concd sulfuric acid & fuming nitric acid in the cold (Ref 4, p194)
x,x,x,x-Tetranitro-3,3'-dimethyldiphenylether, wh powd, mp 203°; insol in hot alc; prepd by nitrating the 3,3'-dimethyl deriv with mixed acid in the cold (Ref 4, p194)
2,6,2',6'-Tetranitro-4,4'-dimethyldiphenylether, tablets (from alc or glac acet ac), mp 246°; sl sol in acet, v sl sol in eth, chlf & benz; insol in cold glac acet ac,& petr eth; obtd with other isomers on nitrating the 4,4'-dimethyl deriv with mixed acid at 10° (Ref 1, p392 & Ref 6)
x,x,x,x-Tetranitro-4,4'-dimethyldiphenylether, yel ndls, mp 84°; prepd by warming the 4,4'-dimethyl deriv with fuming nitric acid in glac acet ac (Ref 4, p207 & Ref 5)

Their expl props were not detd

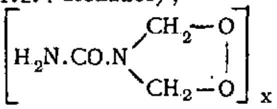
Refs: 1) Beil **6**, 353, (171) & {1250}
2) Beil **6**, 377, (186), [352] & {1303}
3) Beil **6**, 394, (200), [377] & {1360}
4) Beil **6**, (180, 194, 207) & [392] 4) A. Mailhe, BullFr [4] **13**, 171–72(1913)
6) J. Reilly et al, JChemSoc **1927**, 71

Dimethyl-diphenyl-sulfide. See Di(tolyl)-sulfide in this Vol

N,N'-Dimethyl-N,N'-diphenyl-urea. See Centralite 2 in Vol 2 of Encycl, p C137-L and the following addnl refs: A. Apard, MP **22**, 180(1926); H. Gnamm, "Die Lösungsmittel und Weichmachungsmittel", Wissensch-Verlages, Stuttgart(1946); CondChemDict (1961), 395-L

N,N-Dimethyl-diphenylylamine. See N,N-Dimethyl-aminobiphenyl in this Vol

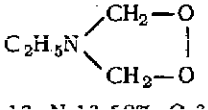
Dimethyleneperoxidecarbamide (Dimethylenperoxyd-carbamid, in Ger) (Was formerly called Tetramethylen-diperoxyd-dicarbamid) (On p [48] of Vol 3, Beilstein called it 4-Aminoformyl-1.2.4-dioxazol),



mw $(118.09)_x$, N 23.72%; crysts, mp – explodes on heating; insol in w, ethanol, methanol, chlf, pyridin and other org solvents; dissolves in concd nitric or sulfuric acids and may be pptd from these solns by w. Was prepd (Refs 1 & 2) by dissolving with cooling urea (1 mole), formaldehyde (2 moles) and 30% hydrogen peroxide in concd nitric acid (See also Ref 3)

Refs: 1) Beil **3**, (27), [48] & {112}
2) C. von Girsewald & H. Siegens, Ber **47**, 2464–66(1914) 3) Ibid, Ber **54**, 493(1921)

Dimethyleneperoxide-ethylamine (Dimethylenperoxyd-äthylamin, in Ger),



mw 103.12, N 13.58%, O 31.03%; colorless oil; bp – expl on heating and puffs off strongly in flame; unstable even at RT; insol in w; sl sol in alc & in eth. It can be prepd by the action of hydrogen peroxide on formaldehyde in the presence of ethylamine

Refs: 1) Beil **27**, [523] 2) F.C. von Girsewald & H. Siegens, Ber **54**, 492 & 497(1921); CA **15**, 2417(1921) 3) J.F. Walker, "Formaldehyde", Reinhold, NY(1953)

Dimethylether (Methylether or Methyloxide), $CH_3.O.CH_3$, mw 46.07; col gas; flammable and explosive, fr p 141.50°, bp 24.82°. Other props & prepn in Refs

Refs: 1) Beil **1**, 281, (139), [269] & {1188} 2) H. Aull, Erdöl u Kohle **3**, 195–96(1950) & CA **44**, 7539(1950) 3) CondChemDict (1961), 395-R

Di(methylether)-peroxide or **Bis(methylmethyl) -peroxide,** $H_3C.O.CH_2—O—O—CH_2.O.CH_3$, mw 74.12, O 21.59%; crysts; obtd when ether solns of HCHO and of hydrogen peroxide were brought together and the ether evapd. There is also formed the very expl compd, hydroxymethyl hydroperoxide (Ref 2)

Refs: 1) Beil, not found 2) A. Rieche & R. Meister, Ber **68B**, 1465–73(1935) & CA **29**, 6877(1935)

Dimethyl-ethyl-acetate and Derivatives

1,1-Dimethyl-ethylacetate, $H_3C.C(CH_3)_2COOCH_3$; mw 116.16; may be considered as the parent compd of its trinitro deriv although not used to prep it.

1,1-Dimethyl-2,2,2-trinitroethylacetate, $(O_2N)_3C.C(CH_3)_2.COOCH_3$; mw 251.16, N 16.73%; mp 25–26°; was obtd when a mixt of nitroform, acetic anhydride & acetone was allowed to stand at RT for four days, the solv was stripped off and the residue distilled (Ref 3). Its expl props were not reported

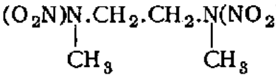
Refs: 1) Beil **2**, not found (Dimethyl-ethyl-acetate) 2) Beil, not found (Dimethyl-trinitroethylacetate) 3) J.C. Conly et al, "USRubberCo Quarterly Report No 28", Aug 1–Nov 1, 1954, p 10(bottom)

Dimethylethylenediamine and Derivatives

N,N'-Dimethylethylenediamine; 2,5-Diazahexane or *N,N'-Dimethyl-N,N'-diaminoethane,* $(H_3C)HN.CH_2.CH_2.NH(CH_3)$, mw 88.15, N 31.78%. Prepn and props are given in Ref 1

N,N'-Dimethyl-N,N'-dinitroso-ethylenediamine or **2,5-Dinitroso-2,5-diazahexane,** $(H_3C)(ON)N.CH_2.CH_2.N(NO)(CH_3)$, mw 148.17, N 37.82%; lt yel-grn pltlts (from alc + eth), mp 60–61°; sol in alc; sl sol in eth; was prepd by reaction of $NaNO_2$ on a HCl soln of the parent compd (Ref 2). Its expl props are not reported

N,N'-Dimethyl-N,N'-dinitroethylenediamine; Me₂EDNA or **N,N'-Dimethyl-ethylenedinitramine;** *2,5-Dinitro-2,5-diazahexane; N,N'-Dimethyl-N,N'-di(nitroamino)-ethane* or *N,N'-Bis(methyl) -ethylenedinitramine,*



$$(O_2N)\underset{\substack{|\\CH_3}}{N}.CH_2.CH_2.\underset{\substack{|\\CH_3}}{N}(NO_2);$$

mw 178.15, N 31.45%; O Bal to CO_2 minus 80.6%; crysts, mp 137.5–138°; does not explode to 360°

Accdg to investigation conducted by NDRC, Div 8 during WWII, it is an expl sl less powerful than TNT (95% by *Ballistic Mortar Test*); *Impact Sensitivity by Bruceton No 3 Machine* – positive with 5kg wt >90cm vs 48–50cm for RDX; *Thermal Stability* at 135° – not acid in 300 mins; *Vacuum Stability* at 120° – 0.72cc to 0.91cc/5g in 48 hrs; *Hygroscopicity* at 25° & RH 90% 0.02% and at RH 100% 1.92% (Refs 6 & 6a)

Its mixt with 25% of MeEDNA gave mp 102–103° and practically the same with 50%. Its mixt with 25% EDNA gave mp 125°, while with 50% it was 145° (Ref 6a)

Me_2EDNA was first prepd by Franchimont & Klobbie (Ref 4) by alkylation of ethylenedinitramine, $(O_2N)HN.CH_2.CH_2.NH(NO_2)$, with methyliodide, CH_3I, and also with methylsulfate, $(CH_3)_2SO_4$. Its structure was not definitely established until 1912 by Backer (Ref 5)

Blomquist & Fiedorek reported its prepn in mixture with MeEDNA (Methylethylenedinitramine) by treating EDNA (Ethylenedinitramine) with methylbromide, CH_3Br (Ref 6a)

Römer (Ref 7) prepd it during WWII in Germany and proposed to use it in the following mixtures, claimed to be suitable for military purposes: a) Me_2EDNA 12, RDX 50, R-Salz 36 & unaccounted 1%;

b) Me_2EDNA 2.5, RDX 96.5 & DPhA (Diphenylamine) 1% (See also Ref 11)
Note: R-Salz is Cyclotrimethylenetrinitrosamine, which is described in Vol 3 of Encycl, p C630-R. RDX is described as Cyclotrimethylenetrinitramine on pp C626ff

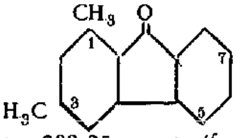
Ultraviolet spectrum of Me_2EDNA was detd in Canada (Ref 8)
Refs: 1) Beil **4**, 250, (415), [689] & {512} (N.N'-Dimethyl-äthylendiamin) 2) Beil **4**, 256 (N.N'-Dinitroso-N.N'-dimethyl-äthylendiamin) 3) Beil **4**, 573, (416) & {546} (N.N'-Dinitro-N.N'-dimethyl-äthylendinitramin) 4) A.P.N. Franchimont & E.A. Klobbie, Rec **7**, 346–47(1888) 5) F. Backer, Rec **31**, 172(1912) 6) A.H. Blatt, OSRD **2014** (1944) 6a) A.T. Blomquist & F.T. Fiedorek, OSRD **4134**(1944) & PB **18867**, pp 6, 67–8 & 74 7) G. Römer, "Report on Explosives", PBL **85160**(1946) 8) R.N. Jones & G.D. Thom, CanJRes **27B**, 830(1949) 9) R.H. Meen & G.F. Wright, JACS **74**, 2079(1952) 10) ADL Synth HE's 4th Rept (1956), p93 11) Fedoroff et al, PATR **2510** (1958), p Ger 36–R

asym-**Dimethylethyleneglycol.** See iso-Butanediol in Vol 2 of Encycl, p B370-L

sym-**Dimethylethyleneglycol.** See Butanediol in Vol 2, p B368-R

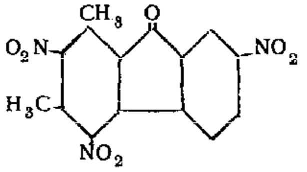
Dimethyl-9-fluorenone and Derivatives

1,3-Dimethylfluorenone or *Diphenyleneketone,*



mw 208.25; crysts (from MeOH), mp 112°; forms an oxime, mp 197–98°. Prepn: N-tosylanthranilyl chloride with m-xylene & AlC_3 gives 2,4-methyl-2'-(tosylamino)benzophenone which is saponified by sulfuric acid & the product is diazotized with $NaNO_2$ (Ref 3)

2,4,7-Trinitro-1,3-dimethyl-9-fluorenone,



mw 343.25, N 12.24%; pltlts (from benz or acet), mp 236°. Preparation: nitration of parent compd with KNO_3 in H_2SO_4 gives a *Dinitro deriv*, mp 238°; nitration of parent compd with nitric and sulfuric acids gives the *Trinitro deriv.* Heating the Trinitro deriv with N_2H_4 6 hrs at 80° in a sealed tube gives 1,3-dimethylfluorene, mp 87°
Refs: 1) Beil, not found (Dimethylfluorenone) 2) Beil, not found (Trinitrodimethylfluorenone) 3) L. Chardonnens & A. Würmli, Helv **33**, 1338(1950) & CA **45**, 1558(1951)

N,N-Dimethylformamide (DMF) (Formyldimethylamine), $HC(:O).N(CH_3)_2$; mw 73.09, N 19.17%, OB to CO_2 minus 120.4%; colorless liquid; sp gr 0.9484 at 22.4°/4°(duPont's 0.9683 at 0°/4° and 0.9445 at 25°/4°), n_D 1.4294 at 22.4° (duPont's 1.4269 at 25°); fr p below minus 55° (duPont's minus 61°), bp 152.8°; flash point (Tag open cup) 153°F; viscosity 0.802cp at 25°; vapor pressure 8mm at 37.8°, 30mm at 62.4° and 100mm at 90°; sp heat 0.5; heat of vaporization 210 Btu/lb, heat of combstn 457.5 kcal/mole (In Ref 4 Q_c^v is given as 464.8 and Q_f^v 54.98). DMF is miscible in all proportions with water, ether, alcohols, esters, ketones and chlorinated compds. It can be prepd by distilling K formate with dry dimethylamine hydrochloride (Ref 1). It is available commercially from the DuPont Co (Ref 3). It is quite toxic when taken orally and less when inhaled or absorbed thru the skin. It is recommended that the concn of DMF in the atm be kept below 50ppm, preferably not above 20ppm. It has no corrosive action on ordinary metals, is not hydrolyzed by w even at 100°, but is decompd by strong alkalies and acids. DMF is relatively stable to heat and light (Refs 1, 3 & 6)

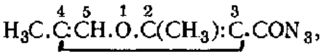
Due to its highly polar nature and unusual combination of functional groups, DMF is an excellent solvent for many compds, such as resins, petroleum products, inorganic and organic salts, lignin, plasticizers, etc. This includes numerous expls, among them TNT, RDX, NC, etc (Ref 3). Solubilities of some expls in DMF are given in Ref 4

Lawson (Ref 2) proposed using DMF as an accelerator in the gelatinization of NC by NG in proplnt and expl mixtures, such as Comp C-2

Refs: 1) Beil **4**, 58 & {122} 2) W. Lawson, USP 2159973(1939) & CA **33**, 7115(1939) 3) DuPont Co (GrasselliChemDept), Pamphlet: "Product Information on DMF", 8 pages and 23 refs 4) F-M. Lang, MP **35**, 213(1953) 5) L. Médard & M. Thomas, MP **39**, 196 & 207(1957) 6) CondChemDict (1961), 395-R 7) Kirk & Othmer, 2nd edit. **10**, 109(1966)

Dimethyl-3-furoyl Azide, $C_7H_7N_3O_2$; mw 165.15, N 25.45%. Two isomers are known:

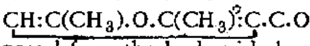
2,4-Dimethyl-3-furoyl Azide,



$H_3C.\underbrace{\overset{4}{C}:\overset{5}{C}H.\overset{1}{O}.\overset{2}{C}(CH_3):\overset{3}{C}}.CON_3$,

oil, no data given; prepd when the hydrazide in aq soln was cooled to 0–5°, $NaNO_2$ & excess acetic acid were added and the azide separated after 1 hr at 5–10°(Ref 2)

2,5-Dimethyl-3-furoyl Azide,



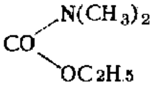
$\underbrace{CH:C(CH_3).O.C(CH_3)\overset{3}{:}C}.C.ON_3$; fr p 24–25°; prepd from the hydrazide by treating it with $NaNO_2$ in dil aq acetic acid and isolating the azide by extraction with benz (Refs 2 & 3)

No expl props of the azides are reported

Refs: 1) Beil, not found 2) A.T. Blomquist & H.B. Stevenson, JACS **56**, 148(1934) & CA **28**, 1696(2934) 3) H.B. Stevenson & J.R. Johnson, JACS **59**, 2529(1937) & CA **32**, 938(1938)

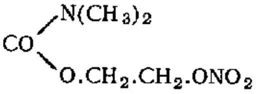
Dimethylglycolurethane and Derivatives

N,N-Dimethylglycolurethane or *Dimethylethylcarbamate,*



mw 117.15, N 11.96%; liq, bp 147°, d 0.9725 at 15°; prepd by various methods; decomp in warm concd sulfuric acid into carbon dioxide & dimethylamine (Ref 1)

N,N-Dimethylglycolurethane Nitrate,

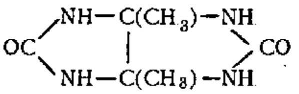


mw 178.15, N 15.73%; liq; bp 110° at 3mm; starts to decomp ca 140°. It was prepd by Desseigne in 85% yield, by adding 1 mole of N,N-dimethylglycolurethane to 5 moles 98% nitric acid, with agitation at temp not above 5°. It is a good gelatinizer for NC (Ref 3)

Refs: 1) Beil **4**, 73, (334) & {144} (Dimethylglycolurethane) 2) Beil, not found (Dimethylglycolurethane Nitrate) 3) G. Desseigne, FrP 1094959(1955) & MP **38**, 437–38(1938)

Dimethylglycoluril and Derivatives

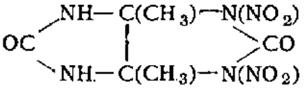
7,8-Dimethylglycoluril (DMGU) or *7,8-Dimethylacetylenediurein,*



mw 170.17, N 32.93%; ndls or plates (from w); mp – starts to brown at 290° without melting; bp decomp completely at 350–55°; sl sol in w & in alc; insol in eth, chlf and benz. It can be prepd either by allowing to stand an aq soln of diacetyl plus urea (Refs 1 & 2) or by allowing to stand an alcoholic soln of urea and of diacetylmonoxime in presence of small amt of concd sulfuric acid (Refs 1&3)

On nitration it gives an explosive dinitro compound

1,3-Dinitro-7,8-dimethylglycoluril (DNDMGU) or **1,3-Dinitro-7,8-dimethylacetylenediurein,**



mw 260.17, N 32.31%; OB to CO_2 minus 61.5%. (Formula not definitely established) Col rhombic crysts; mp darkens above 200°; bp decomp rapidly at 230–35°; sl sol in w

(0.9% at 30°); sl sol in cold abs alc and appreciably in hot alc; nearly insol in eth, chlf and benz; very sol in concd nitric acid; gelatinized by action of concd sulfuric acid, going into soln after stirring

It was first prepd by Franchimont & Klobbie as described in Refs 1 & 2, but the yields were small

Laboratory investigation of prepn of DNDMGU was conducted during WWII at Picatinny Arsenal by Aaronson (Ref 4). He stirred for 1½ hour a mixt of 50g DMGU with 250g of 98% nitric acid, keeping the temp below 10°; after leaving the resulting soln overnight in an ice box, it was drowned in ca 1200g of ice-water, and removing the water by distillation at RT under reduced pressure (ca 2mm); the residue, consisting of crude DNDMGU was recrystallized from abs alc

Shriner (Ref 5) prepd it in 80–87% yield by nitrating DMGU with concd nitric acid in presence of acetic anhydride, while cooling the mixture to 5°

DNDMGU is an explosive ca 70% as powerful as TNT and ca 40% as brisant; it is more sensitive to impact than TNT, being comparable to PETN; vacuum stability at 100° – unsatisfactory. Attempts by Shriner to nitrate it to higher NO_2 content were unsuccessful (Refs 5 & 6)

Refs: 1) Beil **26**, 445 & (131) 2) A.P.N. Franchimont & E.A. Klobbie, Rec **7**, 251–53 (1888) 3) H. Blitz, Ber **41**, 1882(1908) 4) H. Aaronson, PATR **1312**(1943) 5) R.L. Shriner, OSRD **2054**(1943) 6) A.H. Blatt. OSRD **2014**(1944)

Dimethylglyoxime and Derivatives

Dimethylglyoxime or *Butane Dioxime*, $H_3C.C(:NOH).C(:NOH).CH_3$; mw 116.12, N 24.13%; col crysts, mp 240–42°; very sl sol in w; sol in alc & eth. It can be prepd by reacting methylethylketoxime with diacetylmonoxime in dil sulfuric acid at 40° (Ref 1). It is used in analytical chemistry, especially as a reagent for Ni

A small yield (ca 10%) of *Tetranitrobutane* could be obtd on treating it with fuming nitric acid

Dimethylglyoxime Diacetate, $H_3C.C(:NOOC.CH_3).C(:NOOC.CH_3).CH_3$; mw 200.19, N 13.99%; no props reported, except hydrolysis rate; was prepd by Milone (Ref 5) from dimethylglyoxime, Ac_2O & AcONa (Refs 5 & 6)

Treating it with fuming nitric acid gave a small amt of *Tetranitrobutane*

Dimethylglyoxime-bis(O-methylether), $H_3C.C(:NO.CH_3).C(:N.O.CH_3).CH_3$; mw 144.17, N 19.43%; crysts (from alc), pltlts (by condensation), mp 41–44°, bp 158.5° at 780mm press; obtd by reacting diacetyldioxime with dimethylsulfate in NaOH, and and by another method of Thilo (Refs 3 & 4) On treating it with fuming nitric acid it gave a small amt of white ppt which was not identified

Refs: 1) Beil **1**, 772, (398), [826] & {3105} 2) Beil **1**, not found (Diacetate) 3) Beil **1**, [830] 4) E. Thilo, Ber **62**, 870(1929) & CA **23**, 3902(1929) 5) M. Milone, Gazz **62**, 868(1932) & CA **27**, 706(1933) 6) ADL Synthesis HE's, 2nd Rept (1951), p59–60 7) CondChemDict (1961), p396-L

Dimethylguanidine and Derivatives

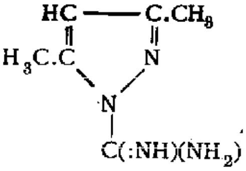
N,N-Dimethylguanidine, $(H_3C)_2N.C(:NH).NH_2$, mw 87.13, N 48.23%; ndls (from alc + eth), mp – produces gas at 144° & melts at 172°; forms many salts which decomp on heating (Ref 1)

N,N-Dimethyl-N^1-nitroguanidine, $(H_3C)_2N.C(:NH).NH(NO_2)$; mw 132.13, N 42.41%; ndls, mp 193.6–94.5°; sol in alc; sl sol in cold w & eth; decomp on heating in hot w; prepd by reacting Nitroguanidine with a 20% aq soln of dimethylamine at 75–80° (Refs 2, 3 & 4). No expl props reported

Refs: 1) Beil **4**, 75, (335), [574] & {146} 2) Beil **4**, [575] (Nitro) 3) T.L. Davis & S.B. Luce, JACS **49**, 2304(1927) 4) A.F. McKay, CanP 519448(1955) & CA **50**, 12107(1956)

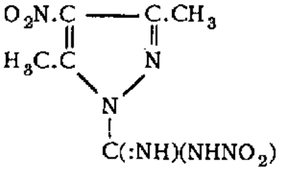
Dimethylguanylpyrazole and Derivatives

3,5-Dimethylguanylpyrazole,



mw 138.17, N 40.55%; known as the Nitrate salt, $C_6H_{10}N_4 + HNO_3$, crysts (from w) or ndls (from alc), mp 168°(dec); prepn & other props in Ref 1

3,5-Dimethyl-4-nitro-1-nitroguanylpyrazole,



mw 228.17, N 36.84%; crysts, mp 126–129°; sol in aq alc; can be prepd by nitrating the 3,5-dimethyl-1-nitroguanylpyrazole with fuming nitric acid at 60° for 1½ hrs (Ref 3). No expl props are reported

Refs: 1) Beil **23**, 76 (Parent) 2) Beil, not found (Dinitro) 3) F.L. Scott et al, JACS **75**, 1296(1953)

Dimethylhexadiene and Derivatives

2,5-Dimethylhexadiene-1,5;

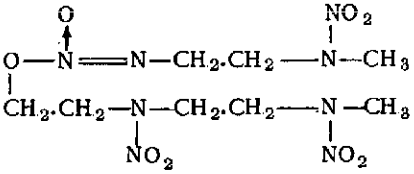
$H_2C{:}C(CH_3)CH_2CH_2C(CH_3){:}CH_2$, mw 110.19; col flammable liq; bp 114.3°; sp gr 0.740–0.760 at 25/25°; n_D 1.426–1.429 at 25°; 90% distln betw 114 & 123°; sol in hydrocarbons, insol in w. Prepn is given in Refs 1, 4 & 5

1,5-Dimethylhexadiene Diozonide,

mw 206.19, O 46.56%; yel viscous syrup, mp – very expl; decomp into formaldehyde; prepd from reaction of 2,5-dimethylhexadiene-1,5 & ozone (Refs 2 & 3)

Refs: 1) Beil **1**, 259, (122), [237] &{1010} (Dimethylhexadiene) 2) Beil **1**, 259(Dimethylhexadiene Dioxonide) 3)(?)Harris & (?) Türk, Ann **343**, 677(1905) 4) A.L. Henne et al, JACS **63**, 3475(1941) 5) CondChemDict (1961), 396–3 (2,5-Dimethylhexadiene-1,5)

1,12-Dimethyl-2,3,5,6,10.11-hexamethylene-7-oxa-8-nitroso-9-nitrilo-1,4,12- trinitrimine; *iso*-**N^1,N^4-Dimethyl-triethylene-tetranitramine** (Ref 2); or **2,10,13-Trinitro-7-oxa-2,5,6,10,13-pentaazatetradec-5-ene-6-oxide** (CA nomenclature),

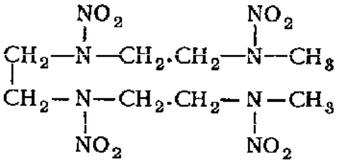


mw 354.28, N 31.63%, crysts, mp 88.5–90°, pH 6.2, expln temp 325°(ignited), at 135° – exploded in 10 mins; power 109.1% of TNT, sensitivity to impact >90cm (TNT = 48–50cm). It was known in Germany under code name BMT–110, one of the by-products in manuf of Hexogen (RDX). Blomquist (Ref 2) obtd it as one of two isomeric compds when N-methyl-ethylenedinitramine was alkylated, using ethylene dibromide (Ref 2). Its UV spectrum was detd by Jones & Thorn (Ref 3)

Refs: 1) Beil, not found 2) A. T. Blomquist, OSRD **4134**(1944), pp 8 & 69 3) R.N. Jones & G.D. Thorn, CanJRes **27B**, 832(1949) & CA **44**, 2848(1950)

NOTE: See following item

1,10-Dimethyl-2,3,5,6,8,9-hexamethylene-1,4,7,10-tetranitrimine or N^1-N^4-Dimethyl-triethylenetetranitramine (Ref 2),



mw 354.28, N 31.63%; crysts, mp 211.5–13°, pH 6.9, expln temp – no expln at 360°, volatility 0.1% (75°C International Test), power 90.4% of TNT, sensitivity to impact >90cm (TNT = 48–50cm). It was known in Germany under code name BMT–90, one of

the by-products in the manuf of RDX. Blomquist (Ref 2) obtd it as one of two isomers when N-methyl-ethylenedinitramine was alkylated, using ethylene dibromide (Ref 2)

Refs: 1) Beil, not found 2) A.T. Blomquist, OSRD **4134** (1944), pp 8 & 68

NOTE: See above item

Dimethylhexane and Derivatives

2,5-Dimethyl-hexane or *Diisobutyl*, $(H_3C)_2CH.CH_2.CH_2.CH(CH_3)_2$; mw 114.22; col liq, sp gr 0.694 at 20/4°, fr p minus 91.2, bp 109.1°. Prepn & other props are in Ref 1

2,5-Dimethylhexane-2,5-dihydroperoxide, $(H_3C)_2C(OOH).CH_2CH_2.C(OOH)(CH_3)_2$; mw 178.22, O 35.91%; fine pdr (from w or benz), non expl to friction; the 90% peroxide, used as high temp catalyst for polyester premix materials and silicone resins (Ref 5). Its prepn from $[(H_3C)_2C(OH)CH_2]_2$ & 75% hydrogen peroxide is given in Ref 4

2,5-Dinitro-2,5-dimethyl-hexane, $(H_3C)_2CH.CH(NO_2).CH(NO_2).CH(CH_3)_2$; mw 204.22, N 13.72%; crysts (from benz), mp 124–25°; sol in benz, alc & hot glac ac acid; sl sol in petr eth & eth; insol in alkalies; was prepd by treating tert-Nitroisobutyl with nitric acid (d 1.075), or by other methods (Ref 3)

x,x,x-Trinitro-2,5-dimethyl-hexane, $C_8H_{15}(NO_2)_3$; mw 249.22, N 16.86%; large colorless pltlts (from benz or petr eth), mp 91°; prepd by nitrating diisobutyl with fuming nitric acid (Ref 3). No expl props are reported. This compd was not found described in later Refs thru 1966

Refs: 1) Beil **1**, 162, (62), [126] & {483} (2,5-Dimethylhexane) 2) Beil **1**, not found (2,5-Dimethylhexane-2,5-dihydroperoxide) 3) Beil **1**, 163 (2,5-Dimethylhexane-2,5-dinitro) and (2,5-Dimethylhexane-x,x,x-trinitro) 4) R. Criegee & H. Dietrich, Ann **560**, 135 (1948) & CA **43**, 6190 (1949) 5) CondChemDict (1961), 396-L

17,17-Dimethyl-7,8,15,16,18,19-hexaoxadispiro-[5.2.5.5]-nonadecane. A CA name for compd described in Vol 2 of Encycl, p B144-R, as Acetone Compound of 1,1'-Bis(hydroperoxycyclohexyl)-peroxide

Dimethylhydantoin and Derivatives

5,5-Dimethylhydantoin or *Acetonylurea,* $OC.NH.CO.NH.C(CH_3)_2$; mw 128.13, N 21.87%; prisms or ndls (from w, alc or eth), mp 174–78°, bp – sublimes; v sol in w & hot alc and AcOH; sol in eth. It was first prepd by Franchimont & Klobbie (Refs 1 & 3). CondChemDict (Ref 6) lists two methods: a) From acetone, urea & Amm carbonate and b) From acetone, K cyanate & hydrocyanic acid. Thermochem props (Ref 5). Gives on nitration mono- and dinitro compds

1-Nitro-5,5-dimethylhydantoin or **Nitroacetonylurea,** $OC.NH.CO.N(NO_2).C(CH_3)_2$; mw 173.13, N 24.27; col ndls; mp 139–41°; bp (dec at 155° with evoln of gas); v sl sol in w & in benz; better sol in alc & in chlf; was first prepd by Franchimont & Klobbie. They evaporated on a steam bath a soln of 1 part of dimethylhydantoin with 5 parts of white nitric acid; the resulting dry residue was purified by dissolving in abs alc and precipitating by addn of benz

It is a stable and practically nonhygroscopic mild explosive of satisfactory thermal stability (Refs 2 & 3)

1,3-Dinitro-5,5-dimethylhydantoin or **N,N'-Dinitroacetonylurea,** $OC.N(NO_2).CO.N(NO_2).C(CH_3)_2$; mw 218.13, N 25.69%, OB to CO_2 minus 51.3%; col plates, mp 98.5–99.5°, bp (puffs off and catches fire, but does not expl even at 360°). Laboratory prepn, described in OSRD **86** (Ref 3), consisted of adding slowly with stirring 15g of dimethylhydantoin to a mixture of 30ml of acetic anhydride and 30ml of white nitric acid. After allowing to stand for 1 hr at 50° the soln was cooled and poured onto cracked ice. The pptd white residue was separated by filtration, washed with water, dried in air and recrystallized from a 3:1 by volume chlf/petr eth

It is an expl comparable in sensitivity with TNT; heat of combstn 604.2kcal/mole, heat of formation 73.1kcal/mole; thermal stability at 75° and 100° – unsatisfactory; not satisfactory from the point of view of hygroscopicity (Refs 3 & 4)

Refs: 1) Beil **24**, 289, (307) & [157] 2) Beil **24**, 293 & [158] 3) A.P.N. Franchimont & E.A. Klobbie, Rec **7**, 240(1888) 3a) R. Adams & C.S. Marvel OSRD **86**(1941), pp9, 26–7 & 56 4) A.H. Blatt, OSRD **2014** (1944) 5) P. Tavernier & M. Lamouroux, MP **37**, 200 & 206(1955) 6) CondChemDict (1961), 396-R

Dimethylhydrazine and Derivatives

uns-**Dimethylhydrazine (UDMH)**; (1,1-Dimethylhydrazine), code name **Dimazine**, $(H_3C)_2N.NH_2$; mw 60.10, N 46.62%; col flammable liq with ammonia-like odor; hygroscopic; sp gr 0.791 at 22°; fr p minus 58°; bp 63°; v sol in w, alc, eth & in hydrocarbons. Following methods of prepn are listed in Refs 1 & 11: a) reaction of dimethylamine (DMA) & chloramine; b) reaction of a DMA salt with Na nitrite, followed by the reduction of the product; c) catalytic oxidation of DMA and ammonia. Desseigne & Baron (Ref 10) prepd it by catalytic reduction of dimethylnitrosamine, $(CH_3)_2N.NH.NO$

Eutectic mixtures of UDMH and of methylhydrazine, $CH_3NH.NH_2$ (col hygroscopic liq, described in Beil 4, 546) for use in proplnts are listed in Ref 15

Dimazine has been used in jet & rocket proplnts; as stabilizer for org peroxide fuel additives and for other purposes (Refs 9, 15 & 16)

Accdg to Ref 14, Aerojet-General has completed a successful test firing of the rocket engine and storable fuels for the US Air Force Titan II Super ICBM. The storable proplnts (based on UDMH, high energy liquid fuel), will give the Titan near instant readiness. Both the proplnt combination and the rocket engine designed to use the proplnts are being developed by Aerojet under Air Force contract (Ref 14)

Explosibility of mixts of UDMH with diethylenetriamine, $(NH_2.C_2H_4)_2NH$ or JP-4 is discussed in Ref 13

Storage and handling of UDMH are discussed in Ref 8 and safety characteristics in Ref 13

US Military requirements for UDMH are discussed in Ref 18. They are as follows:

1) *Assay* – 98% by weight, minimum
2) *Moisture* – 0.3% by wt, maximum
3) *Particulate* – 10mg per liter, maximum

A sample of proplnt shall consist of not less than 600ml. When examined visually by transmitted light, it shall be a homogeneous, transparent liquid

Specification Tests for UDMH:

1) **Assay by Gas Chromatographic Method**

A brief general description of "gas chromatography" is given, (including several refs), in Vol 3 of Encycl, pp C293-R & C294-L

The description given in MIL-P-25604D is not comprehensive and cannot be used by persons not thoroughly familiar with gas chromatography. For this reason we are not including it here, but referring to the analytical procedure of Duigou described in Ref 12

2) **Moisture.** Drying of sample is conducted in an oven set at a temp of 70°(158°F)

3) **Particulate.** Mix the material, which arrived from the proplnt plant, thoroughly by shaking and pour immediately into a clean 500ml graduate. Use this 500ml of proplnt for particulate analysis in order to test for contamination. Use ASTM Designation D-2276-65T, Method A, with the following exceptions. The filter disc should be solvent resistant made from such materials as Millipore LSWP 04700 (Miltex-Teflon), Millipore URWP 04700 (Solvinert), or Gelman VF-6 (Fluoride-Metricel), plain, white 10±3 microns, 47mm diam, instead of the filter specified in Method D-2276-65T of ASTM. The drying oven temp shall be 70° instead of 90° specified by ASTM. Filtered isopropyl alc shall be used for rinsing the sample

bottle and filter holder, instead of petroleum ether specified by ASTM

Analytical procedures for detn of UDMH in mixtures with other substances are as follows:

Duigou (Ref 12) describes two microcolorimetric methods. In the first method, described on p326, p-dimethylaminobenzaldehyde is used as a reagent, but the method is considered not as reliable as the second method based on that of I.M. Kolthoff macrocolorimetric method described in JACS **46**, 2009 (1924)

Duigou Method (p328). Add to 2ml of aqueous UDMH soln, successively, 2ml of 1% aq K iodate soln and 1 ml of 5% "thiodène" soln, which serves as a better indicator than starch. After thorough agitation, add 1 ml of N/20 sulfuric acid, agitate again and, after 15 mins, place into a colorimeter and compare the blue coloration produced with standard solns

Note: Duigou does not give the formula of thiodène, but simply says that it is an iodometric reagent which dissolves rapidly and completely in cold w; it gives with iodine, liberated during reaction with K iodate, a beautiful blue coloration and may be considered as a better indicator than starch

Monard & Garrigues (Ref 11, pp359–64) describe the following method of bromometric titration:

Place in an Erlenmeyer of 250ml capacity (provided with a glass stopper) exactly 10ml of soln (contg ca 1.5g UDMH and 1.5ml sulfuric acid), add 20ml of 5N sulfuric acid and 2g K bromide. Run into Erln, with slight stirring, from a burette contg N/10 K bromate soln (prepd by dissolving 2.784g $KBrO_3$ and 10g KBr in 1 liter of distd w) until the light yellow coloration starts to be persistent. Note the number of mls added (say x) and then add rapidly until the total reading in burette is 4x + 3ml. Stopper the Erlenmeyer immediately, agitate it and allow to stand for 15 mins. Partly lift the stopper and add carefully (in order not to loose any liberated bromine) 10ml of soln contg 83g KI per liter, plus 50ml distd w. Stopper the flask, agitate it and titrate, after 2 mins, the liberated iodine with N/10 Na hyposulfite (Na thiosulfate) soln in presence of starch indicator until disappearance of blue coloration

$$\%UDMH = \frac{60x(N-a)x1000x100}{8x10000x10xp} = \frac{7.5(N-a)}{p}$$

where: N = total number of mls of K bromate used
a = number of mls of thiosulfate
p = grams of UDMH sample used

The analytical procedures described in Ref 17 are as follows:

4) **Assay.** Using a sample of 0.50–0.60g in a sealed glass ampoule, det the content of UDMH by titration with std 0.1M K iodate soln [prepd by dissolving 21.402g of K iodate (dried at 180°C) in distilled water and diluting to 1000ml in a volumetric flask]. The potentiometric procedure requires a calomel-platinum system and the plotting of mls vs emf to locate the point of greatest inflection. The temp thruout the titration should be maintained in the range −5 to +10°, and the titration should be completed within a total time of 5 min. At the end point the soln will be lt yel and the steep potential increase will be in the range 0.67 to 0.70 volts:

$$UMDH, \% = \frac{12.02AM}{W}$$

where: A = std 0.1M iodate soln used in ml
M = molarity of the iodate soln, and
W = wt of sample in grams

5) **Melting Point.** Use a capillary mp tube (1.5 to 2.00mm by 90mm) filled by means of a small hypodermic syringe and a stainless steel needle (20 gauge) and avoid getting any sample on the upper part of the capillary. Seal the tube ca 1 cm from the open end, using a very small hot flame. Place acetone in an unsilvered Dewar flask to a depth of ca 100mm and add dry ice until the temp is −62 to −64°C. Freeze the sample attached to a low-temp ASTM No. 6C thermometer (−80° to +20° in 1°C divisions, 76mm immersion) in an acetone-dry ice mixt, and immerse it in the Dewar flask to 76mm. Allow the temp to rise at a rate not greater than 1.0°C per min, and record the temp at which the sample becomes completely liq. Cali-

brate the thermometer by taking the mp of pure chlf (–63.5) and apply a suitable correction for any difference

6) **Distillation Range.** The vapor temps are recorded when 10ml & 90ml have been collected in the receiving graduate on distilling a 100-ml sample in a special assembly described in the Specification (Ref 17)

7) **Water Content.** Max water content may be calcd from the 90-ml distillation temp as follows:

$$\text{Wt\% } H_2O = 0.14[(\text{corrected 90-ml temp in } ^\circ F) - (146^\circ F)]$$

Since the max water content allowed is 0.3%, it can be calculated that the temp at the 90-ml recovery point cannot exceed 148.1°F if the water-content limitation is met

Alternatively, it may be detd spectrophotometrically

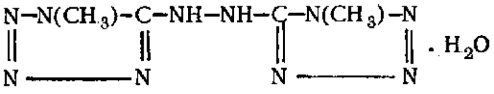
sym-Dimethylhydrazine (1,2-Dimethylhydrazine, N,N'-Dimethylhydrazine or Hydrazomethane), $H_3C.NH.NH.CH_3$, mw 60.10, N 46.62%; hygroscopic liq, sp gr 0.827 at 20/4°, fr p – not listed; bp 81° at 747mm; miscible with w, alc & eth. Prepn is in Beil (Ref 1)

It forms salts which are crystalline materials, some of which are unstable on heating (Ref 2)

N-Nitroso-N,N'-dimethylhydrazine,
$H_3C.NH.N(NO).CH_3$; mw 73.10, N 57.49%; lt yel oil, bp 56°, d 1.09; miscible with water & common org solvs; vol on heating at high temp, decomp gradually in light, deflgr on heating in a flame; was obtd by reaction of N-Nitroso-N-methylhydrazine & dimethylsulfate in alk soln at 35–40°, or from hydrazomethane & sodium nitrite in acet ac soln [Refs 2, p(561) & Ref 3]. See also Ref 4

Refs: 1) Beil **4**, 547, (560), [958] & {1726} (UDMH) 2) Beil **4**, 547, (560), [958] & {1727} (Sym DMH) 3) J. Thiele, Ann **376**, 257(1910) & CA **5**, 457(1911) 4) J.P. Picard & J.L. Boivin, CanJChem **29**, 223–27 (1951) & CA **45**, 9469(1951) (1,2-Dimethylhydrazine, its salts and nitrated derivatives) 5) L.F. Audrieth & B.A. Ogg, "The Chemistry of Hydrazine", Wiley, NY (1951) (Not listed) 6) C.C. Clark, "Hydrazine", MathiesonChemCorp, Baltimore, Md (1953) (Dimethylhydrazine is not listed) 7) J.C. Melville, "Determination of Water in Unsymmetrical Dimethyl Hydrazine (UDMH) by Capacitance Measurements", Wright Air Development Center, Technical Note 58–9, Dec 1957 8) Westvaco Chlor-Alkali Division Food Machinery and Chemical Corp, Bulletin: "Storage and Handling of Dimazine", 3rd ed, NY (1957) 9) W.G. Strunk, Missiles & Rockets, 2, 116–20 (Sept 1957) & CA **52**, 8500(1958) ("Dimazine Comes of Age as Rocket Fuel") 10) G. Desseigne & C. Baron, MP **40**, 343–50(1958) & MP **41**, 299–316(1959) (Prepn of DMH) 11) C. Monard & C. Garrigues, MP **41**, 359–68 (1959) (Analytical procedures for DMH and derivatives) 12) Y. leDuigou, MP **41**, 326–30(1959) (Analytical procedure by colorimetric method) 13) J.A. Herickes et al, USBurMinesReptInvest RI **5635**(1960) & CA **54**, 21763(1960) (Safety characteristics of UDMH) 14) Anon, "Aerojet-General Has Completed a Successful Test Firing", C&EN **38**, p17, June 27, 1960 15) D. Horvitz, USP 2954283(1960) & CA **55**, 6868 (1961) (DMH–MH eutectic mixtures) 16) CondChemDict (1961), 396-R 17) StdMethodsChemAnalysis **2B**, 1316–17(1963) 18) US Military Specification MIL-P-25604D (1969)

1,1'-Dimethyl-5,5'-hydrazo-1H-tetrazole or **5,5'-Hydrazobis[1-methyl-1,2,3,4-tetrazole]**



$$\begin{array}{l}N{-}N(CH_3){-}C{-}NH{-}NH{-}C{-}N(CH_3){-}N\\ \| \qquad\qquad\quad \| \qquad\qquad\qquad\quad \| \qquad\qquad\quad \| \;\cdot H_2O\\ N{-\!-\!-\!-}N \qquad\qquad\quad\;\; N{-\!-\!-\!-}N\end{array}$$

mw 196.19, N 71.40%; crysts, mp – decomp at 158° with evolution of gas; was obtd by reduction of 1,1'-Dimethyl-5,5'-azotetrazole, orn-yel crysts, mp 182°(dec). Its expl props were not investigated

Refs: 1) Beil, not found 2) R. Stollé, et al, JPraktChem **134**, 282–83 & 287–88(1932) & CA **26**, 5565(1932)

Dimethylhydrogenphosphite, $(CH_3.O)_2PHO$; mw 110.06; col liq with mild but toxic odor; sp gr 1.200 at 20°; bp 72–73° at 25mm; flash p 205°F; n_D 1.400 at 25°; sol in w and miscible with many org solvents. It is hazardous to handle and personnel sampling or testing this material should be adequately protected against contact with the liquid and inhalation of the fumes. Sampling and testing should be performed as rapidly as possible because the material decomposes on contact with atm moisture

Dimethylhydrogenphosphite is intended for use as an intermediate in chemical agent production and also as an adhesive & lubricant additive. US Armed Forces requirements are listed in Ref 2

Refs: 1) Beil **1**, 285, (142) & {1203}
2) Spec MIL-D-10396C (1969) 3) CondChemDict (1961), 398-L

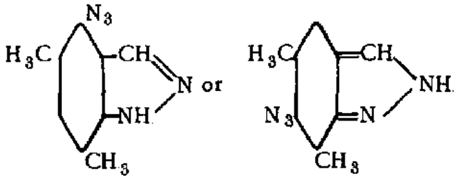
1,4-Di(methylimino)-p-quinone. See Benzoquinone-1,4-di(methylimine) in Vol 2, pB83-R

Dimethyliminotetrazoles, such as 1,3-Dimethyl-5-iminotetrazole and 1,4-Dimethyl-5-iminotetrazole are described under Dialkyliminotetrazoles in this Vol

Dimethylindazole and Derivatives

Dimethylindazole or *Dimethylbenzopyrazole*, $C_9H_{10}N_2$, mw 146.19, N 19.16%. Four isomers are described in Beil (Ref 1)

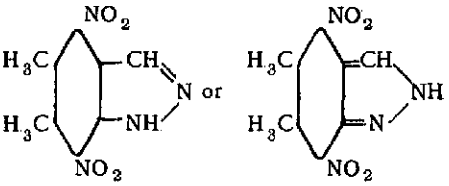
4(or 6)-Azido-5,7-dimethylindazole,



mw 187.20, N 37.41%; yel ndls (from benz), mp 134–37°; sol in alc & benz; insol in petr eth; was prepd by diazotizing 2,4-diaminomesitylene with nitrosyl sulfuric acid in concd sulfuric acid and treating the diazonium salt with NaN_3 in cold water (Refs 2 & 5)

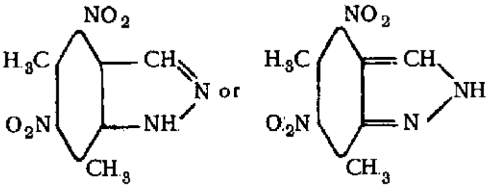
Mononitrodimethylindazole, $C_9H_9N_3O_2$, mw 191.19, N 21.98%. Two isomers are known: *4-Nitro-5,6-dimethylindazole*, pltlts (from petr eth + benz), mp 204°; and *7-Nitro-5,6-dimethylindazole*, lt-yel ndls, mp 180.5–181.5°. Prepn & other props are given in the Refs (Refs 3 & 4)

Dinitrodimethylindazole, $C_9H_8N_4O_4$, mw 236.19, N 23.72%. Two isomers are known: *4,7-Dinitro-5,6-dimethylindazole,*



greenish ndls (from anisole), mp 221–22°; sol in boiling alc, anisole & w contg acetic acid; soln in alkali is orn; prepd by diazotizing 3,6-dinitro-2,4,5-trimethylaniline in glac acet ac & sulfuric acid and treating the diazonium salt with dil boiling sulfuric acid soln (Refs 3 & 4)

4,6-Dinitro-5,7-dimethylindazole,



yel ndls (from alc), mp 247°; prepd by treating 3,5-dinitro-2,4,6-trimethylaniline in concd sulfuric acid with nitrosylsulfate and heating with a dil soln of urea; or by diazotizing in glac acet ac and concentrating the soln by boiling (Refs 3 & 4)

No expl props of the dinitro derivs are reported

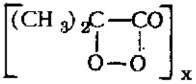
Refs: 1) Beil **23**, 141, 157 & [155, 157] (Dimethylindazole) 2) Beil **23**, [166]
3) Beil **23**, 157, 158 4) E. Noelting, Ber **37**, 2594–97 (1904) 5) G.T. Morgan & G.R. Davies, JChemSoc **123**, 232 (1923) & CA **17**, 1632 (1923)

Dimethylketene and Derivatives

Dimethylketene, $(CH_3)_2C{:}CO$, mw 70.09;

yel liq, fr p minus 97.5°, bp 34° at 750mm; sol in eth; decompd by w or alc (Ref 1)

Dimethylketene Peroxide,



$$\left[\begin{array}{l}(CH_3)_2C-CO\\ \qquad\quad\;\; |\qquad |\\ \qquad\quad\;\; O-O\end{array}\right]_x$$

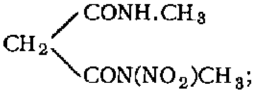
mw 102.09, O 47.02%; jelly (from ethyl acetate) or wh powd (from ether), which when dry is exceedingly expl, detonates with great violence when touched, often even spontaneously; obtd by autoxidation of dimethylketene at low temp (Refs 2 & 3)

Refs: 1) Beil **1**, 731, (380), [789] & {2981} 2) Beil 1, [789] (Dimethylketene Peroxide) 3) H. Staudinger et al, Ber **58**, 1079(1925) 4) Tobolsky & Mesrobian (1954), 178

Dimethylmalonamide and Derivatives

N,N'-Dimethylmalonamide, $CH_2(CO.NH.CH_3)_2$; mw 130.15, N 21.53%; ndls (from benz), mp 101°; sl sol in alc, chlf, acet ac, acet & w; sol in benz; v sl sol in CCl_4 & CS_2; prepd by reaction of CO with methylamine in ether (Ref 1)

N-Nitro-N,N'-Dimethylmalonamide,



$$CH_2\begin{array}{l}\diagup CONH.CH_3\\ \diagdown CON(NO_2)CH_3\end{array};$$

mw 175.15, N 23.99%; ndls (from w), mp 156°; readily sol in hot w, alc, eth & benz; can be prepd by warming N,N'-Dimethylisonitrosomalonamide with concd nitric acid, or by warming N,N'-Dimethyl-C-nitro-barbituric acid with 2 parts KOH in w. It forms crystalline salts with Ba, Cu & K. No expl props of the compd were investigated

N,N'-Dinitro-N,N'-dimethylmalonamide, $CH_2[CO.N(NO_2).CH_3]_2$, mw 220.15, N 25.45%; prisms (from ethyl acetate), mp 150°; sol in eth; sl sol in eth acet; v sl sol in chlf, CS_2 & petr eth; was prepd by nitrating N,N'-dimethylmalonamide with nitric acid in the cold (Refs 3 & 4).

No expl props are reported

Refs: 1) Beil **4**, 62, [565] & {132} 2) Beil **4**, 62 (Mononitro deriv) 3) Beil **4**, 86 (Dinitro deriv) 4) A.P. Franchimont, Rec **4**, 200(1885)

Dimethylmethanediamine and Derivatives

N,N'-Dimethyl-methanediamine, $(H_3C)HN.CH_2.NH(CH_3)$; mw 74.13, N 37.80%; may be considered as the parent compd of its dinitro deriv, although not used to prep it:

N,N'-Dinitro-N,N'-dimethylmethanediamine or **2,4-Dinitro-2,4-diazapentane,** $(H_3C)(O_2N)N.CH_2.N(NO_2)(CH_3)$; mw 164.13, N 34.14%; crysts (from chlf-hexane), mp 49–51°; was prepd by treating paraformaldehyde in 90% sulfuric acid at −2 to −6° portionwise with methylnitramine, the mixt stirred, poured on a large amt of ice, and the solid combined with product from ether extraction (Ref 3). No expl props are reported

Refs: 1) Beil, not found (DMMDA) 2) Beil, not found (Dinitro DMMDA) 3) L. Goodman, JACS **75**, 3020(1953) & CA **49**, 4630(1955)

Dimethylmethyleneaminoguanidine and Derivatives

Dimethylmethyleneaminoguanidine or *Isopropylideneaminoguanidine*, $H_2N.C(:NH).NH.N:C(CH_3)_2$; mw 114.14, N 49.09%; may be considered as the parent compd of the nitrated deriv, although not used to prep it:

N'-Nitrodimethylmethyleneaminoguanidine, $(O_2N)HN.C(:NH).NH.N:C(CH_3)_2$; mw 159.15, N 44.01%, OB to CO_2 minus 105.6%; wh ndls; mp – detonates when brought in contact with a flame; diff sol in w; can be prepd by treating nitraminoguanidine, $(O_2N)HN.C(:NH).NH.NH_2$, with acetone (Refs 1, 2 & 3)

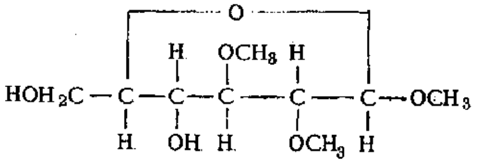
A slightly stronger explosive is obtd by treating nitroaminoguanidine with aqueous formaldehyde

N'-Nitromethyleneaminoguanidine, $(O_2N)HN.C(:NH).NH.N:CH_2$, mw 131.10, N 53.43%, OB to CO_2 minus 30.5%; ndls, mp – explodes (Refs 1, 2 & 3)

Refs: 1) Beil **3**, [101] 2) R. Phillips & J.W. Williams, JACS **50**, 2465–70(1928) 3) E.R. Riegel & K.W. Buchwald, JACS **51**, 492(1929)

Dimethylmethylgalactoside and Derivatives

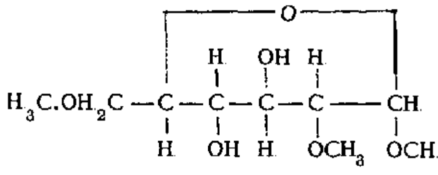
2,3-Dimethyl-α-methylgalactoside,



mw 267.24; clear syrup, n_D 1.4720 at 15°; obtd by graded hydrolysis of 4,6-benzylidene-2,3-dimethyl-α-methylgalactoside with dil HCl (Ref 3)

2,3-Dimethyl-α-methylgalactoside-4,6-dinitrate, $C_9H_{16}N_2O_{10}$; mw 312.24, N 8.97%; crysts (from alc), mp 88–90°; can be prepd by nitrating the parent compd with fuming nitric acid in chlf soln, or by direct action of fuming nitric acid on 4,6-benzylidene-2,3-dimethyl-α-methylgalactoside in chlf, but the yield is reduced to 67% (Ref 3)

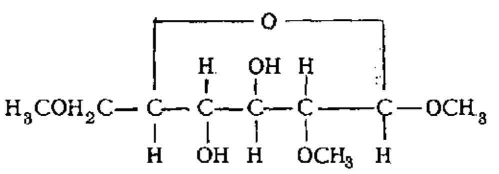
2,6-Dimethyl-β-methylgalactoside,



mw 267.24; ndls (from eth), mp 45–46.5°; prepd by hydrolyzing 3,4-acetone-2-methyl-β-methylgalactoside (Ref 4)

2,6-Dimethyl-β-methylgalactoside-3,4-dinitrate, $C_9H_{16}N_2O_{10}$; mw 312.24, N 8.97%; ndls (from MeOH), mp 88–88.5°; prepd by nitrating the parent compd with 30% fuming nitric acid in dry chlf for 10 mins (Ref 4)

2,6-Dimethyl-α-methylgalactoside-3,4-dinitrate,



mw 312.24, N 8.97%; stout ndls (from petr eth), mp 50–51°; obtd in nearly quantitative yield on treating 2,6-dimethyl-3,4-isopropylidene-α-methylgalactoside for 30 mins at 0° with 30% fuming nitric acid in dry chlf (Ref 5)

3,4-Dimethyl-β-methylgalactoside, $C_9H_{18}O_6$; mw 267.24; small ndls (from chlf), mp 102–03°; was obtd when the dinitrate deriv (below) was refluxed in alc with 30% NaOH saturated with hydrogen sulfide for 30 mins, removal of the alc, addn of K_2CO_3 and extraction with chlf (Ref 6)

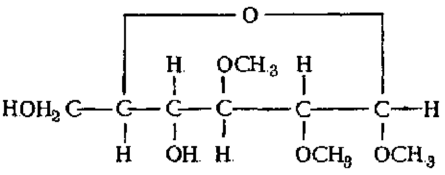
3,4-Dimethyl-β-methylgalactoside-2,6-dinitrate, $C_9H_{16}N_2O_{10}$; mw 312.24, N 8.97%; plates (from aq MeOH), mp 75–76°; obtd by repeated methylation of β-methylgalactoside with MeI & Ag_2O in dry acetone at 45° (Ref 6)

No expl props of the dinitro derivs are reported

Refs: 1) Beil **31**, 180 (2,3-DMMG) 2) Beil, not found (DMMGDN) 3) G.R. Robertson & R.A. Lamb, JChemSoc **1934**, 1321 & CA **29**, 129 (1935) 4) J.W.H. Oldham & D.J. Bell, JACS **60**, 323 (1938) & CA **32**, 2513 (1938) 5) D.J. Bell & S. Williamson, JCS **1938**, 1200 & CA **32**, 8372 (1938) 6) J.S.D. Bacon & D.J. Bell, JCS **1939**, 1870 & CA **34**, 1620 (1940)

Dimethylmethylglucoside and Derivatives

2,3-Dimethyl-β-methylglucoside,



mw 222.23; col syrup, n_D 1.4690; obtd from benzylidene-β-methylglucoside by methylation and hydrolysis using N/20 HCl in acetone (Ref 4). See also Refs 7 & 8

Dimethylmethylglucoside-6-nitrate, $C_9H_{17}NO_8$; mw 267.24, N 5.24%; props & methods of prepn of two derivs are given in Refs 4 & 9

2,3-Dimethylmethylglucoside-4,6-Dinitrate, $C_9H_{16}N_2O_{10}$; mw 312.24, N 8.97%; large prisms (from abs alc), mp 98–99°; insol in petr eth, v sl sol in eth & cold alc; obtd by nitrating the parent compd with 30% nitric acid in chlf (Refs 4, 5 & 6). The **2,4-Dimethyl-β-methylglucoside-3,6-dinitrate** is found in Ref 9

Refs: 1) Beil, not found (DMMG) 2) Beil, not found (DMMGN) 3) Beil, not found (DMMGDN) 4) J.W.H. Oldham & J.K. Rutherford, JACS **54**, 366(1932) & CA **26**, 968(1932) 5) J.C. Irvine & J.K. Rutherford, JACS **54**, 1491(1932) & CA **26**, 2710 (1932) (DMMGDN) 6) D.J. Bell, JChemSoc **1936**, 186 & CA **30**, 2926(1936) (DMMGDN) 7) D.J. Bell & R.L.M. Synge, JCS **1937**, 1711 & CA **32**, 504(1938) (Prepn 4,6-deriv) 8) Ibid, JChemSoc **1938**, 833–36 & CA **32**, 6627 (Prepn 2,6- deriv) 9) J. Dewar & G. Fort, JCS **1944**, 492ff & CA **39**, 912–14(1945) (Nitrate and Dinitrate of DMMG)

Dimethylnaphthalene and Derivatives

2,6-Dimethylnaphthalene, $(CH_3)_2C_{10}H_6$; mw 156.22, wh lflts, sp gr 1.142 at 0°/4°, mp 110–11°, bp 261–62° at 767mm. Prepn & other props in Ref 1

1,4,5 (?)-Trinitro-2,6-dimethylnaphthalene, $(CH_3)_2C_{10}H_5(NO_2)_3$; mw 291.22, N 14.93%; fine lt-yel ndls (from AcOH), mp 243°; obtd with dinitro derivs when 1-nitro-2,6-dimethylnaphthalene in glac acet ac is nitrated with concd nitric acid (d 1.51) (Refs 1 & 2). No expl props are reported

Refs: 1) Beil **5**, 570, (268), [468] & {1651} 2) Beil **5**, [469] (TNDMN) 3) F. Mayer & E. Alken, Ber **55B**, 2282(1922) & CA **17**, 758(1923)

Dimethylnitramine. See under Dimethylamine and Derivatives in this Vol

2,4-Di(methylnitramino)-3,5-dinitrotoluene. See 2,4-Bis(methylnitramino)-3,5-dinitrotoluene in Vol 2, p B150-L

Di(methylnitraminomethyl) -methylamine. See Bis(methylnitraminomethyl)-methylamine in Vol 2, pp B149-R & B150-L

4,4'-Di(methylnitramino)-3,5,3',5'-tetranitrobenzophenone. See 4,4'-Bis(methylnitramino)-3,5,3',5'-tetranitrobenzophenone in Vol 2, p B149-L

4,4'-Di(methylnitramino)-3,5,3',5'-tetranitrodiphenylmethane. See 4,4'-Bis(methylnitramino)-3,5,3',5'-tetranitrodiphenylmethane in Vol 2, p B149-R

3,5-Di(methylnitramino)-2,4,6-trinitrotoluene. See 3,5-Bis(methylnitramino)-2,4,6-trinitrotoluene in Vol 2, p B150-L

Di(methyl-nitropyrazolylyl) -triazene. See 1,3-Bis(3-methyl-4-nitro-5-pyrazolyl)-triazene in Vol 2, p B150-R

Dimethylnitrosamine. See under Dimethylamine and Derivatives

2,7-Dimethyl-3,5-octadiyn-2,7-dihydroperoxide. See under Diacetylenic Dihydroperoxides in this Vol

Di(methylphenylamino)-propane. See 1,3-Bis(2'-methylphenylamino)-propane in Vol 2, p B151-L

Di(methyltetrazolyl)-diazene. See Bis(1-methyl-1,2,3,4-tetrazolyl-5)-diazene in Vol 2, p B151-L

Di(methyltriazolyl)-formamidine. See N,N'-Bis(5-methyl-s-triazol-3-yl)-formamidine in Vol 2, p B151-L

Di(methyltrinitrophenyl) -amine. See Bis(3-methyl-2,4,6-trinitrophenyl)-amine in Vol 2, p B151-R

Di(methylol)-amine and Derivatives

Di(methylol)-amine; N,N'-Bis(β-hydroxymethyl)-amine, or *Di(methanol)-amine*, $HN(CH_2OH)_2$; mw 77.08, N 18.17%; may be considered as the parent compd of its nitrated deriv, although not used to prep it:

Di(methylol)-nitramine Dinitrate or **Nitroimino-dimethanol Dinitrate,** $(O_2N)N(CH_2ONO_2)_2$; mw 212.08, N 16.41%; a precursor product obtd during formation of 1,5-endomethylene-3,7-dinitro-1,3,5,7-tetrazacyclooctane on neutralization with NH_4OH of the filtrate from the prepn of RDX by the method of Hale (CA **20**, 40);

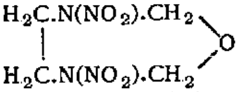
mp 45–49°, yield 35% from diacetoxydimethylnitramine (Refs 2 & 3)

Refs: 1) Beil, not found 2) W.J. Chute et al, CanJRes **27B**, 218(1949) & CA **43**, 8354(1949) 3) E. Aristoff et al, CanJRes **27B**, 520(1949) & CA **43**, 9075(1949)

Di(methylolamino)-ethane and Derivatives

N,N'-Dimethylol-1,2-diamino-ethane, $HOH_2C.NH.CH_2.CH_2.NH.CH_2OH$; mw 120.15, N 23.32%; may be considered as the parent compd of its nitro derivs, although not used to prep them:

N,N'-Dimethylol-1,2-dinitramino-ethane or **[Ethylenebis(nitroimino)]-dimethanol,** $HOH_2C.N(NO_2).CH_2.CH_2.N(NO_2).CH_2OH$, mw 210.15, N 22.66%; compd not isolated, but formed when 1,2-dinitraminoethane, $(CH_2NHNO_2)_2$, was heated in 37% formalin (HCHO) to 60° (Ref 3)

N,N'-Dimethylol-1,4-dinitramino-ethane Dinitrate or **Ethylenebis(nitroimino)-dimethanol Dinitrate,** $(O_2NO)H_2C.N(NO_2).CH_2.CH_2.N(NO_2).CH_2(ONO_2)$; mw 328.17, N 27.37%; crysts, mp 112–17°(dec); was obtd when 3,6-dinitro-1-oxa-3,6-diazacycloheptane, 

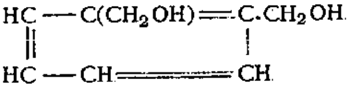
was added gradually to 98% nitric acid at –15°; and after 10 mins the mixt was added dropwise to anhyd ether at –40° (Ref 4). This compd also prepd by Simkins & Wright (Ref 5) and by Majer & Denkstein (Ref 6) is reported to be very unstable

Refs: 1) Beil, not found [Di(methylolamino)-ethane] 2) Beil, not found (Dinitramino compd) 3) G.S. Myers & G.F. Wright, CanJRes **27B**, 490(1949) & CA **43**, 9074 (1949) 4) D. Woodcock, JChemSoc **1949**, 1638 & CA **44**, 1411(1950) 5) R.J.J. Simkins & G.F. Wright, JACS **77**, 3157 & CA **50**, 3469(1956) 6) J. Majer & J. Denkstein, CollCzeckChemCommun **31**(6), 2547(1966) (Ger) & CA **65**, 7042(1966)

Dimethylolbenzene and Derivatives

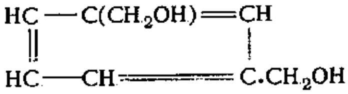
Dimethylolbenzene or **Bis(hydroxymethyl)-benzene,** $C_8H_{10}O_2$, mw 138.16, O 23.16%. Three isomers are known:

o- or 1,2-Dimethylolbenzene,



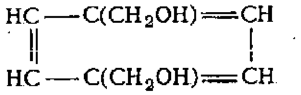
crysts (from petr eth) or tablets (from eth), mp 64–66.5° (Ref 1)

m- or 1,3-Dimethylolbenzene,



crysts (from benz) having a bitter taste, mp 46–47°, bp 154–59° at 13mm, d 1.161 at 18°; sol in eth; v sl sol in w (Ref 2)

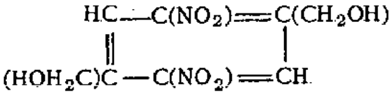
p- or 1,4-Dimethylolbenzene,



ndls (from w) having a bitter taste, mp 114–16°; v sol in w, alc & eth (Ref 3)

Other props & methods of prepn are given in the Refs

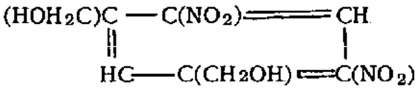
3,6-Dinitro-1,4-dimethylolbenzene or **1,4-Dinitro-2,5-dimethylolbenzene,**



mw 228.16, N 12.28%

crysts; mp – not reported; bp – not reported; can be prepd from 2,5-dimethylbenzene(p-xylene) by the following series of reactions: chlorination to dichloro-p-xylene, nitration to dinitro-p-xylene-dichloride and hydrolysis to replace chlorines by hydroxyls. It is an expl compd (Refs 5 & 6)

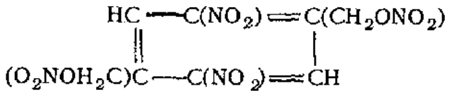
4,6-Dinitro-1,3-dimethylolbenzene or **1,3-Dinitro-4,6-dimethylolbenzene,**



mw 228.16, N 12.28%; crysts, mp – not reported, bp – not reported; can be prepd from 4,6-dimethylbenzene(m-xylene) by the following series of reactions: chlorination to dichloro-m-xylene, nitration to dinitro-m-xylene-dichloride and hydrolysis to replace

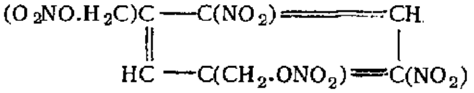
chlorine atoms by hydroxyl groups. It is an expl compd (Refs 5 & 6)

1,4-Dinitro-2,5-dimethylolbenzene Dinitrate,



mw 318.16, N 17.61%, OB to CO_2 minus 45.3%; crysts, mp 76°; bp – explodes at higher temp. It can be prepd by nitrating 1,4-Dinitro-2,5-dimethylolbenzene. It is an expl slightly more powerful than Tetryl but more sensitive to impact. Its thermal stability is unsatisfactory (Refs 5 & 6)

1,3-Dinitro-4,6-dimethylolbenzene Dinitrate,



mw 318.16, N 17.61%, OB to CO_2 minus 45.3%; crysts, mp 114°; bp – explodes at higher temps. It can be prepd by nitrating 1,3-Dinitro-4,6-dimethylolbenzene. It is an explosive comparable in power and sensitivity to Tetryl but of unsatisfactory thermal stability (Refs 5 & 6)

Refs: 1) Beil **6**, 910 & {4587} 2) Beil **6**. 914, (446) & {4600} 3) Beil **6**, 919, (446), [891] & {4608} 4) Beil **6**, not found (Dinitro- and Dinitrodinitrate derivs) 5) C.M. Stine, USP 1309551 & 1311926(1919) & CA **13**, 2282 & 2598(1919) 6) C.M. Stine, USP 1370067(1921) & CA **15**, 1622(1921)

Dimethylolbutanol or Bis(hydroxymethyl)-butanol and Derivatives

2,2-Dimethylol-1-butanol or *Trimethylolethylmethane*,

$CH_2(OH).C(CH_2OH)_2.CH_2.CH_3$; mw 134.17; crysts (from eth + acet), mp 58.8–59°; was prepd by condensation of butyraldehyde with formaldehyde in w contg $Ca(OH)_2$ (Refs 1 & 4)

3,3-Dimethylol-2-butanol,

$CH_3.CH(OH).C(CH_2OH)_2.CH_3$; mw 134.17; crysts, mp 59 to 66°; bp 134–37° at 3–4mm Hg; can be prepd by the oxidation of 3,3-dimethyl-2-butanol. It was nitrated to dinitrate & trinitrate derivs. The dinitrate was an unstable & volatile oil and for this reason not of interest for military applications (Ref 3, p3). The Trinitrate is described below

2,2-Dimethylol-1-butanol Trinitrate,

$CH_2(ONO_2).C(CH_2.ONO_2)_2.CH_2.CH_3$; mw 269.17, N 15.61%; OB to CO_2 minus 50.5%; crysts (from acet + alc), mp 51.2°; prepd by nitrating the 2,2-dimethylol parent compd with HNO_3–H_2SO_4 mixed acid (Ref 2)

3,3-Dimethyl-2-butanol Trinitrate,

$CH_3.CH(ONO_2).C(CH_2.ONO_2)_2.CH_3$, mw 269.17, N 15.61%; (crude product obtd as an oil by Warren had N 14.1%), OB to CO_2 minus 50.5%. The oily product puffed off at 245° (after 5 seconds heating), and it did not explode on impact of 100cm fall of a 2kg wt using USBurMines machine; thermal stability by 82.2°C KI Test, 1 min; volatility at 27° in 48 hrs, 0.25% and hygroscopicity at 30° & 90% RH, 4.1%. It was prepd at PicArsn Lab by Warren on adding in small portions and with agitation 25g of pulverized dimethylolbutanol to cooled to 0° mixed acid consist of nitric acid 25.3, sulfuric acid 59.6 & water 15.1% in the ratio of 1.5 parts of nitric acid to 1 part of dimethylbutanol; time of nitration after addn of butanol was ca 1.5 hrs. After nitration the batch was poured on cracked ice with stirring and the trinitrate dissolved in aqueous mixed acid extracted with factory ether; the ethereal soln was neutralized with Na bicarbonate soln, washed with w and the separated oil decanted and dried overnight over anhyd Na sulfate, followed by evaporation of residual ether in a vacuum desiccator over Ca chloride

Refs: 1) Beil **1**, {2349} 2) Beil **1**, {2350} 3) K.S. Warren, "Study Nitrates of Polyhydric Alcohols as Explosives", PATR **1103**(1941) 4) L. Médard & M. Thomas, MP **35**, 158(1953) (2,2-Dimethylol-1-butanol and its Trinitrate) 5) No later Refs found thru 1966

Dimethylolbutanone and Derivatives

3,3-Dimethylol-2-butanone; Dimethylol-

methylethylketone or *β-Methyl-β-hydroxymethyl-γ-ketobutanol*, $CH_3.CO.C(CH_2OH)_2.CH_3$, mw 132.16; crysts, mp 54–59°, bp 121–125° at 9–14mm; can be prepd by condensation of methylethyl ketone with 2 moles of formaldehyde in presence of an alkaline catalyst, such as 2N NaOH (Ref 3)

It gives on nitration an explosive dinitrate

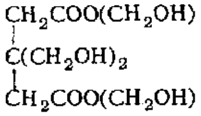
3,3-Dimethylol-2-butanone Dinitrate, $CH_3.CO.C(CH_2.ONO_2)_2.CH_3$, mw 222.16, N 12.61% (theory) (12.4% found in oily product of Warren); OB to CO_2 minus 72.0%. The oily product puffed off at 187° after heating for 5 secs; it did not explode by impact of 2kg wt at 100cm; hygroscopicity at 30° & 90% RH 0.40%; volatility at 27° in 48 hrs 0.34%; thermal stability at 82.2° KI test 1 min. Warren prepd it at PicArsn Lab by adding in small portions and with agitation, pulverized dimethylolbutanone to cooled to ca 5° mixed acid consisting of nitric acid 25.3, sulfuric acid 59.6 & water 15.1% in the ratio of 1.5 parts nitric acid per 1 part of butanone; time of nitration 1 hour. Separation of crude product from acid and further purification was the same as described under 3,3-Dimethylol-2-butanol Trinitrate

Refs: 1) Beil, not found (Dimethylolbutanone) 2) Beil, not found (Dinitrate) 3) Sir G. Morgan et al, Chem & Ind **57**, 887(1938) 4) K.S. Warren, PATR **1103**(1941)

Dimethylol-dimethylmethane. See Dimethylolpropane or Pentaglycol in this Vol

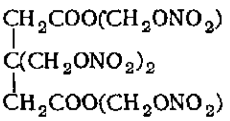
Dimethylol-dimethylol-glutarate and Derivatives

(2,2-Dimethylol)-dimethylol Glutarate,



$$\begin{array}{l}CH_2COO(CH_2OH)\\ |\\ C(CH_2OH)_2\\ |\\ CH_2COO(CH_2OH)\end{array}$$

mw 252.22; no information available on this, the parent compd of its nitrated deriv **(2,2-Dimethylol)-dimethylol-glutarate Tetranitrate** called by Blatt **Pentaerythritol Diglycolate Tetranitrate,**



$$\begin{array}{l}CH_2COO(CH_2ONO_2)\\ |\\ C(CH_2ONO_2)_2\\ |\\ CH_2COO(CH_2ONO_2)\end{array}$$

mw 432.22, N 12.96%, OB to CO_2 minus 29.6%; crysts; mp – explodes on heating to high temp. It is an expl ca 86% as powerful as TNT or 79% of PA, by Lead Block Test; can be prepd by nitration of parent compd (Refs 3, 4 & 5)

Refs: 1) Beil, not found (Parent compd) 2) Beil, not found (Tetranitrate) 3) Westfälisch-AnhaltischeSprengstoffAG, GerP 638432-3(1936) & CA **31**, 1212(1937) 4) A.H. Blatt, OSRD **2014**(1944), p XVII 5) ADL Rept on Study of Pure Explosive Compounds, Part 1(1947), p105

Dimethylol-di(oxymethylene)-peroxide or **Bis(hydroxymethylether)-peroxide,** $(HO)H_2C.O.CH_2.O_2CH_2O.CH_2(OH)$; mw 154.12, O 62.29%; heavy oil, sp gr 1.2765 at 16.7°, n_D 1.4216 at 16.7°; it is a weak expl, sol in w. It can be prepd by treating an ethereal soln of Di(methylol)peroxide, $HOH_2C.O_2.CH_2OH$, with 2 mols of formaldehyde, HCHO (Ref 2)

Note: On long standing, the oil gradually changes to a crystalline substance, $C_2H_{12}O_7$, which partly sublimes at 140–145°; the balance melting at 152°. A very expl subst can be prepd from it, called *Cyclodioxymethyleneperoxide* (called *Pertrioxymethylen*, in Ger) (See Vol 3 of Encycl, p C594-L)

Refs: 1) Beil, not found 2) A. Rieche & R. Meister, Ber **66**, 709(1933)

Di(methylol)-ethane or Bis(hydroxymethyl)-ethane and Derivatives

1,1-Di(methylol)-ethane; Bis(methylol)-methylmethane or *1,1-Di(hydroxymethyl)-ethane*, $(HOH_2C)_2CH.CH_3$; mw 90.12; no info on this compd was found in the literature thru 1966. It may be considered the parent compd of its nitrated derivs: 1,1-Di(methylol)-2-nitroethane and 1,1-Di(methylolnitrate)-2-nitroethane

1,1-Di(methylol)-2-nitroethane or *Bis(methylol)-nitromethylmethane*, $(HOH_2C)_2.CH.CH_2(NO_2)$; mw 135.12, N 10.37%; no info on this compd was found in the literature thru 1966. It was used by Wyler (Ref 3)

1,1-Di(methylolnitrate)-2-nitroethane; Bis(nitroxymethyl)-nitromethylmethane or **1,1-Di(methylol)-nitromethylmethane Dinitrate**, $(O_2NO.H_2C)_2CH.CH_2(NO_2)$; mw 225.12, N 18.67%, OB to CO_2 minus 24.88%; crysts, mp 39.5°; explodes at higher temps; was prepd by Wyler (Ref 3) on nitration of 1,1-di(methylol)-2-nitroethane and proposed for use as an explosive

1,2-Di(methylol)-ethane, $(HOH_2C)CH_2.CH_2(CH_2OH)$, mw 90.12; no info on this compd was found in the literature thru 1966. It may be considered as the parent compd of its nitrated derivs: 1,2-Di(methylol)-1-nitroethane and 1,2-Di(methylolnitrate)-1-nitroethane

1,2-Di(methylol)-1-nitroethane, $(HOH_2C).CH(NO_2).CH_2(CH_2OH)$; mw 135.12, N 10.37%; no info on this compd was found in the literature thru 1966

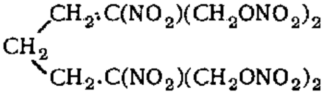
1,2-Di(methylolnitrate)-1-nitroethane; 1,2-Di(nitroxymethyl)-1-nitroethane; or **1,2-Bis(nitroxymethyl)-1-nitroethane**, $(O_2NO.H_2C).CH(NO_2).CH_2(CH_2ONO_2)$, mw 225.12, N 18.67%, OB to CO_2 minus 24.88%; no info on this compd was found in the literature thru 1966

Refs: 1) Beil, not found [Di(methylol)-ethane] 2) Beil, not found [Nitrated derivs of Di(methylol)-ethane] 3) J.A. Wyler, USP 2195551(1940) & CA **34**, 5283(1940) 4) Le Vétérinaire Commandant SALLE, MP **36**, 305(1954) (Toxicity properties)

Di(methylol)-heptanediol or Bis(hydroxymethyl)-heptanediol and Derivatives

2,6-Di(methylol)-1,7-heptanediol, $(HO)H_2C.CH(CH_2OH).CH_2.CH_2.CH_2.-CH(CH_2OH).CH_2(OH)$, mw 192.25; may be considered as the parent compd of its nitrated deriv although not used to prep it:

2,6-Dinitro-2,6-Dimethylol-1,7-heptanediol Tetranitrate; 2,6-Bis(hydroxymethyl)-2,6-dinitro-1,7-heptanediol Tetranitrate; or **1,5-Dinitro-1,1,5,5-tetramethylolpentane Tetranitrate** (called by Blatt, Ref 3)



$$CH_2\begin{cases}CH_2.C(NO_2)(CH_2ONO_2)_2\\ CH_2.C(NO_2)(CH_2ONO_2)_2\end{cases}$$

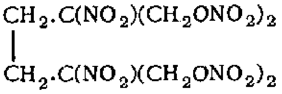
mw 462.25, N 18.18%; OB to CO_2 minus 31.2%; crysts, mp 85°. It is an expl more powerful than TNT (137% TNT by Ballistic Mortar Test); temperature of explosion (calcd) 3731°K; heat of expln at const vol 498.3kcal/mol; and heat of combstn at C_v 1104.4kcal/mol. It can be prepd by condensation of 1,5-dinitropropane with formaldehyde, followed by nitration of the product

Refs: 1) Beil, not found 2) H.B. Haas, OSRD Rept **2016**(1943) 3) A.H. Blatt, OSRD **2014**(1944) 4) Anon, OSRD **5746** (1945) 5) ADL Pure Expl Compds, Vol 1(1947), pp119 & 148; Vol 4(1952), p585 (compd No 82)

Dimethylolhexanediol or Bis(hydroxymethyl)-hexanediol and Derivatives

2,5-Dimethylol-1,6-hexanediol, $(HO)CH_2.CH(CH_2OH).CH_2.CH_2.CH(CH_2OH).-CH_2(OH)$; mw 178.22; may be considered as the parent compd of its nitrated deriv, although not used to prep it:

2,5-Dinitro-2,5-dimethylol-1,1-hexanediol Tetranitrate; 2,5-Bis(hydroxymethyl)-2,5-dinitro-1,6-hexanediol Tetranitrate or **1,4-Dinitro-1,1,4,4-tetramethylolbutane Tetranitrate** (called by Blatt, Ref 3)



$$\begin{array}{l}CH_2.C(NO_2)(CH_2ONO_2)_2\\ |\\ CH_2.C(NO_2)(CH_2ONO_2)_2\end{array}$$

mw 448.22, N 18.75%, OB to CO_2 minus 21.4%, crysts, mp 134°. A powerful expl: Ballistic Mortar Test 147% TNT; temp of expln (calcd) 4187°K; heat of expln at const vol 554.6kcal/mol; heat of combstn at const vol 958.6kcal/mol; sensitivity to impact — equal to PETN. It can be prepd by conden-

sation of 1,4-dinitrobutane & formaldehyde, followed by nitration of the product
Refs: 1) Beil, not found 2) H.B. Haas, OSRD Rept **2016**(1943) 3) A.H. Blatt, OSRD **2014**(1944) 4) ADL Pure Expl Compds, Vol **1**(1947), p119 (Compd No 81) & Vol **4**(1952), p585

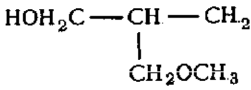
Dimethylolmethane, Propanediol, Trimethyleneglycol or Bis(hydroxymethyl)-methane and Derivatives

Dimethylolmethane, $HOH_2C.CH_2.CH_2OH$; mw 76.09; liq, fr p minus 32°, bp 214.2°, d 1.0538 at 20°, n_D 1.4389–1.4397 at 20°. Other props & methods of prepn are found in Beil. (Ref 1)

Dimethylolmethane Dinitrate, $(O_2NO)CH_2.CH_2.CH_2(ONO_2)$; mw 166.09, N 16.87%, OB to CO_2 minus 28.9%, lt-yel oil, having a sweet taste, fr p below −20°, d 1,3952 at 20°; miscible with MeOH, eth, chlf, benz & acet; v sl sol in CS_2. It was first prepd by Blechta (Ref 3) by nitrating trimethyleneglycol with mixed acid at 8°. This compd showed the same sensitiveness to impact as NG (drop test of 4cm with 2kg wt)
Refs: 1) Beil **1**, 475, (247), [540] & {2158} 2) Beil **1**, [542] & {2161} 3) F. Blechta, SS **17**, 57(1922) & CA **16**, 2991(1922) 4) Clift & Fedoroff, Vol **2**(1943), p D7

1,1-Di(methylol)-methoxyethane; [Bis(hydroxymethyl)-2-methoxy]-ethane or 2-Methylmethoxy-propane and Derivatives

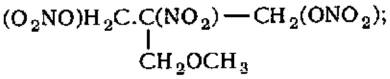
1,1-Di(methylol)-2-methoxy-ethane,



$HOH_2C-CH(CH_2OCH_3)-CH_2OH$

; mw 120.15, may be considered as the parent compd of its nitrated deriv, although not used to prep it:

1,1-Dimethylol-2-methoxy-1-nitroethane Dinitrate or **2-Methylmethoxy-2-nitropropane Dinitrate,**

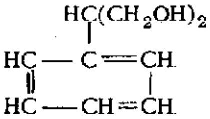


$(O_2NO)H_2C.C(NO_2)(CH_2OCH_3)-CH_2(ONO_2)$;

mw 255.15, N 16.47%, OB to CO_2 minus 34.5%. Liquid explosive resembling NG in its properties, but is less stable to heat. It is sl less powerful than Blasting Gelatin (91%) described in Vol 2 of Encycl, p B211-R. 1,1-Dimethylol-2-methoxy-1-nitroethane Dinitrate can be prepd by condensing formaldehyde & 1-methoxy-2-nitroethane in alkaline medium and nitrating the product (Ref 3)
Refs: 1) Beil, not found 2) Beil, not found (Nitrated deriv) 3) A.H. Blatt, OSRD **2014** (1944) 4) Not found in later Refs thru 1966

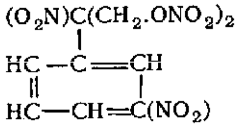
Di(methylol)-methylbenzene; Di(methylol)-phenylmethane or Di(methylol)-toluene and Derivatives

1,1-Di(methylol)-methylbenzene, or 1,1-Di(methylol)-phenylmethane,



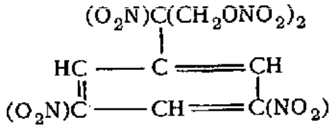
mw 152.19; may be considered as the parent compd of its nitrated derivs, although not used to prep them:

1,1-Di(methylolnitrate)-nitromethyl-3-nitrobenzene or **1,1-Di(methylol)-m-nitrophenylnitromethane Dinitrate,**



mw 332.19, N 16.87%, OB to CO_2 minus 57.8%; crysts; ignites at 270°; does not expl up to 360°; power by Ballistic Mortar Test 114% TNT, less sensitive than TNT and is of satisfactory stability. It can be prepd by condensing m-nitrophenylnitromethane with formaldehyde and nitrating the product (Refs 2 & 3)

1,1-Di(methylolnitrate)-nitromethyl-3,5-dinitrobenzene or **1,1-Di(methylol)-3,5-dinitrophenyl-nitromethane Dinitrate,**



mw 377.19, N 18.57%, OB to CO_2 minus 40.3%; crysts; mp 114–115°; ignites at 360°; power by Ballistic Mortar Test 126% TNT; sl less

sensitive than PETN; stability adequate; heat of combstn 1022.9kcal/mole. It can be prepd by condensing phenylnitromethane with formaldehyde and nitrating the product (Refs 2 & 3)

Refs: 1) Beil, not found 2) A.H. Blatt & F.C. Whitmore, OSRD **1085**(1942), pp13–14 3) A.H. Blatt, OSRD **2014**(1944)

Di(methylol)-methylenediamine or Bis(hydroxymethyl) - methylenediamine and Derivatives

N,N'-Di(methylol)-methylenediamine, $(HOH_2C)NH.CH_2.NH(CH_2OH)$; mw 106.13, N 26.40%; may be considered as the parent compd of its nitrated deriv, although not used to prep it:

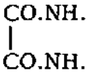
N,N'-Di(methylol)-methylenedinitramine or **N,N'-Bis(hydroxymethyl)-methlenedinitramine,** $(HOH_2C)(O_2N)N.CH_2.N(NO_2)(CH_2OH)$; mw 196.13, N 28.57%; prisms, mp 70–75° (decomp), expl at high temp. It can be prepd by passing gaseous formaldehyde to saturation thru a soln of methylenedinitramine, $(O_2N)HN.CH_2.NH.(NO_2)$, in ethyl acetate, followed by cooling the mixt to 0° (Ref 3)

Refs: 1) Beil, not found 2) Beil, not found (Nitrated deriv) 3) F. Chapman et al, JCS **1949**, 1635–36 & CA **44**, 1411(1950) 4) Not found in later Refs thru 1966

Di(methylol)-methylmethane or Bis(hydroxymethyl) -methylmethane. See under Di(methylol)-ethane

Di(methylol)-oxamide or Bis(hydroxymethyl) - oxamide and Derivatives

N,N'-Bis(hydroxymethyl) -oxamide,



$$\begin{array}{l} CO.NH.CH_2OH \\ | \\ CO.NH.CH_2OH \end{array}$$

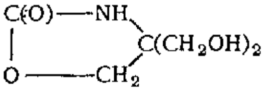
; mw 148.12, N 18.91%; crysts (from 50% acetic acid), mp 204°; prepd by condensation of formaldehyde (40%) with oxamide, $(CONH_2)_2$, in the presence of K_2CO_3 (Ref 2). Welcher (Ref 3) reported a novel synthesis from $(CN)_2$, paraformaldehyde & 12N HCl at 28–32°

Note: No nitrated derivs of N,N'-Di(methylol)-oxamide were found in the literature thru 1966

Refs: 1) Beil, not found 2) O. Moldenhauer et al, Ann **583**, 37(1953) & CA **49**, 1694(1955) 3) R.P. Welcher, USP 2953599 (1960) & CA **55**, 3441(1961) (mp reported 244–55°) 4) M.M. Movsumzade & N.G. Kerimova, AzerbKhimZh **1963**(2), 13 & CA **59**, 13818(1963) (Prepd simple ethers of N,N'-Dimethylol-oxamide) 5) K.A. Kornev & L.V. Litvinenko, KhimPromInformNaukTekhnZb **1964**(3), 36 (Ukrain) & CA **61**, 16029(1964) (Prepd compd, mp 204°, and epoxy monomers) 6) Kirk & Othmer, 2nd edit. **14**(1967), p367 (Report mp 214°)

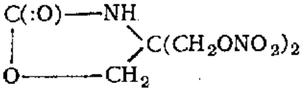
Di(methylol)-oxazolidone or Bis(hydroxymethyl) - oxazolidone and Derivatives

4,4-Di(methylol)-1-oxazolidone, or *4,4-Bis-(hydroxymethyl)-1-oxazolidone,*



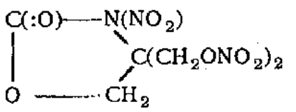
mw 147.13, N 9.52%; crysts (from methanol), mp 108–109° (Ref 3) & 109.5–110.7° (Ref 4). It was prepd from trimethylolaminomethane, $H_2N.C(CH_2OH)_3$, and chlorourethane (ethylchlorocarbonate), $ClCO_2C_2H_5$, in aq NaOH soln, as described on pp 50 & 128–29 of Ref 3

4,4-Di(methylolnitrate)-1-oxazolidone or **4,4-Bis(nitroxymethyl)-1-oxazolidone,**



mw 237.13, N 17.71%; crysts (from methanol), mp 106–107°; was prepd by treating the above compd with 98% nitric acid at 5–10° (Ref 3), pp 129–30). Its expl props are not reported

4,4-Di(methylolnitrate)-3-nitro-1-oxazolidone or **3-Nitro-4,4-bis(nitroxymethyl)-1-oxazolidone,**



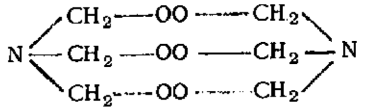
mw 282.13, N 19.86%; crysts (from methanol), mp 122–123°; expl at higher temps; was prepd by nitrating 4,4-di(methyl)-1-oxazolidone with

mixed nitric-sulfuric acid at 5–10°, followed by heating at 50° during 30 minutes (Ref 3, p 130–31). Its expl props were not investigated. See also Refs 4 & 5

Refs: 1) Beil, not found 2) Beil, not found (Nitrated derivs) 3) A.T. Blomquist & F.T. Fiedorek, OSRD **4134** (PB 18867) (1944), pp 50–1 & 128–31 4) J. Cason & F.S. Prout, JACS **71**, 1220(1949) & CA **43**, 6575(1950) 5) A.T. Blomquist & F.T. Fiedorek, USP 2485855(1949) & CA **44**, 3517(1951)

Di(methylol)-peroxide· Bis(hydroxymethyl)-peroxide; Dimethylenediol-peroxide; Dihydroxydimethyl-peroxide or Diformalperoxidehydrate [Called in Ger Dioxy-dimethyl-peroxyd or Bis(oxymethyl)-peroxyd], $(HOH_2C)—OO—(CH_2OH)$; mw 92.08, O 69.51%; large prisms (from eth or chlf), mp 64–65° (with sl decompn); v sol in w & alc; less sol in cold eth; sl sol in benz & chlf. When in the dry state it is stable in storage; explodes on heating to 70° in a direct flame or when introduced into NH_3 gas; catches fire on contact with reduced Fe, Pt-black, or warm CuO; detonates from friction or impact

It was first prepd by Legler (Ref 2) as one of the products of incomplete combustion of ether. He erroneously called the product "Hexaoxymethylenperoxyd". Nef (Ref 3) prepd di(methylol)-peroxide by treating formaldehyde with aq hydrogen peroxide and called the product "Diformalperoxydhydhydrat". Baeyer & Villager (Ref 4) confirmed the results of Nef and also showed that by treating Nef's product with ammonia, an extremely violent explosive was produced. They named it **Hexamethylentriperoxyd-diamin**. Its structure was detd as:



mw 208.17, N 13.46, O 46.12%. It is described in Beil **27**, 771 and in Blatt [OSRD **2014**(1944) under the name Hexamethylenetriperoxide-diamine]

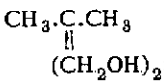
Fenton (Ref 5) prepd Di(methylol)-peroxide by evaporating equal volumes of formaldehyde and hydrogen peroxide. Wieland & Wingler (Ref 6) prepd it in 80–90% yield from an ethereal soln of formaldehyde and hydrogen peroxide. There are other methods of prepn, such as the action of ozone on ethylene or on β-butylene (Ref 1), p[642]) (See also Refs listed below)

Refs: 1) Beil **1**, 576, (302), [642] & {2582} 2) L. Legler, Ber **14**, 602(1881); **15**, 1255 (1882); **18**, 3343(1885) and Ann **217**, 381 (1883) 3) J.U. Nef, Ann **298**, 292 & 328 (1897) 4) A. Baeyer & V. Villager, Ber **33**, 2480 & 2485(1900) 5) H. Fenton, ProcRoySoc **90**, 493(1914) 6) H. Wieland & A. Wingler, Ann **431**, 302 & 306(1923) 7) H.G. Fischer, Ann **476**, 244(1929) 8) E. Briner & P. Schnorf, Helv **12**, 154(1929) 9) H. Wieland & H. Sutter, Ber **63**, 66(1930) 10) A. Rieche & R. Meister, Ber **66**, 724(1933) 11) Tobolsky & Mesrobian (1954) 45, 47, 171 & 179 12) N.A. Milas et al, JACS **77**, 254(1955) & CA **50**, 5512(1956) 13) M. Vanpée, CR **242**, 373(1956) & CA **50**, 6933(1956) 14) F.H. Sobels, Nature **177**, 979(1956) & CA **50**, 15965(1956) 15) Sax (1963) (Not found) 15a) Walker (1964), pp39, 245 & 578

Di(methylol)-phenylmethane or **Di(methylol)-toluene.** See Di(methylol)-methylbenzene in this Vol

Di(methylol)-propane; Di(methylol)-dimethyl-methane; Dimethyl-propanediol; Bis(hydroxymethyl)-propane; or Pentaglycol (PGc) and Derivatives

2,2-Di(methylol)-propane or *2,2-Dimethyl-propane-1,3-diol,*



$CH_3.\underset{(CH_2OH)_2}{\underset{\|}{C}}.CH_3$; mw 104.15 col ndls; mp 127–129°; bp 210° at 760mm and 110° in vacuo; v sol in w, alc and in hot trichloroethylene. It was first prepd in 1894 by Apel & Tollens (Ref 2) by heating dilute formaldehyde with isobutyraldehyde and slaked lime. Walker & Turnbull (Ref 3) patented method of prepn by adding 1 mol NaOH to a mixture of two equivalents of 37% formaldehyde and 1 mol of isobutyraldehyde

$2CH_2O + (CH_3)_2CH.CHO + NaOH =$
$HCOONa + (CH_3)_2C(CH_2OH)_2$

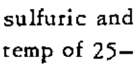
Walker (Ref 6) also patented a method for prepn of polyhydroxy compds, including pentaglycol, by treating aliphatic aldehydes with formaldehyde in a small amount of alkali, which serves as a catalyst (See also Ref 4)

Its laboratory method of prepn consists of adding dropwise KOH soln in absolute alcohol to a mixture of formaldehyde and isobutyraldehyde contained in a separatory funnel (Ref 5)

Nitration of pentaglycol gives explosives

Refs: 1) Beil 1, 483, (251), [550] & {2147} 2) M. Apel & B. Tollens, Ber **27**, 1088(1894) 3) J.F. Walker & N. Turnbull, USP 2135063 (1938) & CA **33**, 997(1939) 4) J.F. Walker, "Formaldehyde", Reinhold, NY (1944), p154 5) A.T. Blomquist & F.T. Fiedorek, OSRD **4134**(1944) & PB 18867(1944) 6) J.F. Walker, USP 2400724(1945) & CA **40**, 6094 (1946) 7) Walker (1964), pp292–93 & 579

2,2-Di(methylol)-propane Dinitrate; 2,2-Di(nitroxymethyl)-propane; Pentaglycol Dinitrate; 2,2-Dimethyl-1,3-propanediol Dinitrate or **Dimethyloldimethylmethane Dinitrate,** abbreviated as **PGcDN,**



$$\begin{array}{c} CH_3.C.CH_3 \\ \| \\ (CH_2.ONO_2)_2 \end{array}$$

, mw 194.15, N 14.43%, OB to CO_2 minus 74.2%; lt yel crysts, sp gr 1.38 melting at ca 28° to yellow viscous liquid resembling glycerin; bp explodes on heating, nearly insol in water. Spaeth (Refs 3 & 4) prepd PGcDN by adding pentaglycol slowly with stirring to a mixture contg 55% sulfuric and 45% nitric acids at an initial temp of 25–30°. After the addn the temp was raised to 40° and the PGcDN, being lighter than the acid, separated on the surface. Then it was decanted, neutralized with aq Na carbonate and washed with w. Blomquist & Fiedorek (Ref 5) prepd the PGcDN by nitrating pentaglycol with 98.5% nitric acid

Following properties are listed in Blatt (Ref 6) under 2,2-Dimethylpropane-1,3-diol Dinitrate:

Heat of Combustion – 715.85kcal/mol
Heat of Formation – 97.7kcal/mol
Ignites at 270° but does not explode up to 360°
Impact Sensitivity – Bruceton No 5 Machine, negative with 5kg wt at 90cm
Power by Ballistic Mortar – 108% TNT
Thermal Stability at 135° – acid in 40 mins (which is considered not very satisfactory)

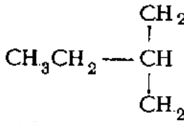
Spaeth (Refs 3 & 4) claimed the dinitrate to be suitable for use in expl mixts with NC & NG, especially since it is a freezing point depressant for NG. Following are examples of expl mixtures proposed by Spaeth:

a) *Gelatin Dynamite:* PGcDN 10, NG 20, NC 1.0, Na nitrate 50, carbonaceous combustible 18 & chalk 1%
b) *Propellant:* PGcDN 30, NC(12.85 to 13.35% N) 65 & petroleum jelly 5%
c) *Propellant:* PGcDN 35 & NC (12.85 to 13.35% N) 65%, plus 1% of a stabilizer, such as DPhA
d) *Propellant:* PGcDN 5 to 25, NG 25 to 5, NC(12.85 to 13.35 N) 65 & petroleum jelly 5%

Rinkenbach (Ref 7) patented blasting explosives consisting of PGcDN and *1,1,1-Tris(hydroxymethyl)-ethane Trinitrate.* These mixts were of low sensitivity and practically nonfreezing in winter and were claimed to be suitable replacements for mixts based on NG–NGc or NG–Nitrosugars

Refs: 1) Beil 1, {2199} (PGc) 2) Beil, not found (PGcDN) 3) C. Spaeth, USP 1883045 (1931) & CA **27**, 845(1933) 4) C. Spaeth, CanP 339804(1934) & CA **28**, 3235(1934) 5) A.T. Blomquist & F.T. Fiedorek, OSRD **4134**(1944) & PB 18867(1944) 6) A.H. Blatt, OSRD **2014**(1944) (2,2-Dimethylpropane-1,3-diol Dinitrate) 7) W.H. Rinkenbach, USP 2709130(1955) & CA **49**, 14325(1955)

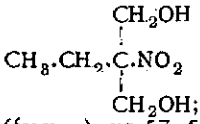
1,1-Di(methylol)-propane or *2-Ethyl-1,3-dimethylpropane-1,3-diol,*



$$\begin{array}{l} \qquad\quad CH_2OH \\ \qquad\quad | \\ CH_3CH_2 — CH \\ \qquad\quad | \\ \qquad\quad CH_2OH \end{array}$$

; mw 104.15; may be considered as the parent compd of its nitrated derivs, although not used to prep them:

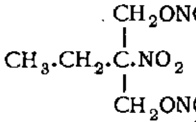
1,1-Di(methylol)-1-nitropropane or *2-Ethyl-2-nitropropane-1,3-diol,*



$$\begin{array}{l} \qquad\quad CH_2OH \\ \qquad\quad | \\ CH_3.CH_2.C.NO_2 \\ \qquad\quad | \\ \qquad\quad CH_2OH \end{array}$$

; mw 149.15, N 9.39%; ndls (from w), mp 57–58°, decomp when distilled at 10mm press; sol in alc, w & eth; was prepd by condensation of 1-nitropropane & formaldehyde in the presence of K_2CO_3(Ref 1)

1,1-Di(methylol)-1-nitropropane Dinitrate or **2-Ethyl-2-nitro-1,3-propanediol Dinitrate,**



$$\begin{array}{l} \qquad\quad CH_2ONO_2 \\ \qquad\quad | \\ CH_3.CH_2.C.NO_2 \\ \qquad\quad | \\ \qquad\quad CH_2ONO_2 \end{array}$$

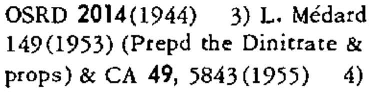
; mw 239.15, N 17.57%; liq, d 1.443 at 20°, n_D 1.4734 at 20° (Ref 3); it withstands 82.2° Heat Test for 5 mins; power by ballistic mortar test 92% of Blasting Gelatin; was prepd by condensing formaldehyde with 1-nitropropane in an alkaline medium, and nitrating the product. It was reported to be a poor gelatinizer for soluble NC used in blasting compns

See also the Refs listed below

Refs: 1) Beil **1**, 483 & {2199} 2) Blatt, OSRD **2014**(1944) 3) L. Médard, MP **35**, 149(1953) (Prepd the Dinitrate & detd its props) & CA **49**, 5843(1955) 4) L. Médard & M. Thomas, MP **35**, 157, 164, 172(1953) (Detd $Q^{v}_{comb.s.m}$ as 693.5kcal/mole) & CA **49**, 11284(1955) 5) J. Sallé, MP **36**, 305 (1954) (Detd the Dinitrate to be a vasodilator, less effective than NG, but excercising a more lasting effect) & CA **49**, 16218(1955) 6) K. Szyc-Lewanska & M. Syczewski, Nitro Compds, ProcInternSymp, Warsaw **1963**, 257 (Pub 1964 in Engl) (Detd the Dinitrate to be a good polymerizer for Me methacrylate) & CA **63**, 14989(1965)

Di(methylol)-toluene. See Di(methylol)-methylbenzene in this Vol

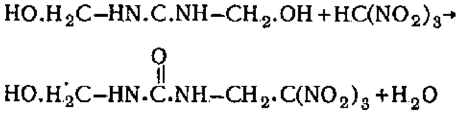
N,N'-Di(methylol) Urea and Derivatives



$$\begin{array}{c} \qquad\quad O \\ \qquad\quad \| \\ HO.H_2C—HN.C.NH—CH_2OH \end{array}$$

; mw 120.11, N 23.33%; crysts, mp 126°; sol in w & methanol; insol in eth; can be prepd by combination of urea and formaldehyde in the presence of salts of alkaline catalysts. It is capable of polymerization to synthetic resins

When dimethylolurea was treated with one mole of nitroform at the laboratory of US Rubber Co (Ref 3), the following reaction was believed to take place:



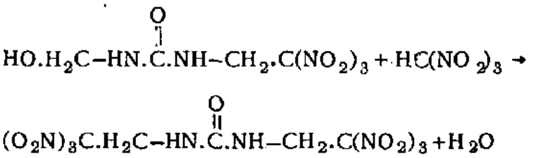
$$HO.H_2C–HN.C.NH–CH_2.OH + HC(NO_2)_3 \rightarrow$$

$$\begin{array}{c} \qquad O \\ \qquad \| \\ HO.H_2C–HN.C.NH–CH_2.C(NO_2)_3 + H_2O \end{array}$$

The resulting solid substance had mw 253.14, N 27.66%, melted at 183–184° with decompn and could be detonated with difficulty by hammer blow

The name for the above explosive was not given in USRubberCo reports No 3 & No 4. We suggest **N-(Methylol)-N'-(2,2,2-trinitroethyl) Urea**

When this substance was treated with another mol of nitroform, the following reaction was believed to take place:



$$\begin{array}{c} \qquad O \\ \qquad \| \\ HO.H_2C–HN.C.NH–CH_2.C(NO_2)_3 + HC(NO_2)_3 \rightarrow \end{array}$$

$$\begin{array}{c} \qquad\quad O \\ \qquad\quad \| \\ (O_2N)_3C.H_2C–HN.C.NH–CH_2.C(NO_2)_3 + H_2O \end{array}$$

The resulting solid substance had mw 386.16, N 29.02%, melted at 189–90° with decompn and detonated readily and sharply when struck with a hammer

The name for the above substance was not assigned in USRubberCo reports No 3 & No 4, but it is probably correct to name it **N,N'-Bis(2,2,2-trinitroethyl) Urea**

Refs: 1) Beil **3**, 59, (27), [49] & {111} 2) R.L. Shriner, OSRD **2054**(1943) (Dimethylolurea) 3) I.J. Schaffner, USRubberCo (Passaic, New Jersey), QuarterlyProgRept **No 3**, Nord 10129, Apr 30 to July 31, 1948, pp 24–8 (Preliminary data) 4) I.J. Schaffner, Ibid, Rept **No 4**, July 31–Sept 30, 1948, pp 5–6 (Final results) 5) CondChemDict (1961), p397-R (Dimethylolurea) 6) Walker (1964), pp220 & 279 (Dimethylolurea)

Dimethyloxamide and Derivatives

N,N'-Dimethyloxamide (abbr as **DMO**), $H_3C.NH.CO.CO.NH.CH_3$; mw 116.12, N

24.13%; ndls or plates, sp gr 1.3 at 4°, mp 209–217° (corr); bp sublimes; sl sol in cold w (2.44 at 9.4°); sol in hot w; less sol in alc; fairly sol in chlf. Various methods of prepn are given in Beil (Ref 1). Desseigne (Ref 15) claimed to prep it in 98% yield by treating a 40% aq soln of methylamine with ethyl oxalate at RT. Davis (Ref 11) stated that DMO can be prepd by the interaction of methylamine with an ester of oxalic acid. Allenby & Wright (Ref 13) used diamyl oxalate because it can be easily prepd by azeotropic distn of oxalic acid and amyl alcohol

N,N′-Dinitroso-N,N′-dimethyloxamide (DNsoDMO)

$H_3C.N(NO).CO.CO.N(NO).CH_3$, mw 174.12, N 32.18%; crysts, mp – not reported. It was prepd by Reimlinger (Ref 17) by action of nitrous gases or N_2O_4 in CCl_4 on DMO. He found it stable and esp suitable as an intermediate for the prepn of CH_2N_2(Diazomethane)

Preussman (Ref 18) stated that in one of several expts in prepn of DNsoDMO, the chlf used as the solvent for nitrosation was not distd in vacuo but removed at atm pressure on a hot water bath in a hood. An extremely violent expln took place toward the end of the distn, resulting in complete destruction of the distn app and considerable damage to the hood, while 2 persons standing by suffered facial cuts and hearing damage from the pressure wave. It is therefore advisable, when working with DNsoDMO, to operate at reaction temps as low as possible

N,N′-Dinitro-N,N′-dimethyloxamide (DNDMO)

(Code named **MNO**),

$H_3C.N(NO_2).CO.CO.N(NO_2).CH_3$; mw 206.12, N 27.18%, OB to CO_2 minus 38.8%; col ndls; sp gr 1.52 (crysts), mp 122–124°(decomp), bp – does not expl even at 360°. It is sl sol in water, beginning to hydrolyze at ca 40°. Desseigne (Ref 15) gives its solubilities in 100g of other solvents at 20° as follows: acetone 31.0, ethyl acetate 17.3, chloroform 10.6, benzene 9.6, ethylol (95%) 0.74 & carbon tetrachloride 0.56. It was first prepd by Franchimont (Refs 2 & 4) on nitration of dimethyloxamide with very strong nitric acid, allowing to stand and pouring into water. Thiele & Meyer (Ref 5) dissolved dimethyloxamide in crude nitric acid, added fuming sulfuric acid and poured onto ice. Allenby & Wright (Ref 13) prepd it in 98% yield as follows: Stir a mixture of 130g concd sulfuric acid, 167g of 15% oleum and 100g of nitric acid (sp gr 1.42) in a 500ml round bottom flask and cool to 20°. Add with stirring 60g of dimethyloxamide over a period of 1 hr and continue stirring for addnl 30 mins. Pour onto cracked ice, collect ppt on a filter, wash with w and dry. Purify it by recrystallization from 1:1 by vol of chlf/high boiling petr eth, using ca 250ml of solvent

Desseigne (Ref 15) prepd DNDMO in 97% yield by nitrating dimethyloxamide with mixed nitric-sulfuric acid contg the amt of nitric acid 20% in excess of requirement. Mixed acid was of such compn that resulting spent (residual) acid contained less than 12.5% water

Following are expl and other properties of pure DNDMO:

Abel's Test at 80° – more than 1 hour(Ref 15)

Heat of Combustion – 511.9kcal/mol at C_v (Ref 12)

Heat of Formation – 74.5kcal/mol, based on heat of combstn of 508.4kcal/mol (Ref 13)

Hygroscopicity at 25° – gains 0.03% at 90% RH and 0.20% at 100% RH (A thin coating of paraffin prevents hygroscopicity and hydroysis) (Ref 2)

Impact Sensitivity – FI (Figure of Insensitiveness) 89% of TNT, which means that it is less sensitive than TNT. Sensitivity using Bruceton No 3 Machine with 5kg weight – more than 100cm vs 70cm for TNT (Ref 12) Coating with thin film of paraffin reduces sensitivity ca 10%

International Test at 75° – loses 0.5%

Power by Ballistic Mortar – 116% TNT (Ref 12)

Power by Trauzl Test – 110% PA or 115% TNT (Ref 12); 105% TNT (Ref 13)

Rate of Detonation – 5050m/sec at sp gr 1.0; 6760 at 1.42; and 7050 at 1.5 (Refs 12 & 13)

Reactivity – stable to hot concd HCl; reacts with alkalies with formation of salts of methylnitramine (Ref 12)

Sensitivity to Impact – See Impact Sensitivity
Sensitivity to Sympathetic Detonation From 60% Dynamite – more sensitive than TNT (Ref 13)
Stability in Storage. See Surveillance
Surveillance. Storage for 6 months in a closed tube at 65° caused no decrease in stability. Storage for 30 days at 100° produced no red fumes of nitrogen dioxide (Ref 12)
Thermal Stability at 135° – not acid and no expln in 300 minutes (Ref 12)
Vacuum Stability at 120° – 5g sample evolved 5cc in 5 hours (Ref 12)
Velocity of Detonation. See Rate of Detonation

Accdg to Davis (Ref 11, p395), DNDMO has interesting expl props, but it is limited in use because it develops acidity when wet with water

Accdg to Naoum (Ref 7) as quoted by Davis (Ref 11) and Blatt (Ref 12), 30 parts of DNDMO and 70 parts of PETN yield an eutectic which melts at 100° and can be cast to form an HE insensitive enough to pass the Rifle Bullet Test. Its rate of deton of 8500m/sec is equal to that of PETN under best conditions. The further addn of DNDMO or incorporation of camphor lowers the mp still more; it affects the brisance only slightly, but has a significant phlegmatizing action. A mixture of PETN 60, DNDMO 30 and dimethyloxalate 10% melts at 82° and has, when cast, a vel of deton 7900m/sec which is higher than vel of cast Picric Acid

Herz & Naoúm (Ref 10) reported moldable expls formed by mixing 50 parts of DNDMO with TNT, TNNaphthalene, TNChlorobenzene, etc

Allenby & Wright (Ref 13) reported prepn of a pourable HE with setting point 75° when mixing DNDMO in equimolecular amt with Picric Acid. The rate of deton of this expl mixt was 6680 to 7500m/sec, depending on the densities of 1.41 to 1.59

Accdg to Blatt (Ref 12), DNDMO has been evaluated as betw Tetryl & TNT in performance, but of lower stability

Refs: 1) Beil **4**, 61, (330), [564] & {131} (DMO) 2) Beil **4**, 86, [585] & {168} (DNDMO) 3) Beil, **4**, not found (DNsoDMO) 4) A.P.N. Franchimont, Rec **2**, 96 (1883); **4**, 197 (1885) & **13**, 311 (1894) 5) J. Thiele & C. Meyer, Ber **29**, 961 (1896) 5a) J.B. Tingle, AmChemJ **20**, 318 (1898) 6) Westfälisch-AnhaltischeAG, GerP 203190 [CA **3**, 717 (1909)] and 291830 [CA **15**, 1997 (1921)] (A detonator and proplnt contg DNDMO) 7) P. Naoúm (to Dynamit AG), GerP 499403 [CA **24**, 4160 (1930)] (Formation of eutectic with 70% PETN melting below 100°) 8) A. Schmidt, SS **29**, 262 (1934) (Thermochemical measurements) 9) A. Haid, F. Becker & P. Dittmar, SS **30**, 67 (1935) (Stability measurements) 10) E. Herz & P. Naoúm, GerP 65972 (1938) & CA **32**, 6868 (1938) (Moldable expl chges contg DNDMO) 10a) Clift & Fedoroff, Vol **2** (1943), pD4 11) Davis (1943), 394–95 12) A.H. Blatt, OSRD **2014 (1944)** 13) O. Allenby & C.F. Wright, CanadJRes **25B**, 295–300 (1947) & CA **41**, 6722 (1947) 14) P. Arthur Jr, USP 2484481 (1949) & CA **44**, 1749 (1950) (Use of DNDMO as a catalyst in condensation of aldehyde resins) 15) G. Desseigne, MP **30**, 110–22 (1948) & CA **46**, 904 (1952) (Prepn and some props of DNDMO) 16) L. Médard & M. Thomas, MP **37**, 129–30, 138 (1955) & CA **51**, 716 (1957) (Some props of DNDMO, including $Q^{v}_{combstn}$ 2475.5cal/g) 17) H.K. Reimblinger, ChemBer **94**, 2547 (1961) & CA **56**, 1337 (1962) [Use of DNsoDMO as a starting material for Diazomethane(qv)] 18) A. Preussmann, AngewChem **75**(13), 642 (1963) & CA **59**, 4967 (1963) (Expln in the synthesis of Dinitrosodimethyloxamide) 19) Walker (1964), p 243 (DMO)

Dimethyl-oxetane and Derivatives

3,3-Dimethyl-oxetane, Bis(methyl)-oxetane or *Trimethylene oxide,*

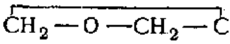
$\overline{\mathrm{CH_2{-}O{-}CH_2{-}C}}\mathrm{.(CH_3)_2}$; mw 86.13, O 18.58%; liq, bp 79.5–80°; obtd from 3-chloro-2,2-dimethyl-1-propanol by stirring with powd KOH in tert-amyl alcohol at 95°, and boiling at 48° to separate the oxetane (Ref 4)

3,3-Di(or Bis)(nitratomethyl)oxetane,

$\overline{\mathrm{CH_2{-}O{-}CH_2.C}}\mathrm{(CH_2ONO_2)_2}$; mw 208.13, N 13.46%; crysts, sp gr 1.76, mp 88–91°, ignition temp 233°; somewhat sensitive to impact (34cm with 2.5kg wt); thermal stability by vacuum test, 1.12ml gas/g at 100° in 48 hrs. It was prepd at Hercules Powder Co by Kouba et al by treating Pentaerythritol Trinitrate with alcoholic KOH, as described in detail in Ref 2. Elrick et al (Ref 3) prepd the compd by reaction of Pentaerythritol Trinitrate & Na ethylate or alcoholic NaOH at 70° for 8 hrs

Refs: 1) Beil, not found 2) D.L. Kouba, H.D. McNeil & R.H. Saunders, Monthly Progress Report on High Explosives, Navy-Contract **NOrd-11280**, Task A, Experiment Station, Hercules Powder Co, Wilmington, Delaware, May 1952 3) D.E. Elrick et al, JACS **76**, 1374(1954) 4) A.L. Rocklin, BelgP 632243(1963) & CA **61**, 644(1964)

3,3-Di(or Bis)(nitromethyl)oxetane,

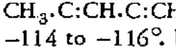


$\overline{CH_2 - O - CH_2 - C}(CH_2NO_2)_2$; mw 176.13, N 15.91%; long ndls (from alc), mp 147–48°; was obtd when a mixt of 3,3-bis(iodomethyl)-oxetane, $AgNO_2$, and dry ether was stirred for 4 hrs at 22°, then heated under reflux for 22 hrs; the Ag salts were removed by filtration and the filtrate concentrated to remove eth; the oily residue was triturated with ice-cold alc to yield crysts which were thrice recrystallized from alc (Ref 2). No expl props were reported

Refs: 1) Beil, not found 2) A.T. Nielsen & W.G. Finnegan, Tetrahedron **22**(3), 929 (1966) & CA **64**, 17516–17(1966)

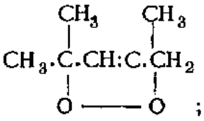
2,4-Dimethyl-1,3-pentadienne and Derivatives

2,4-Dimethyl-1,3-pentadienne,



$CH_3.\overset{CH_3}{\overset{|}{C}}:CH.\overset{CH_3}{\overset{|}{C}}:CH_2$; mw 98.17, liq, fr p −114 to −116°. bp 93.1°, d 0.7368 at 20°, n_D 1.4412 at 20°; other props & methods of prepn are found in Beil (Ref 1). It forms a dimer, $C_{14}H_{24}$, & higher polymers on treating with 80% sulfuric acid at 0°

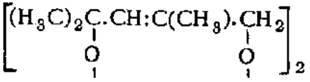
2,4-Dimethyl-1,3-pentadienne Peroxide,



$$\begin{array}{lllll} & CH_3 & & CH_3 & \\ & | & & | & \\ CH_3. & C & .CH:C. & CH_2 & \\ & | & & | & \\ & O & \text{———} & O & \end{array}$$

; mw 128.17, O 24.97%; solid, mp – forms a dimer, $C_{14}H_{24}O_4$ when heated to 80° and decomposes explosively when heated rapidly to 100–120° It can be prepd by oxidation of 2,4-dimethyl-1,3-pentadienne by means of long exposure to UV light (Refs 2, 3 & 4)

Note: The structure of this compd has not been definitely established. Another formula was also proposed by Jacquemain who did not isolate the compd



$$\left[\begin{array}{lll} (H_3C)_2C & .CH:C(CH_3). & CH_2 \\ \;\;\;\;\;\;\;\;\;\;| & & | \\ \;\;\;\;\;\;\;\;\;\;O & & O \\ \;\;\;\;\;\;\;\;\;\;| & & | \end{array}\right]_2$$

Refs: 1) Beil **1**, 257, (121), [235] & {1003} 2) Beil **1**, {1004} 3) R. Jacquemain, CR **215**, 200(1942) & CA **38**, 4903(1944) 4) Tobolsky & Mesrobian (1954), 31–2

Dimethylpentane and Derivatives

Dimethylpentane or Heptane, C_7H_{16}; mw 100.20. Four isomers are described in the literature:

2,2-Dimethyl-pentane, $H_3C.C(CH_3)_2.CH_2.CH_2CH_3$; liq, bp 79.2° (Ref 1)

2,3-Dimethylpentane, $H_3C.CH(CH_3).CH(CH_3).$-$CH_2.CH_3$; liq, bp 89.8° (Ref 2)

2,4-Dimethyl-pentane, $H_3C.CH(CH_3).CH_2.$-$CH(CH_3).CH_3$; liq, bp 80.5° (Ref 3)

3,3-Dimethyl-pentane, $H_3C.CH_2.C(CH_3)_2.$-$CH_2.CH_3$; liq, bp 86.1° (Ref 4)

3-Nitroso-3-nitro-2,4-dimethylpentane, $[(H_3C)_2CH]_2C(NO).NO_2$; mw 174.20, N 16.08%; non-freezing blue oil, bp – dec at 54°; was prepd by treating diisopropylketoxime in ether soln with NO_2 under cooling & with exclusion of light

Dinitro-dimethylpentane, $C_7H_{14}N_2O_4$; mw 190.20, N 14.73%. Four isomers are found in Beil:

2,3-Dinitro-2,3-dimethylpentane, $H_5C_2.C(CH_3)(NO_2).C(CH_3)_2.NO_2$; crysts (from alc), mp 88–88.4° (Ref 6)

2,4-Dinitro-2,3-dimethylpentane, $H_3C.CH(NO_2).CH(CH_3).C(CH_3)_2.NO_2$; pale yel oil, bp 90–92° at 0.5mm press; sol in alkalies (Ref 6)

2,4-Dinitro-2,4-dimethylpentane, $(H_3C)_2C(NO_2).CH_2.C(NO_2)(CH_3)_2$; prisms, mp 81–82°; sol in eth & petr eth (Ref 5)

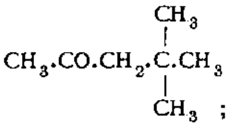
3,3-Dinitro-2,4-dimethylpentane, $(H_3C)_2CH.C(NO_2)_2.CH(CH_3)_2$; liq, bp 203–207° at 717mm press (with partial decompn) (Ref 5)

Their methods of prepn are given in Refs. No expl props reported

Refs: 1) Beil 1, 157, [120] & {442} 2) Beil 1, [120] & {445, 449} 3) Beil 1, 158, (59), [121] & {449} 4) Beil 1, 158, (59), [121] & {452} 5) Beil 1, 158 (Nitroso-nitro deriv & Dinitro derivs) 6) Beil 1, {448} (Dinitro derivs)

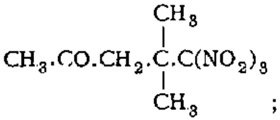
Dimethylpentanone and Derivatives

4,4-Dimethyl-2-pentanone,



$CH_3.CO.CH_2.\overset{CH_3}{\underset{CH_3}{C}}.CH_3$; mw 114.18, liq, bp 125–30°; prepd by oxidation of 2,4,4-trimethyl-2-pentanol (Ref 1). May be considered as the parent compd of its trinitro deriv, although not used to prep it:

4,4-Dimethyl-5,5,5-trinitro-2-pentanone,



$CH_3.CO.CH_2.\overset{CH_3}{\underset{CH_3}{C}}.C(NO_2)_3$; mw 249.18, N 16.86%; lt yel liq, bp 109–13° at 8mm, or 92–93° at 2mm; n_D 1.4717 at 20°; burned in flame rapidly but was not detonated by a hammer blow. It was prepd in the lab of USRubberCo at Passaic NJ from nitroform $CH(NO_2)_3$, and mesityl oxide, $CH_3.CO.CH:C(CH_3)_2$, as described by Schaffner (Ref 2)

Refs: 1) Beil 1, 702 2) I.J. Schaffner, USRubberCo, Quarterly Progress Reports, **No 4**, pp 7–8 (1948) and **No 5**, p 27 (1948–49)

Di(3-methylpentynyl)-3-peroxide or **Bis(1-methyl-1-ethyl-2-propynyl)-peroxide.** See under Acetylene Hydroperoxides and Peroxides in Vol 1 of Encycl, p A66-R

Dimethylperoxide or **Methylperoxide,** $H_3C.OO.CH_3$; mw 62.07, O 51.56%; gas at RT with a sweet ethereal odor; sp gr 0.8677 0°/4°; fr p minus 100–105°, bp 13.5° at 740mm; n_D 1.3503 at 0°; detonates easily when in the gaseous or liquid state by heat or impact with the evolution of a yel flame; insensitive to impact when in the solid state; sol in w and warm concd sulfuric acid; v sol in cold eth and alc; sol in toluene and ethyl acetate. It was prepd by treating dimethylsulfate with 10% hydrogen peroxide and K hydroxide in nitrogen atmosphere at 15° (Refs 1–5)

Sensitiveness of peroxide was determined by Baker et al (Ref 6)

Refs: 1) Beil 1, [271] 2) A. Rieche & W. Brumshagen, Ber **61**, 951 (1928) 3) A Rieche & F. Hitz, Ber **62**, 218 (1929) 4) R.H. Wiley, USP 2357298 (1944) 5) Tobolsky & Mesrobian (1954), 19, 164 & 177 6) G. Baker et al, Chem & Ind (London) **1964** (48), 1988 & CA **62**, 2661 (1965)

N,N-Dimethylphenylamine. See under N,N-Dimethylaniline and Derivatives

Di(methylphenylamino)-ethane or **Di(toluidino)-ethane.** See Bis(methylphenylamino)-ethane in Vol 2 of Encycl, p B150-R

Di(methylphenylamino)-propane or **Di(toluidino)-propane.** See Bis(methylphenylamino)-propane in Vol 2, p B151-L

N,N-Dimethyl-p-phenylaniline. See N,N-Dimethylaminobiphenyl or N,N-Dimethylxenylamine

Dimethylphenylcarbamide. See Dimethylphenylurea

N,N′ Dimethyl-m-phenylenediamine; N,N′-Bis(methyl)-diaminobenzene; or 1,3-Di-(methylamino)-benzene and Derivatives

N,N′-Dimethyl-m-phenylenediamine (abbr **DMPDA**), $H_3CHN.C_6H_4.NHCH_3$; mw 136.19, N 20.57%; oily liq; browns on standing; bp 165–170° at 10mm; was prepd by Fischer in the following manner: N-methyl-m-phenylenediamine was heated for 4 hrs with formic acid and the excess acid was distd off. The resulting viscous mass was crystallized from benz and the crysts dissolved in methanol. The soln was treated with Na methylate, followed by methyl iodide and the resulting dimethylformyl derivative was freed of methanol by distn and heated for several hours with dil sulfuric acid for complete saponification

It reacts with Na nitrite giving dinitrosamine. It forms also mono-, di-, tri-, tetra- & pentanitro compounds

Refs: 1) Beil **13**, 39 2) O. Fischer, Ann **286**, 173(1895)

N,N'-Dimethyl-N,N'-dinitroso-m-phenylene diamine, $C_6H_4[N(NO).CH_3]_2$; mw 194.19, N 28.85%; pltlts (from alc), mp 109–10°; was prepd by nitriting the parent compd with HNO_2 (Refs 1 & 2). No expl props were reported

Refs: 1) Beil **13**, 53 2) O. Fischer, Ann **286**, 168(1895)

N^1,N^1-Dimethyl-4-mononitro-m-phenylenediamine, $H_2N.C_6H_3(NO_2).N(CH_3)_2$; mw 181.19, N 23.19%; yel crysts (from alc), mp 135°; sol in dil sulfuric acid with a yel color; was prepd from 3,4-dinitrodimethylaniline & alc ammonia under prolonged heating or by heating in a tube at 120° (Refs 1 & 2)

Refs: 1) Beil **13**, 57 & [30] 2) P. van Romburgh, Rec **42**, 804(1923) & CA **18** 49(1924)

Dimethyldinitro-m-phenylenediamine, $C_8H_{10}N_4O_4$; mw 226.19, N 24.77%. Four isomers are found in Beil:

N^1,N^1-Dimethyl-2,4-dinitro-m-phenylenediamine, $H_2N.C_6H_2(NO_2)_2.N(CH_3)_2$; crysts (from acet), mp 160–62°; was prepd by treating 2,3,4-trinitrodimethylaniline with alcoholic ammonia at RT (Refs 1, 5 & 6)

N,N'-Dimethyl-2,4-dinitro-m-phenylenediamine, $(O_2N)_2C_6H_2(NH.CH_3)_2$; yel crysts, mp mp 169–70°; was prepd by action of an alc soln of methylamine on 2,4,6-trinitromethylaniline or on 2,4-dinitro-3-methylaminodimethylaniline in a tube at 125°(Refs 2 & 5)

N^1,N^1-Dimethyl-4,6-dinitro-m-phenylenediamine, $H_2N.C_6H_2(NO_2)_2N(CH_3)_2$- no description given in CA or in Beil; was prepd by action of alc ammonia on 2,4,6-trinitrodimethylaniline (Refs 3, 5 & 6)

N,N'-Dimethyl-4,6-dinitro-m-phenylenediamine, $(O_2N)_2C_6H_2(NH.CH_3)_2$; yel ndls (from acet), mp >290° (dec); obtd with other products by warming 5-chloro-1,2,4-trinitrobenzene or 5-bromo- deriv with ca 4 moles of benzaldehyde-methylimide in MeOH (Refs 4 & 7)

No expl props of the Dinitro derivs were reported in the literature

Refs: 1) Beil **13**, [31] 2) Beil **13**, 59 & [31] 3) Beil **13**, 59 & [32] 4) Beil **13**, [32] 5) P. van Romburgh & D.W. Wensink, VerslAkadAmsterdam **23**, 967(1914) & CA **9**, 1175(1915) 6) A. Forster & W. Coulson, JChemSoc **121**, 1995(1922) & CA **17**, 76 (1923) 7) M. Giua, Gazz **53**, 55–56(1923) & CA **17**, 2273(1923)

Dimethyltrinitro-m-phenylenediamine, $C_8H_9N_5O_6$; mw 271.19, N 25.83%. Three isomers are known:

N^1,N^3-Dimethyl-N^1,4,6-trinitro-m-phenylenediamine, $H_3C.NH.C_6H_2(NO_2)_2.N(NO_2).CH_3$; crysts, mp 181.5°; was prepd by treating 4,6-dinitro-3-methylnitramino-phenetole with an alcoholic methylamine soln; can be nitrated to form an expl pentanitro deriv (Refs 1 & 5)

N,N'-Dimethyl-2,4,6-trinitro-m-phenylenediamine, $(O_2N)_3C_6H(NH.CH_3)_2$; yel crysts, mp 235–40°(dec), bp – explodes on heating to high temp; was prepd by treating 2,3,4,6-tetranitromethylaniline with an aq soln of methylamine (Refs 2,4, 5 & 6)

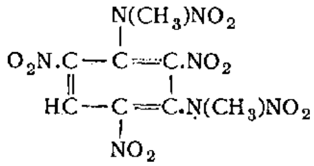
N,N-Dimethyl-2,4,6-trinitro-m-phenylenediamine, $H_2N.C_6H(NO_2)_3.N(CH_3)_2$; crysts, mp 187°; was prepd by action of cold aq dimethylamine soln on 2,3,4,6-tetranitroaniline, or by treating 2,3,4,6-tetranitrodimethylaniline with cold aq ammonia (Refs 3 & 5)

Refs: 1) Beil **13**, [32] 2) Beil **13**, 60 & [33] 3) Beil **13**, [33] 4) J.J. Blanksma, Rec **21**, 324(1902) 5) A. Forster & W. Coulson, JCSoc **121**, 1992, 1996(1922) & CA **17**, 76(1923) 6) J.J. Blanksma & P.G. Fohr, Rec **65**, 817(1946) & CA **41**, 2704(1947)

N^1,N^3-Dimethyl-N^1,2,4,6-tetranitro-m-phenylenediamine, $H_3C.NH.\ C_6H(NO_2)_3.N(NO_2).CH_3$; mw 316.19, N 26.58%; golden-yel crysts (from acet acid), mp 190–92°, bp – explodes at high temp; can be prepd by treating 2,4,6-trinitro-3-methylnitraminophenetole with an alcoholic methylamine soln and by other methods (Refs 1 & 2)

Refs: 1) Beil **13**, 61 & [34] 2) A. Forster & W. Coulson, JChemSoc **121**, 1993(1922) & CA **17**, 76(1923)

N,N′-Dimethyl-2,4,6-trinitro-N,N′-dinitrophenylenediamine; N,N′-Bis(methyl)-N,N′-dinitramino-2,4,6-trinitrobenzene; 1,3-Bis(methylnitramino)-2,4,6-trinitrobenzene or N,N′-Dimethyl-N,N′,2,4,6-pentanitrophenylene-1,3-diamine; called by the British **Ditetryl.** This must not be confused with American *Ditetryl,* called by the British *Octyl,* which is 1,2-Bis(2′,4′,6′-trinitroanilino)-ethane (See Vol 2 of Encycl, p B131-R),



$$\begin{array}{l} \qquad\qquad N(CH_3)NO_2 \\ \qquad\qquad\ | \\ O_2N.C - C = C.NO_2 \\ \qquad\ \| \qquad\ \ | \\ \quad HC - C = C.N(CH_3)NO_2 \\ \qquad\qquad\ | \\ \qquad\qquad NO_2 \end{array}$$

mw 361.19, N 27.15%, OB to CO_2 minus 42.1%; brown crysts (from acet + alc), mp 206°(corr) with decompn; sol in acet & in boiling glac AcOH; sl sol in boiling toluene; nearly insol in w and common org solvents such as alc, eth, etc. It was first prepd by van Romburgh in 1887 (Refs 1 & 2) by boiling N,N′-dimethyl-m-phenylenediamine with fuming nitric acid. The same investigator (Ref 3) prepd it by dissolving 1 volume of DMPDA in 2 vols of concd sulfuric acid and adding this soln gradually to calcd amt of concd nitric acid. This caused the mixture to turn red and to evolve copious vapors of nitrogen oxides. After the evoln of gas stopped, the liq was heated to boiling, then cooled and poured into a large vol of ice-water. The pptd yel ndls were dissolved in acet and reprecipitated by alc

Colver (Ref 5) prepd it by nitrating DMPDA with mixed nitric-sulfuric acid

Van Duin & van Lennep (Ref 6) prepd it by treating m-Nitrotetryl(2,3,4,6-Tetranitrophenylmethylnitramine with methylamine, whereby the 3rd nitrogroup was replaced by methylamino group and the resulting product was nitrated with mixed nitric-sulfuric acid. This method is listed in Blatt (Ref 10)

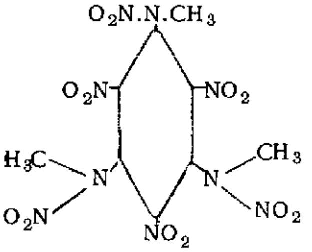
Clift & Fedoroff (Ref 9) mentioned that the Société Anonyme d Explosifs of Paris (Ref 4) prepd N,N′,2,4,6-Pentanitrodimethyl-m-phenylenediamine by nitration with concd nitric acid of $(O_2N)_2C_6H_2[N(NO_2)CH_3]_2$, which is the compd N,N′-Dimethyl-2,4-dinitro-m-phenylenedinitramine

Explosive and other properties of Pentanitrodimethylphenylenediamine are as follows:
Action of Flame – ignites and burns briskly in the open, a slightly luminous flame, but does not explode
Compatibility with Metals does not corrode them
Explosion Temperature – expl at 214° when heated at the rate of 20°/min
Impact Sensitivity with Kast apparatus using 2kg weight, max fall for 0/6 shots 21–26cm vs 49–51cm for Tetryl; min fall for 6/6 shots 36–37cm vs >60cm for Tetryl; sensitivity is comparable with Guncotton
Power by Trauzl Test – greater than 75% Guhr Dynamite
Thermal Stability by the Dutch Method – decomposes with evoln of nitrogen oxides after heating at 95° for 4 days (Refs 4, 5, 8 & 10)

Pentanitrodimethylphenylenediamine was patented in 1907 in France (Refs 4 & 9) for filling shells, mines, torpedoes and detonators either alone or mixed with other expls or with oxidizers such as nitrates, chlorates or perchlorates. Its mixture with MF detonates easily and therefore can be used for reducing the amt of MF in detonators

Van Duin & van Lennep (Ref 6) prepd a water-sol compd exploding at 160–170° by treating the Pentanitrodimethylphenylenediamine with ammonia (in water, methanol or ethanol), isolating the brown compd and treating it (after recrystallization from methyl salicylate) with aq Na hydroxide

Pentanitrodimethylphenylenediamine was discussed by Médard (Ref 11) as a compd being intermediate betw Tetryl and 1,3,5-Tris(methylnitramino)-2,4,6-trinitrobenzene,



Refs: 1) Beil **13**, 61, (18) & [34] 2) P. van Romburgh, Rec **6**, 251(1887) & JCS **54** (II), 1079(1888) 3) P. van Romburgh, Rec **7**, 1(1888) & JCS **54**(II), 1185(1888) 4) Société Anonyme d'Explosifs, FrP 391107(1907) & CA **4**, 2733(1910) 5) Colver (1918), 387 & 711–12 6) C.F. van Duin & B.C.R. van Lennep, Rec **39**, 161–70(1920) 7) A. Forster & W. Coulson, JCS **121**, 1992 & 1996(1922) & CA **17**, 76 (1923) (Nitro derivs of m-Nitrodimethylaniline) 8) A.H. Blatt & F.C. Whitmore, OSRD **1085**(1942), p105 9) Clift & Fedoroff, vol 2(1943), pp P4 & P5 (N,N',2,4,6-Pentanitro-dimethyl-m-phenylenediamine) 10) A.H. Blatt, OSRD **2014**(1944) [2,4,6-Trinitro-1,3-di(methylnitramine)-benzene] 11) L. Médard, MP **33**, 46(1951)

Dimethylphenylhydrazine and Derivatives

Dimethylphenylhydrazine, $C_8H_{12}N_2$; mw 136.19, N 20.58%. Five isomers are listed in Beil:

2,3-Dimethylphenylhydrazine, $(H_3C)_2C_6H_3.NH.NH_2$; ndls (from alc), mp 108°; dec on storage; sol in hot alc (Ref, p 171)

2,4-Dimethylphenylhydrazine, ibid, wh crysts or ndls (from eth), mp 85°, dec on exposure to light; its Hydrochloride salt, $C_8H_{12}N_2 + HCl + 2H_2O$, ndls – dec ca 183° and the Nitrate salt, $C_8H_{12}N_2 + HNO_3$, pltlts – dec 146–47° (Ref, pp549 & 173)

2,5-Dimethylphenylhydrazine, ibid, col ndls, mp 74–78°; dec in the light; sol in org solvs; insol in w; forms stable cryst salts with acids (Ref, pp 552 & 175)

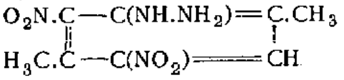
2,6-Dimethylphenylhydrazine, ibid, ndls (from petr eth), mp – not sharply at 46°; not stable (Ref, pp 548 & 172)

3,4-Dimethylphenylhydrazine, formula same as above, yel ndls (from eth), mp 57°; its Hydrochloride salt, $C_8H_{12}N_2 + HCl$, scales (from w), dec at 197° (Ref, p 172)

Other props & methods of prepn are found in the Refs

Ref: Beil. **15**, 548, 549, 552 & (171, 172, 173, 175)

4,6-Dinitro-2,5-dimethylphenylhydrazine,



$$\begin{array}{l} O_2N.C-C(NH.NH_2)=C.CH_3 \\ \quad\;\| \qquad\qquad\qquad\qquad\quad | \\ H_3C.C-C(NO_2)=\!=\!=\!=CH \end{array}$$

mw 226.19, N 24.77%; yel prisms (from alc), mp 180° (dec); sol in hot benz, chlf, eth & acet; v sl sol in petr eth; the soln in alc or acet gives a brick-red soln in presence of alkali; was prepd by reacting 2,3,5-trinitro-p-xylene & hydrazine-hydrate in hot alc (Refs 1 & 2)

Refs: 1) Beil **15**, (175) 2) M. Giua, Gazz **49** II, 168(1919)

Trinitrodimethylphenylhydrazine, $C_8H_9N_5O_6$; mw 271.19, N 25.83%. Two derivs are known:

N,N'-Dimethyl-N-(2,4,6-trinitrophenyl)-hydrazine or α,β-Dimethyl-picrylhydrazine, called by Knorr *Dimethylpikrazid,* $(O_2N)_3C_6H_2.N(CH_3).NH(CH_3)$; yel-red lflts, mp 141°; bp – puffs off with evolution of flame at higher temps; sol in chlf & ether; diffc sol in water & alcohol; was prepd by Knorr & Köhler from picryl chloride and N,N'-dimethylhydrazine in alc (Refs 1 & 3)

N,N-Dimethyl-N'-(2,4,6-trinitrophenyl)-hydrazine or **β,β-Dimethylpicrylhydrazine,** $(O_2N)_3C_6H_2.NH.N(CH_3)_2$, red crysts (from alc), mp 136.5°, bp – explodes on heating above its mp; readily sol in benz; diffc sol in eth; was prepd from reaction of N,N-dimethylhydrazine & picryl chloride in alcoholic KOH (Refs 2 & 4)

Refs: 1) Beil **15**, 493 2) Beil **15**, (147) 3) L. Knorr & A. Köhler, Ber **39**, 3264(1906) 4) H.J. Backer, Rec **31**, 152(1912) & CA **6**, 2744(1912)

Dimethylphenylurea and Derivatives

N,N-Dimethyl-N'-phenylurea, $C_6H_5.NH.CO.N(CH_3)_2$; mw 164.20, N 17.06%;

ndls (from w) or crysts (from alc), mp 134°; readily sol in alc, eth & benz; was prepd by reaction of aniline & dimethyl carbamic acid chloride in ether (Refs 1 & 2)
Refs: 1) Beil **12**, 348 & [205] 2) R. Stollé, JPrChem [2] **117**, 201(1927)

N,N-Dimethyl-N^1-(2-nitrophenyl)-urea, $O_2N.C_6H_4.NH.CO.N(CH_3)_2$; mw 209.20, N 20.09%; yel oil, fr p – becomes viscous at –15° w/o crystn at –30°; obtd by warming dimethylaniline & 2-nitrophenylisocyanate in ether soln (Ref)
Ref: Beil **12**, 694

Dinitrodimethylphenylurea, $C_9H_{10}N_4O_5$, not found in Beil

N,N'-Dimethyl-N-(2,4,6-trinitrophenyl)-urea or **N,N'-Dimethyl-N-picrylurea,** $(O_2N)_3C_6H_2.N(CH_3).CO.NH.CH_3$; mw 299.20, N 23.41%; yel ndls, mp–volatile in a capillary tube at 150° & melts (w decompn) at 169°; was prepd by reaction of dimethylcarbodiimide & Picric Acid in eth. No expl props were reported (Refs 1 & 2)
Refs: 1) Beil **12**, [424] 2) H. Lecher et al, Ann **445**, 54(1925) & CA **20**, 374(1926)

o-Dimethylphthalate (DMePh) or **Methylphthalate,** $C_6H_4(COOCH_3)_2$, mw 194.18; col liq, sp gr 1.189 at 25°/25°, n_D 1.5138 at 20°, fr p 5.5°, bp 280° at 734mm; 283.7°; sl sol in w to the extent of 0.43% at RT; sol in eth, benz, alc and ligroin; heat of combstn at C_V 1120.1kcal/mol. It can be prepd by methods listed in Refs 1, 2 & 4, such as by treating phthalic anhydride with methanol in presence of concd sulfuric acid

It is a non-explosive plasticizer for NC, as good as DBuPh (dibutylphthalate). It is used in US principally in double-base proplnts. Accdg to Davis (Ref 3) complete gelatinization of 100 parts of Pyrocellulose is achieved by 70 pts DMePh when dissolved in benzene, by 95 pts when dissolved in alc and by 115 pts when dissolved in ligroin

DMePh acts also as a flash-reducing agent

US proplnts using DMePh are listed in conf SPIA/M2 (Ref 5)

Accdg to Ullmann (Ref 2), DMePh has been used in Germany under the name "Palatinol M" as plasticizer in plastic and rubber industries

Requirements of the US Armed Forces, covered by Specification JAN-D-709(1), include:

1) *Color* – no darker than a "standard" prepd by dissolving 0.5ml of 0.1N iodine soln in 100ml of distd water
2) *Specific Gravity* at 15.5°/15.5° 1.196±0.10
3) *Ester Content* (as DMePh) – min 99%
4) *Acidity* (as phthalic acid) – max 0.03%
5) *Ash* – max 0.01%

Specification Tests for DMePh

1) *Color:* Compare the color with soln prepd by adding 0.5ml of 0.1N iodine soln to 100ml distd w

2) *Specific Gravity.* Det the sp gr at 15.5°/15.5° by means of a picnometer or Westphal balance. These methods are described in Vol 3 of Encycl, pp D68 & D69

3) *Ester Content (as DMePh)* Transfer 1.5g of the sample to a 250ml Pyrex round bottom flask (provided with ground-joint neck) and add (by means of calibrated buret or pipet) 50ml of 0.5N alcoholic KOH (prepd by adding 28g of reagent-grade KOH to 1 liter of 95% ethanol). Fit the flask by means of ground joints to a reflux condenser and heat on a water bath until the pptn appears to be complete. Add ca 15ml of distd w to dissolve the K phthalate and reflux, with occasional swirling of the flask, for at least 45 mins. Wash down the sides of the reflux condenser and the ground joints with ca 25ml of distd w and cool the flask with soln to 25°. Titrate with 0.5N HCl using 3 drops of phenolphthalein indicator [previously prepd by dissolving 1 g of phpht in 100ml of 85% (by vol) ethanol] until discoloration of soln

At the same time, run a blank determination on 50ml of alcoholic KOH, which was subjected to the same reflux distillation as above

$$\%\text{DMePh} = \frac{9.709(V-v)N}{W}$$

where: N = normality of the acid used
V = ml of acid used to titrate blank
v = ml of acid used to titrate excess of KOH after saponification
W = wt of sample

4) *Acidity.* Place 100ml of 95% ethanol into a 250ml Erlenmeyer flask, add 3 drops of

phpht indicator, and neutralize the alcohol with a few ml of 0.1N NaOH to a faint pink color. Add to the flask an accurately measured 50ml portion of the sample and titrate the mixture with 0.1N NaOH till reappearance of faint pink coloration

$$\%\text{Phthalic Acid} = \frac{0.166VN}{G}$$

where: N = normality of the NaOH soln
V = ml of NaOH soln required for titration of sample
G = sp gr of sample (as detd in test 2)

5) *Ash.* Weigh a 10g sample in an accurately tared porcelain crucible and evaporate nearly to dryness over a low flame or on a hot plate. Ignite the residue at a red heat, cool in a desiccator and weigh. Repeat the ignition to constant weight. The increase in wt in the crucible times 10 gives % ash

Note: The use of the term "Methylphthalate" instead of "Dimethylphthalate" (as it is done by some investigators) should be avoided because there is a true monomethyl compd, such as described in Beil **9**, 797

Refs: 1) Beil **9**, 797, (350) & [584] 2) Ullmann **8**(1931), 471–72 3) Davis (1943), 322 4) CondChemDict (1961), 398-R 5) Propellant Manual, SP1A/M2(1969) (Conf) Units 1027–1029 6) USSpecification JAN-D-709(1) (June 1949)

N,N-Dimethylpicramide. See 2,4,6-Trinitro-N,N-dimethylphenylamine under N,N-Dimethylaniline and Derivatives

α,β-Dimethylpicrylhydrazine. See N,N'-Dimethyl-N(2,4,6-trinitrophenyl)-hydrazine under Dimethylphenylhydrazine and Derivatives

Dimethylpicrylurea. See 1,3-Dimethyl-2,4,6-trinitro-phenylurea under Dimethylphenylurea

Dimethylpropane and Derivatives

2,2-Dimethylpropane; Neopentane or Tetramethylmethane, $CH_3.C(CH_3)_2.CH_3$; mw 72.15; col gas or very volatile liq; sp gr 0.603 at 0°/0°, fr p –20°, bp 9.5°; sol in alc, insol in w; present in small amts in natural gas; can be prepd by reaction of tert-butyliodide & Zn dimethyl, or from butyl Mg iodide & dimethylsulfate in ether (Refs 1 & 14)

2,2-Dimethyl-1-nitro-propane or Nitroneopentane, $(O_2N)CH_2.C(CH_3)_2.CH_3$; mw 117.15, N 11.96%; liq, fr p 13–14°, bp 146–51°, d 0.9494 at 20°, n_D 1.4099 at 30°; obtd with other products by treating 1-nitro-2-methyl-1-propane with methyl-Mg-iodide in ether at 0–10°, by action of NaOH on 3-nitro-2,2,4-trimethyl-4-pentanol, or by nitration of neopentane at 410° in the vapor phase (Refs 2, 6 & 7)

2,2-Dimethyl-1,3-dinitropropane or **Dinitroneopentane,** $(O_2N)CH_2.C(CH_3)_2.CH_2(NO_2)$; mw 162.15, N 17.28%, OB to CO_2 minus 109%; crysts, mp 93–94°, bp 120° at 10mm. It was prepd in the US during WWII in 57% yield by condensing acetone and nitromethane in presence of dimethylamine as a catalyst (Refs 3 & 5)

Following props are given by Blatt (Ref 5)

Explosion Temperature – decomposes but does not expl below 360°

Heat of Formation – 72.0kcal/mol

Hygroscopicity – no gain of wt at 25° & 100% RH

Impact Sensitivity – comparable to TNT

Initiation – difficult to detonate completely

Power – could not be determined on account of incomplete detonation

Thermal Stability at 135° – not acid and no expln after 300 mins

(See also Refs 3, 9, 11, 12 & 13)

2,2-Dimethyl-1,1,1-trinitropropane or **Trinitroneopentane,** $(O_2N)_3C.C(CH_3)_2.CH_3$; mw 207.15, N 20.29%; solid, mp 139–41°; burned rapidly but did not detonate from hammer blow. It was prepd in the lab of USRubberCo Passaic, NJ (Ref 10) from nitroform and isobutylene. This compd was prepd also by Plummer (Ref 18) and patented as an expl. Its method of prepn given in CA is not clear

2,2-Dimethyl-1,1,3-trinitropropane, $(O_2N)_2HC.C(CH_3)_2.CH_2NO_2$; crysts (from (from eth + petr eth), mp 121–22°; this compd and other polynitrocompds were prepd by nitration of mono- & dinitro- compds with nitrite ions (from sodium nitrite) & Ag nitrate in the presence of alk or neutral aqueous media (Refs 16 & 17). Its expl props were not reported

Refs: 1) Beil **1**, 141, (50) & [104] (Dimethylpropane) 2) Beil **1**, {373} (Mononitrodimethylpropane) 3) Beil **1**, {373} (Dinitrodimethylpropane) 4) Beil, not found (Trinitrodimethylpropane) 5) A.H. Blatt, OSRD **2014** (1944) (1,3-Dinitro-2,2-dimethylpropane) 6) G.D. Buckley, USP 2408607(1946) & CA **41**, 1232(1947) (1-Nitro-2,2-dimethylpropane) 7) G.D. Buckley, BritP 571804(1945) & CA **41**, 4508(1947) (Same as in Ref 6) 8) M. Senkus, USP 2418237(1947) & CA **41**, 4510 (1947) (1,3-Dinitro-2,2-dimethylpropane) 9) A. Lambert & H.A. Piggott, BritP 584789 (1947) & CA **41**, 5143(1947) (Same as above) 10) I.J. Schaffner, USRubber Co Quarterly Rept **No 4**(1948), p12 (Trinitro deriv) 11) G.S. Skinner & P.R. Wunz, JACS **73**, 3814(1951) & CA **46**, 2078(1952) (Dinitro deriv) 12) N. Radin & T. de Vries, AnalChem **24**, 971(1952)& CA **46**, 8539(1952) (Polarographic study) 13) J.F. Brown, JACS **77**, 6341(1955) & CA **50**, 2297(1956) (IR spectrum) 14) CondChemDict (1961), 789-L (Neopentane) 15) V.I. Solvetskii et al, IzvestAkadNauk, OtdelKhimNauk **1961**, 330 & CA **55**, 19473(1961) (Molecular absorption spectrum of trinitro deriv) 16) R.B. Kaplan & H. Shechter, JACS **83**, 3535(1961) & CA **56**, 8533(1962) 17) H. Shechter & R.B. Kaplan, USP 2997504(1961) & CA **58**, 5516(1963) 18) C.W. Plummer, USP 3049570(1962) & CA **57**, 15404(1962)

Dimethylpropanediol. See Di(methylol)-propane

Dimethylpropylenediamine and Derivatives

2,2-Dimethyl-propylene-1,3-diamine or *2,2-Dimethyl-1,3-diaminopropane*, $H_2N.CH_2.C(CH_3)_2CH_2.NH_2$, mw 102.18, N 27.52%; no info found in literature, although it is listed in CA 7th Collective Formula Index (1962-66), p376F (Refs 8 & 9)

Meyersberg (Ref 4) prepd it in small yield by condensation of acetone & Nitromethane, followed by reduction of resulting dinitrodimethylpropane; and Haas (Ref 5) outlined this procedure as:

$$H_3C.CO.CH_3 + 2CH_3NO_2 \rightarrow (H_3C)_2C(CH_2NO_2)_2 \xrightarrow{[H]} (H_3C)_2C(CH_2.NH_2)_2$$

Blomquist & Fiedorek (Ref 7) did not consider the method as promising and proposed the synthesis represented by the following equation:

$$(H_3C)_2CH.CHO \xrightarrow{HCHO} (H_3C)_2C(CH_2OH)_2 \xrightarrow{PBr_3} (H_3C)_2C(CH_2Br)_2 \xrightarrow{NH_3} (H_3C)_2C(CH_2NH_2)_2$$

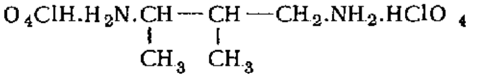
2,2-Dimethylpropylene-1,3-dinitramine; 2,2-Dimethyl-1,3-dinitraminopropane or **Pentaglycol Dinitramine,** $(O_2N.HN)H_2C.C(CH_3)_2.CH_2.(NH.NO_2)$, mw 192.18, N 29.16%; crysts, no info available on its props & method of prepn. This compd is listed in CA 7th Collective Formula Index (1962-66), p369F (Ref 10), but is not found in abstract

As the direct nitration of dimethylpropylenediamine was not promising, Blomquist & Fiedorek (Ref 7) proposed the following reactions:

$$(H_3C)_2C(CH_2NH_2)_2 \xrightarrow{Cl.COOC_2H_5} (H_3C)_2C(CH_2.NH.COOC_2H_5)_2 \xrightarrow[HNO_3]{Concd} (H_3C)_2C[CH_2N(NO_2).COOC_2C_5]_2 \xrightarrow[HCl]{NH_3} (H_3C)_2C[CH_2.NH(NO_2)]_2$$

It is an explosive, but its props have not been reported in the literature

2,3-Dimethylpropylene-1,3-diamino-diperchlorate,



$$O_4ClH.H_2N.\underset{CH_3}{CH}-\underset{CH_3}{CH}-CH_2.NH_2.HClO_4$$

mw 303.14, N 9.24%; crysts; no info reported on its props & method of prepn

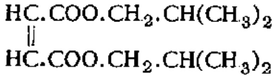
It is an explosive which cannot be satisfactorily detonated by MF (Ref 6)

Refs: 1) Beil, not found (Dimethylpropylenediamine) 2) Beil, not found (Dimethylpropylenedinitramine) 3) Beil, not found (Dimethylpropylenediamino-diperoxide) 4) (?) Meyersberg, Monatsh **26**, 43; Ann **3** (1905) 5) H.B. Haas, ChemRevs **32**, 373 (1943) 6) A.H. Blatt, OSRD **2014**(1944) (Under Perchlorates) 7) A.T. Blomquist & F.T. Fiedorek, OSRD **4134** & PB 18867 (1944), 21–2 & 97–9 8) J.R. Caldwell & W.J. Jackson Jr, USP 3236816(1966) & CA **64**, 14369(1966) 9) M.H. Goodrow, USP 3272781(1966) & CA **65**, 20252(1966)

10) K. Borer, JExptlBotany **17**(51), 378 (1966) & CA **65**, 4557(1966)

Di(methylpropyl)-maleate or Bis(methylpropyl) maleate and Derivatives

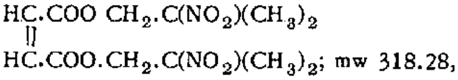
2,2-Di(methylpropyl)-maleate (called Diisobutylmaleinat in Ger),



$$\begin{array}{l}HC.COO.CH_2.CH(CH_3)_2\\ \|\\ HC.COO.CH_2.CH(CH_3)_2\end{array}$$

; mw 228.28, O 28.04%; liq, bp 125.6° at 6mm press, d 0.9820 at 20° & 0.9265 at 86.5°; n_D 1.4454 at 20°; was prepd by reaction of maleic acid, isobutyl alcohol & concd sulfuric acid in boiling benz (Ref 1)

Di(2-nitro-2-methylpropyl)-maleate,



$$\begin{array}{l}HC.COO\ CH_2.C(NO_2)(CH_3)_2\\ \|\\ HC.COO.CH_2.C(NO_2)(CH_3)_2\end{array}$$

; mw 318.28, N 8.80%; crysts (from alc), mp 78.5–79°; sol in benz, toluene, dioxane & acetone; insol in w & benzin. Its prepn was patented by C.J. Mighton [USP 2365717(1944) & CA **39**, 4627(1945)]: thru heating of maleic acid anhydride & 2-nitro-2-methyl-1-propanol in the presence of p-toluenesulfonic acid (Ref 2)

It was found to be a suitable plasticizer of NC when it was desired to obtain flexible solventless rolled sheet propellants (Ref 3)

Refs: 1) Beil **2**, {1925} [Di(methylpropyl)-maleate] 2) Beil **2**, {1925} [Di(2-nitro-2-methylpropyl)-maleate] 3) Anon, Summary-TechRept of Div 8, NDRC, Vol **1**(1946), p119

1,1-Dimethyl-2-propynylhydroperoxide or **3-Methyl-3-hydroperoxy-1-butyne.** See Vol 1 of Encycl, p A66-L under Acetylene Hydroperoxides and Peroxides

1,1-Dimethyl-2-propyn-1-ol and Derivatives

1,1-Dimethyl-2-propyn-1-ol or *3,3-Dimethyl-1-propyn-3-ol* (called 2-Methyl-3-butyn-2-ol in CA; and Dimethyl-acetylenylcarbinol or Dimethyl-äthinyl carbinol in Ger, $HC\vdots C.C(CH_3)_2OH$; mw 84.11, O 19.02%; very mobile liq of characteristic penetrating odor, fr p 3.01°, bp 104°; d 0.8624 at 20°; n_D 1.4212 at 20°; sol in w; was first prepd by reaction of acetylene & Na or K acetone in eth soln, and later by many other methods (Ref 1)

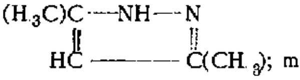
Its *Potassium salt*, deliquescent crysts; *Mercury salt*, pltlts, dec >240° and *Silver salt*, voluminous ppt, explodes violently when heated; have been prepd in addn to many other derivs (Ref 1)

1,1-Dimethyl-2-propyn-1-nitrite; 3,3-Dimethyl-1-propyn-3-ol (called Nitrite Ester of 2-Methyl-3-butyn-2-ol in CA), $HC\vdots C.C(CH_3)_2.ONO$; mw 113.11, N 12.38%; liq, bp 80–82° at 750mm press; n_D 1.3926 at 20°; was prepd when NOCl & ether were added to a stirred soln of 3-methyl-1-butyn-3-ol (parent compd) & C_5H_5N at 20–30°. This compd was patented as a proplnt fuel and for other uses (Ref 3)

Refs: 1) Beil **1**, (235), [505] & {1982} (Parent compd) 2) Beil, not found (Nitrite ester) 3) D.C. Rowlands, USP 2955131 (1960) & CA **55**, 9280(1961)

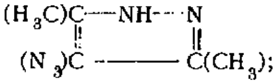
Dimethylpyrazole and Derivatives

3,5-Dimethylpyrazole,



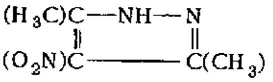
; mw 96.13, N 29.14%; plates, sp gr 0.864 at 20°/4°, mp 106–107°, bp 218° at 759mm; sol in w, alc, eth, benz & chlf; can be prepd by heating 3,5-dimethylpyrazole-carboxylic acid-1-anilide with water or alc, and by other methods (Ref 1)

3,5-Dimethyl-4-azido-pyrazole,



; mw 137.15, N 51.07%; lfts (from benz + petr eth); mp 81° (dec); bp – expl at higher temp; dec explosively on contact with concd sulfuric acid; easily sol in org solvents. It can be prep from 3,5-dimethylpyrazole-4-diazonium chloride and Na nitrite in acid soln (Refs 2 & 3)

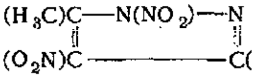
3,5-Dimethyl-4-nitro-pyrazole,



mw 141.13, N 29.87%; crysts, mp 124–128°; bp 325° at 749mm press; readily sol in alc,

eth & warm water; prepd by nitrating 3,5-dimethylpyrazole with mixed nitric and sulfuric acids, and by other methods (Refs 3 & 5). This compd dec on heating at high temp

3,5-Dimethyl-1,4-dinitropyrazole,



$$\begin{array}{lcl} (H_3C)C & -N(NO_2)- & N \\ \| & & \| \\ (O_2N)C & \text{———} & C(CH_3) \end{array}$$

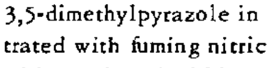
; mw 186.13, N 30.10%; crysts, mp 66–67°; was obtd together with 3,5-dimethyl-1-nitropyrazole when 3,5-dimethylpyrazole in acetic acid was nitrated with fuming nitric acid, then dropwise with acetic anhydride and the suspension gently warmed (Ref 6)

No expl props of this compd are reported

Refs: 1) Beil **23**, 74, (25) & [65] (Dimethylpyrazole) 2) Beil **23**, (25) (Azidodimethylpyrazole) 3) Beil **23**, 78, (25) & [69] (Nitrodimethylpyrazole) 4) Beil **23**, not found (Dinitrodimethylpyrazole) 5) G.T. Morgan & I. Ackerman, JChemSoc **123**, 1310 (1923) (Nitrodimethylpyrazole) 6) R. Hüttel & F. Büchele, ChemBer **88**, 1586 (1955) & CA **50**, 10088 (1956) (Dinitrodimethylpyrazole)

2,4-Dimethylpyridine or α,γ-Lutidine and Derivatives

2,4-Dimethylpyridine,



$$\begin{array}{lcl} HC & -N= & C(CH_3) \\ \| & & | \\ HC & -C(CH_3)= & CH \end{array}$$

; mw 107.15, N 13.07%; liq, sp gr 0.938 at 0°; 0.9331 at 20°; bp 157.3°; sol in w, alc or eth; can be prepd by the dry distillation of bituminous coal and by other methods; forms cryst compds with mineral acids (Refs 1 & 4)

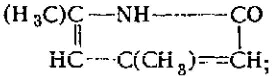
2,4-Dimethylpyridino-diazido-copper (II) Complex, $[CuC_7H_9N](N_3)_2$; mw 278.76, N 35.18%; solid, mp – expl at 208–209°, with a loud report, on preheated block; expl under light blow; was prepd by Cirulis & Straumanis (Ref 3) by dissolving $Cu(N_3)_2$ in 2,4-dimethylpyridine and addg an acid followed by MeOH or ether to ppt the product, or by addg NaN_3 to a 2,4-dimethylpyridine soln of a Cu^{++} salt to ppt the product

This and other complex compds of Cu(II) Azide are assigned the structure of non-electrolytes rather than that of penetration complexes because of their low solubility, low elec conductivity, stability toward water, and green-brn color (Ref 3)

Refs: 1) Beil **20**, 244, (85) & [160] 2) Beil **20**, not found (Cu diazide) 3) A. Cirulis & M. Straumanis, JPraktChem **162**, 319 (1943) & CA **38**, 1970 (1944) 4) CondChemDict (1961), 684-R (2,4-Lutidine)

Dimethylpyridone and Derivatives

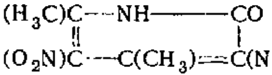
4,6-Dimethyl-2(1H)-pyridone or *2,4-Dimethyl-6-pyridone,*



$$\begin{array}{lcl} (H_3C)C & -NH- & CO \\ \| & & | \\ HC & -C(CH_3)= & CH \end{array}$$

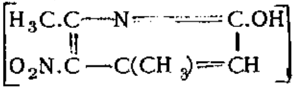
; mw 123.15, N 11.37%; ndls (from alc), mp 180–81°, bp 305–307°; sol in alc; sl sol in w & chlf; v sl sol in petr eth, eth & benz; was prepd by heating 6-hydroxy-2,4-dimethyl-5-cyanpyridine with concd HCl and by other methods (Ref 1)

4,6-Dimethyl-3,5-dinitro-2(1H)-pyridone,



$$\begin{array}{lcl} (H_3C)C & -NH- & CO \\ \| & & | \\ (O_2N)C & -C(CH_3)= & C(NO_2) \end{array}$$

; mw 213.15, N 19.72%; yel crysts (from 95% alc), mp 238° (dec), darkens 10° before mp & finally dec to a black liq; was prepd by addg a mixt of acetic anhydride & fuming nitric acid at 0° to 4,6-dinitro-5-nitro-2-pyridinol,



$$\left[\begin{array}{lcl} H_3C.C & -N= & C.OH \\ \| & & | \\ O_2N.C & -C(CH_3)= & CH \end{array}\right]$$

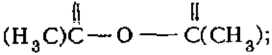
in Ac_2O at –5° for 10 sec, stirring, allowing the temp to rise to 40°, addg cracked ice to ppt the product (Ref 2)

Heating the dinitro deriv with PCl_5 for 4 hrs at 180° and pouring onto ice gives 4,6-Dimethyl-3,5-dinitro-2-chloropyridine, mp 84° & distilling at 120cc/min (Ref 2)

No expl props of the compds are reported

Refs: 1) Beil **21**, 51, (204) & [36] 2) R.P. Mariella, JOrgChem **20**, 1726 (1955)

2,6-Di(methyl)-4-pyrone,



$$\begin{array}{lcl} HC & -CO- & CH \\ \| & & \| \\ (H_3C)C & -O- & C(CH_3) \end{array}$$

; mw 124.13; wh crysts, mp 132°, bp 248–50°; was prepd by Collie & Tickle (Refs 1 & 2) by rapidly heating diacetylacetone with PCl_5 & HCl on a steam bath

It was tried in France before WWII (Ref 3) as a possible gelatinizer for NC in prepn of "poudres SD" (solventless proplnts). Although its gelatinizing power is remarkably great, it had to be rejected because, due to its basicity, it could attack the NC

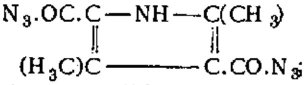
Refs: 1) Beil **17**, 291, (152) & [315] 2) J.N. Collie & T. Tickle, JCS **75**, 710 (1899) & **85**, 971(1904) 3) Anon, MP **29**, 7–9(1939)

2,6-Di(methylpyrone)-methyl Perchlorate, $[C_8H_{11}O_2]ClO_4$· mw 238.55; crysts, mp 190°; sl sol in alc & acet; v sl sol in cold w; was prepd by Piccard by warming dimethylpyrone with dimethyl sulfate & MeOH at 50°, cooling and treating with 20% $HClO_4$

It was suggested as a component in flash compns for blasting caps

Refs: 1) Beil **17**, (153) 2) J. Piccard, USP 1964077(1934) & CA **28**, 5242(1934)

2,4-Dimethylpyrrole-3,5-dicarbonyl Azide,



$$\begin{array}{ccc} N_3.OC.C & —NH— & C(CH_3) \\ \| & & \| \\ (H_3C)C & ——— & C.CO.N_3 \end{array}$$

; mw 233.20, N 42.05%; solid, mp – dec explosively at 119°; was prepd by treating the hydrazone deriv of 2,4-dimethyl-3,5-dicarboxylic acid with HCl & sodium nitrite (Ref 2)

Refs: 1) Beil, not found 2) H. Fischer & A. Waibel, Ann **512**, 195(1934) & CA **29**, 779(1935)

Dimethyl Sebacate (DMeSeb) (Called *Methylsebacate* by some investigators), $(CH_2)_8(COOCH_3)_2$, mw 230.30; ndls or plts, sp gr 0.9896 at 20/20°, n_D 1.4376 at 20°; mp 38°(26°) (Ref 1); 24.5°(Ref 4); bp 293–294° at 754mm; flash point 145°(293°F); sol in alc, benz, eth, ligroin; heat of combustion 1634kcal/mol at C_V. It can be prepd by heating sebacic acid $(CH_2)_8(COOH)_2$ with methanol and hydrochloric acid. It has been used as a plasticizer for NC in proplnts, as for example Unit No 398 listed in Ref 5. Davis (Ref 3) stated that for complete gelatinization of 100 parts of Pyrocellulose are required 80 parts of DMeSeb if in alcoholic soln, 70 in benzene and 105 in ligroin

Note: There exists also a monomethylsebacate listed in Beil **2**, [608]

US Armed Forces Specification requirements are unknown since there is no current specification

Refs: 1) Beil **2**, (293) & [608] 2) Ullmann **9**(1932), 356 3) Davis (1943), 322 4) CondChemDict (1961), 399-L 5) Proplnt Manual SP1A/M2 (1962) (Conf)

Dimethylsulfamide or Dimethylsulfonamide and Derivatives

N,N'-Dimethylsulfamide, $H_3C.NH.SO_2.NH.CH_3$; mw 124.16, N 22.56%; mp 77.6°(Grasselli). It was first prepd by Franchimont (Refs 1 & 3) by interaction of sulfuryl chloride with methylamine. Shriner dissolved them in chlf (Ref 7)

N,N'-Dinitro-N,N'-dimethylsulfamide or **N,N'-Dinitro-N,N'-dimethylsulfonamide,** $H_3CN(NO_2).SO_2.N(NO_2).CH_3$; mw 214.16, N 26.16%; OB to CO & SO_2 minus 21.4%; crysts (from benz); mp 88.5–90°; bp ca 160° and its vapor expl above 160°(Franchimont); accdg to Aaronson (Ref 6) it decompd at 170°(in 5 sec) and detonated at 180°, while below 180° only light grey smoke was observed. Accdg to Shriner (Ref 7) it decompd at 135° in 8 mins. It is sl sol in w; very sol in benz, chlf & hot alc; sol in eth & petr eth; was first prepd by Franchimont (Ref 3) by nitration of dimethylsulfamide with 98% nitric acid. Aaronson prepd it at PicArsnLab (Ref 6) by nitrating pure dimethylsulfamide (obtd from the Grasselli Chemical Department of the duPont Co) with 95% nitric acid. In one of the nitrations he used 10g of pulverized dimethylsulfamide and added it in small portions to 110g acid, while stirring and maintaining the temp below 20° by means of an ice-water bath. After continuing the nitration for an hour at ca 15°, the soln was poured into about 750ml ice-water and the resulting wh ppt filtered thru a Büchner funnel. After washing the residue ten times with w, it was dissolved in CP benz, transferred to a separatory funnel and the residual acid extracted by washing the benz soln with several portions of w. After drawing off nearly all the wash w from the last portion, the benz soln was shaken with an excess of anhydrous Ca chloride and the mixt filtered thru a Büchner. After evaporating the filtrate to a small volume, the crop of wh, lustrous crysts (8.6g) was obtd. Its mp was 89.4° (corr). Second & third crops were obtd from alc (4.2g), giving total of three crops as 12.8g. They were combined and recrystallized from a mixt of eth + petr eth and dried in a desiccator over Ca chloride. Their mp was 89.6° vs 90° obtd by Franchimont

Following properties of Dimethylsulfonamide were determined by Aaronson, Shriner and others:

Abel (KI) Test at 65.5° – 60+ minutes

Behavior Towards Sulfuric Acid. When 2 drops of concd acid were allowed to touch ca 5mg sample placed in a test tube, a slight flash and a rather loud pop resulted

Brisance by Sand Test. 0.2g being initiated by 0.24g MF plus 0.15g Tetryl crushed 29.5g sand, which is somewhat above that crushed by 0.2g Tetryl

Heat of Combustion – 472.1kcal/mol (Ref 5)

Hygroscopicity – nil

Impact Sensitivity (by BurMinesMachine) with 2kg weight 7cm vs 17cm for PETN

International Heat Test at 100° and Atmospheric Pressure: Loss in 1st 48 hrs 86.07% and in the 2nd 48 hrs 0.60%

Power by Ballistic Mortar – 126.5% TNT

Volatility at 100° – extremely high as shown by the International Heat Test

Not recommended by Aaronson for military purposes because of its high volatility, high impact sensitivity and low decompn temp (170°). He recommends instead to evaluate the properties of its higher homologue, *N,N^1-Dinitro-N,N^1-diethyl-sulfamide* (See under Dinitro compds in this Vol)

Naoúm (Ref 4) was of different opinion because he recommended its use in low-melting eutectic mixtures for cast-loading bombs, shells, etc

Refs: 1) Beil **4**, 83 & {165} (Dimethylsulamide) 2) Beil **4**, 86 (Dinitrodimethylsulfamide) 3) A.P. Franchimont, Rec **3**, 419(1884) 4) P. Naoúm (to DynamitAG), GerP 499403(1928) & CA **24**, 4160(1930) 5) A. Schmidt, SS **29**, 264(1934) 6) H. Aaronson, PATR **1374**(1943) 7) R.L. Shriner, OSRD **2054**(1943), pp10 & 14

Dimethylsulfate or **Methylsulfate,** $(CH_3)_2SO_4$; mw 126.13, col liq with very poisonous vapors; sp gr 1.352 at 0/4°, fr p minus 26.8°, bp 186° (decomp); sol in alc & eth; v sl sol in w. It can be prepd by adding fuming sulfuric acid to methanol and distilling in vacuo

It is a blister gas known in France before WWII as *Rationite* and in Ger as *D-Stoff* (Refs 2 & 4)

Refs: 1) Beil **1**, 283, (140), [271] & {1197} 2) C. Wachtell, "Chemical Warfare", ChemPubCo, Brooklyn, NY (1941) 3) CondChemDict (1961), 399-L 4) Encycl of Expls **2** (1962), p C169-L, item c

N,N$'$-Dimethylsulfonamide. See N,N^1-Dimethylsulfamide in this Vol

Dimethylsulfoxide (DMSO), $(CH_3)_2SO$, mw 94.13, col oil; sp gr 1.100 at 20/20°; fr p 18.45°, bp 189°; miscible with w, alc or eth; can be prepd by the liquid phase oxidation of dimethylsulfide using nitrogen oxide or concd nitric acid (Refs 1 & 2)

An expln can be produced on contact of DMSO with 70% perchloric acid as was reported by Kharasch (Ref 3). Vacuum distn of DMSO over anhyd Mg perchlorate by the method described in Ref 4, performed by a graduate student in a lab of Washington State Unit resulted in a dangerous expln, which was briefly described in Ref 6. A note in the Dec 1965 issue of the JCEduc **42**, 674 warned of the use of DMSO as a solvent for toxic compds. The wearing of synthetic rubber gloves does not protect the wearer as it was found that penetration thru the rubber was possible (See also Ref 7)

A procedure for drying DMSO with Ca hydride is described in Ref 5

Refs: 1) Beil **1**, 289, [277] & {1217} 2) CondChemDict (1961), 399-R 3) N. Kharasch, "Organic Sulfur Compounds", Pergamon Press, NY (1961) 4) M.L. Tobe & D.W. Watts, JCS **1964**, 2991–3000 5) R.E. Desay et al, JACS **86**, 28(1964)

6) Anon, C&EN **43**, No 37, p62 (Sept 1965)
7) E.A. Wynne, "Safety Corner", The Indicator, Wagner College, Staten Island, NY 10301 (March 1966), p23

N,N′-Dimethyltartramide or N,N′-Dimethyldiaminotartaric Acid and Derivatives

N,N′-Dimethyltartramide,
$H_3C.HN.OH.CH(OH).CH(OH.CO.NH.CH_3$, mw 176.17, N 15.90%; cryst s, mp 189° (Ref 3); sol in warm alc, w & pyridine; insol in cold alc; was prepd by Frankland& Slator (Ref 3) by introducing methylamine into a soln of methyl-d-tartrate in MeOH and cooling

Urbański (Ref 4) also prepd N,N′-Dimethyldiaminotartaric Acid, but its mp 213–214° did not agree with the mp of the compd prepd by Frankland & Slator. Urbanski also prepd N-Methylaminotartaric Acid and its mp was reported as 198° (Ref 1)

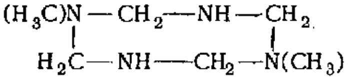
N,N′-Dimethyl-N,N′-dinitro-tartramide Dinitrate; 1,6-Dimethyl-3,4-dinitroxy-tartar-1,6-dinitramide or 1,6-Dimethyldinitramino-3,4-dinitroxy-tartaric Acid,
$H_3C.(O_2N)N.OC.CH(ONO_2).CH(ONO_2).CO.-N(NO_2).CH_3$; mw 356.17, N 23.60%, OB to CO_2 minus 18%; crysts, mp 114°; bp expl at 117° when heated at the rate of 20° per minute; sol in acet, benz, chlf, CCl_4, and alc. It can be prepd by dissolving N,N′-dimethyltartramide in acet at below 0° and adding to it concd nitric acid, followed by acetic anhydride

It is a very powerful expl with sensitivity to shock betw that of MF & LA and stable below 117°; was proposed as a component in primer mixtures

Refs: 1) Beil **4**, 79, [582] & {159} (Dimethyltartardiamide) 2) Beil **4**, {169} (Dinitrodimethyltartardiamide) 3) P. Frankland & A. Slator, JCS **83**, 1360(1903) 4) T. Urbański, RocznikiChemii **16**, 334 (1936) & CA **31**, 1770(1937)

Dimethyltetraazacycloöctane and Derivatives

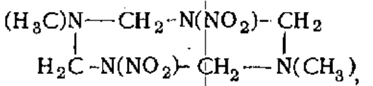
1,5-Dimethyl-1,3,5,7-tetraazacycloöctane,



$$\begin{array}{l}(H_3C)N - CH_2 - NH - CH_2 \\ \quad | \qquad\qquad\qquad\qquad\quad | \\ \quad H_2C - NH - CH_2 - N(CH_3)\end{array}$$

mw 144.22, N 38.85%; may be considered as the parent compd of its dinitro deriv, although not used to prep it:

3,7-Dinitro-1,5-dimethyl-1,3,5,7-tetraazacycloöctane,

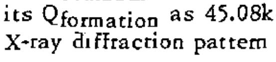


$$\begin{array}{l}(H_3C)N - CH_2 - N(NO_2) - CH_2 \\ \quad | \qquad\qquad\qquad\qquad\qquad\quad | \\ \quad H_2C - N(NO_2) - CH_2 - N(CH_3)\end{array}$$

mw 234.22, N 35.88%; ndls (from acet), mp 124° & 134–35° (Ref 6); sol in acet, alc, chlf & 10% aq NaOH; insol in eth & petr eth; was obtd by Chute et al (Ref 4) by condensing $MeNH_2$ with a formalin-nitramide soln. Chapman et al (Ref 6) prepd this compd (mp 125–26°, dec) when a soln of methylene dinitramine in ethyl acetate was saturated with dry gaseous formaldehyde at 0° and a 33% methylamine in alc was added. See also Ref 5

Refs: 1) Beil, not found (1,5-Dimethyltetraazacycloöctane) 2) Beil, not found (3,7-Dinitro-1,5-dimethyltetraazacycloöctane) 3) Beil, not found (5,7-Dinitro-1,3-dimethyltetraazacycloöctane) 4) W.J. Chute et al, CanJRes **27B**, 225 & 232–33(1949) 5) E. Aristoff et al, CanJRes **27B**, 523 & 531(1949) 6) F. Chapmán et al, JChemSoc **1949**, 1639 & CA **44**, 1412(1950)

1,5-Dimethyl-1,2,3,4-tetrazole or 1,5-Dimethyl-1H-tetrazole,

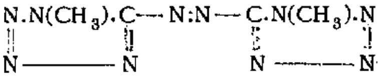


$$\begin{array}{l}(H_3C)C - N(CH_3) - N \\ \quad \| \qquad\qquad\qquad\quad \| \\ \quad N \text{———————} N\end{array}$$

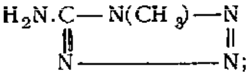
; mw 98.11, N 57.11%; crysts (from petr eth), mp 71–74°; was obtd when acetone was slowly added to a mixt of hydrazoic acid, benz & sulfuric acid (Ref 1). This compd was also prepd by Harvill et al (Ref 2). McEwan & Rigg detd its $Q_{combstn}$ as 532.19kcal/mol & calculated its $Q_{formation}$ as 45.08kcal/mol at 25°. Its X-ray diffraction pattern is also reported

Refs: 1) Beil **26**, [199] 2) E.K. Harvill et al, JOrgChem **15**, 662(1950) & CA **44**, 9441(1950) 3) W.S. McEwan & M.W. Rigg, JACS **73**, 4726(1951) & CA **46**, 4350(1952) 4) L.A. Burkardt & D.W. Moore, Anal Chem **24**, 1583(1952) & CA **47**, 2010(1953)

Di[1-methyl-tetrazolyl-5]-diazene; 1,1'-Dimethyl-5,5'-azotetrazole or **Azo-di(1-methyltetrazole-5),**



N.N(CH$_3$).C—N:N—C.N(CH$_3$).N; N———N N———N; mw 194.17, N 72.14%; orn-yel crysts, mp 182°(dec); bp puffs off with a loud report on rapid heating; easily sol in hot w; sl sol in hot alc; can be prepd by shaking and then warming on a water bath, the 1-methyl-5-amino-tetrazole,



$H_2N.C—N(CH_3)—N$; N———N; with an aq soln of Ca hypochlorite

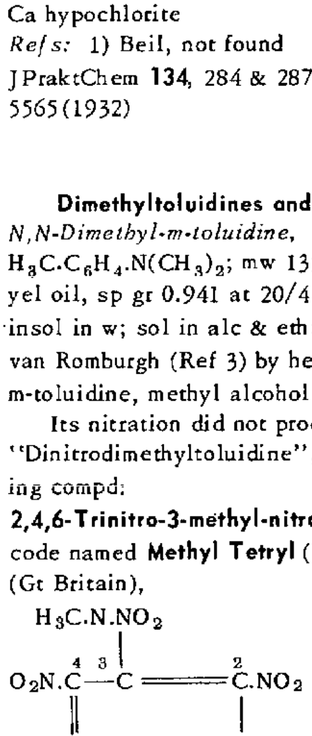
Refs: 1) Beil, not found 2) R. Stollé, JPraktChem **134**, 284 & 287(1932) & CA **26**, 5565(1932)

Dimethyltoluidines and Derivatives

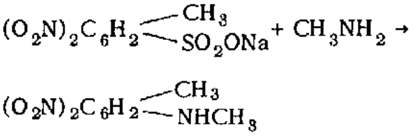
N,N-Dimethyl-m-toluidine, $H_3C.C_6H_4.N(CH_3)_2$; mw 135.20, N 10.36%; yel oil, sp gr 0.941 at 20/4°; bp 213–215°; insol in w; sol in alc & eth; was prepd by van Romburgh (Ref 3) by heating for 24 hrs m-toluidine, methyl alcohol & HCl at 200°

Its nitration did not produce the expected "Dinitrodimethyltoluidine", but the following compd:

2,4,6-Trinitro-3-methyl-nitraminotoluene, code named **Methyl Tetryl** (USA) or **MethylCE** (Gt Britain),



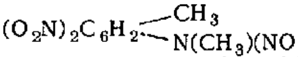
mw 301.18, N 23.26%, OB to CO$_2$ minus 61.1%; pale yel crysts, mp 100–102°; expl at higher temp. It was first obtd in 1884 by van Romburgh (Ref 3), instead of expected Dinitrodimethyltoluidine, on treating dimethyltoluidine with mixed nitric-sulfuric acid. Its structure was not established until 1902 when Blanksma (Ref 4) prepd a compd identical to van Romburgh compd from 2,4,6-Trinitro-methylaminotoluene. It was also prepd by Garner & Abernethy (Ref 5). Davis (Ref 6) and Clift & Fedoroff (Ref 7) described prepn of MeTetryl from beta- and gamma-Trinitrotoluene isomers always present as impurities on the surface of crysts of crude alpha-TNT. The β and γ isomers can be removed by washing the crysts of crude α-TNT with 8% aqueous Na sulfite soln (called *Sellite*). This converts the isomers into water-soluble dinitrotoluene sulfonates. Then, on their treatment with methylamine the following reaction takes place:



$$(O_2N)_2C_6H_2\begin{matrix}\nearrow CH_3\\ \searrow SO_2ONa\end{matrix} + CH_3NH_2 \rightarrow$$

$$(O_2N)_2C_6H_2\begin{matrix}\nearrow CH_3\\ \searrow NHCH_3\end{matrix}$$

Nitration of this product with mixed nitric-sulfuric acid gives Methyltetryl,



$$(O_2N)_2C_6H_2\begin{matrix}\nearrow CH_3\\ \searrow N(CH_3)(NO_2)\end{matrix}.$$

In experiments conducted in Canada and US during WWII, it was possible to obtain about 30 lbs of Methyltetryl from 2000 lbs of crude TNT (Refs 6, 7 & 8)

Following props for MeTetryl are listed in Blatt (Ref 8)

Heat of Combustion: 1009.3kcal/mol

Impact Sensitivity expressed as FI 67% of Picric Acid

Power by Ballistic Mortar 110% TNT and by Trauzl Test 103% PA

Refs: 1) Beil **12**, 857, (398) & [466] 2) Beil **12**, 880 & [482] 3) P. van Romburgh, Rec **3**, 414(1884) 4) J.J. Blanksma, Rec **21**, 327(1902) 5) W.E. Garner & C.L. Abernethy, ProcRoySoc **99A**, 213(1921) 6) Davis (1943), 146–47 7) Clift & Fedoroff, Vol **4**(1944), p57 8) A.H. Blatt, OSRD **2014**(1944) (2,4,6-Trinitro-3-methylnitraminotoluene)

N,N-Dimethyl-dinitro-m-toluidine, $H_3C.C_6H_2(NO_2)_2.N(CH_3)_2$; mw 225.20, N 18.66%. Three isomers are known:

N,N-Dimethyl-2,4-dinitro-m-toluidine, compd listed in CA Formula Index (1956–65), p 109F but no info is given in abstract (Ref 3)

N,N-Dimethyl-x,x-dinitro-m-toluidine(?), yel cryst granules, mp 168°; was prepd from dimethyl-m-toluidine by treating with dil nitric acid or cold nitric/sulfuric mixed acid (Ref 1, p879)

4,4-Dimethyl-4,6-dinitro-m-toluidine, yel ndls, mp 107°; sol in org solvs; can be prepd by reaction of 2,4,5-trinitrotoluene, dimethylamine chloride & concd ammonia in boiling alc (Refs 1, 2 & 3)

No expl props of dinitro derivs are reported

Refs: 1) Beil **12**, 879 & [480] 2) O.L. Brady & W.H. Gibson, JChemSoc **119**, 102 (1921) & CA **15**, 1281(1921) 3) D.S. Deorha & H.L. Sharma, JIndianChemSoc **40** (9), 819(1963) & CA **60**, 2807(1964)

N,N-Dimethyl-o-toluidine, $H_2C.C_6H_4.N(CH_3)_2$; mw 135.20, N 10.36%; liquid, sp gr 0.929 at 20/4°, fr p minus 61.3°; insol in w; sol in alc & eth; was prepd by heating for 2 days o-toluidine, methyl alcohol & HCl at 200–220°(Ref 1). Other methods of prepn & other props are given in Beil (Ref 1)

N,N-Dimethyl-dinitro-o-toluidine, $H_3C.C_6H_2(NO_2)_2.N(CH_3)_2$; mw 225.20, N 18.66%. Compd not found in the literature thru 1966

Refs: 1) Beil **12**, 785, (376) & [435]

N,N-Dimethyl-p-toluidine, $H_3C.C_6H_4.N(CH_3)_2$; mw 135.20, N 10.36%; liquid, sp gr 0.937 at 20/4°; bp 210–211°; insol in w; sol in alc & ether; was prepd by reaction of methylchloride on hot p-toluidine & methyl-p-toluidine, and by several other methods (Ref 1)

N,N-Dimethyl-dinitro-p-toluidine, $H_3C.C_6H_2(NO_2)_2.N(CH_3)_2$; mw 225.20, N 18.66%. Four isomers are known:

N,N-Dimethyl-2,3-dinitro-p-toluidine, compd listed in CA Formula Index (1947–56), p455F but not found in abstract (Ref 3)

N,N-Dimethyl-2,5-dinitro-p-toluidine, red-golden shiny pltlts (from alc), mp 103–04°; readily sol in eth, chlf, ethyl acetate, hot alc & hot petr eth; sl sol in cold alc & cold petr eth; was prepd by nitrating 2-nitro-4-dimethylaminotoluene with a soln of nitric-sulfuric acid and pouring into w (Ref 2, pp 1009 & 442)

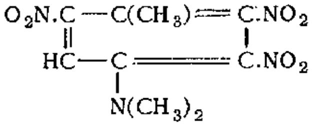
N,N-Dimethyl-2,6-dinitro-p-toluidine, yel ndls (from acet ac), mp 192°; was prepd by heating 2,6-dinitro-4-aminotoluene with dimethylsulfate at 160–65°(Ref 2, p442)

N,N-Dimethyl-3,5-dinitro-p-toluidine, scarlet-red ndls (from petr eth) or prisms (from acet ac), mp 95°; sl sol in cold alc & petr eth; otherwise readily sol in org solvs; was prepd by nitrating dimethyl-p-toluidine with 30% nitric acid & dil sulfuric acid solns (Ref 2, pp1010 & 442)

No expl props of the dinitro derivs are reported

Refs: 1) Beil **12**, 902, (413) & [491] 2) Beil **12**, 1009, 1010 & (442) 3) W. Qvist, ActaAcadAboensis, Math et Phys **19**, No 1, 3–12(1953) (Publ 1954 in Ger) & CA **49**, 8993(1955)

N,N-Dimethyl-2,4,6-trinitro-p-toluidine (called 2.3.6-Trinitro-4-dimethylamino-toluol in Ger),



mw 272.0, N 20.74%; red ndls or tablets (from alc), mp 137°; was prepd by nitrating 2,6-dinitro-4-dimethyltoluidine with nitric acid (d 1.4) & dil sulfuric acid at 95°(Refs 1 & 2). No expl props of this compd are reported

Refs: 1) Beil **12**, (445) 2) G.T. Morgan & A. Clayton, JChemSoc **99**, 1942(1911) & CA **6**, 634(1912) 3) No later refs found thru 1966

N,N-Dimethyl-tetranitro-p-toluidine, $C_9H_9N_5O_8$; not found in the literature thru 1966

N,N'-Dimethyltriazene; 1,3-Dimethyltriazene or Diazoaminomethane, $H_3C.N:N.NH.CH_3$; mw 73.10, N 57.49%; col liquid, fr p minus 12°; bp 92°(with decompn) and exploded with a sharp report when heated in a capillary tube; sol in w, imparting a sweet taste to it; was prepd by treating methylazide with methyl-magnesium iodide:

$CH_3.MgI + N_3.CH_3 \rightarrow CH_3.N{:}N.N(MgI).CH_3$

and then decomposing the resulting product with water:

$CH_3.N{:}N.N(MgI).CH_3 + H_2O \rightarrow CH_3.N{:}N.NH.CH_3$

Several salts were prepd by Dimroth (Ref 2), such as the copper, $CuC_2H_6N_3$, mp 185–86° and the silver, $AgC_2H_6N_3$, mp – decomp above 150°, which might be explosive but they were not investigated from this point of view

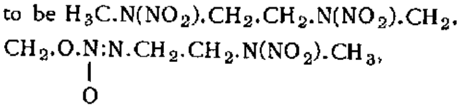
Brinkman et al (Ref 4) synthesized a series of 1,3-Dimethyltriazene-metal derivs of Cu (I), Zn, B, Al, Si, Sn, Ti, Zn & P and examined their props. Because of the expl hazards associated with these compds or with their addn complexes, heating above 110° is to be carefully avoided. Their prepn & props are described in great detail (Ref 4)

Refs: 1) Beil **4**, 578 & {1757} 2) O. Dimroth, Ber **39**, 3905–12(1906) 3) H.C. Ramsperger & J.A. Leermakers, JACS **53**, 2061–71(1931) & CA **25**, 4174(1931) (Thermal decompn studies) 4) F.E. Brinckman et al, InorgChem 4(7), 936–42(1965) & CA **63**, 3872(1965)

Di(methyltriazolyl)-formamidine. See N,N'-Bis(5-methyl-s-triazol-3-yl)-formamidine in Vol 2 of Encycl, p B151-L

Dimethyltriethylenetetramine and Derivatives

1,10 or N,N'-Dimethyltriethylenetetramine, $H_3C.NH.CH_2.CH_2.NH.CH_2.CH_2.NH.CH_2.CH_2.NH.CH_3$; mw 174.29, N 32.15%; may be considered as the parent compd of its nitrated derivs, although not used to prep them. It is a liq of bp 152° at 17mm press and n_D 1.4718 at 25°(Ref 13). Prepn is given in Ref 14

N,N'-Dimethyltriethylene-tetranitramine or **1,10-Dimethyl-1,4,7,10-tetranitro-triethylenetetramine,** $H_3C.N(NO_2).CH_2.CH_2.N(NO_2).CH_2CH_2.N(NO_2).CH_2.CH_2.N(NO_2).CH_3$; mw 354.29, N 31.63%; OB to CO_2 minus 76.8%. Crysts, mp 211.5–213°(decomp); bp – did not boil and did not expl on being heated to 360°; was prepd by Blomquist & Fiedorek (See Ref 5) by refluxing a mixture of N-methyl-ethylenedinitramine, $(CH_3)(O_2N)N.CH_2.CH_2.NH(NO_2)$ with ethylene dibromide, $Br.CH_2.CH_2.Br$ in aq alc contg KOH as described in detail in Refs 3 & 4, pp 4–5. After separating the ppt, the filtrate yielded on evaporation a solid substance melting at 88.5–90° and believed to be 

$H_3C.N(NO_2).CH_2.CH_2.N(NO_2).CH_2.CH_2.O.\underset{\underset{O}{|}}{N}{:}N.CH_2.CH_2.N(NO_2).CH_3$,

mw 354.29, N 31.63%; OB to CO_2 minus 76.8%; mp 88.5–90°, bp – exploded at 135° in 10 mins. Other props:

Hygroscopicity at 25° & RH 90% 0.02% and at RH 100% 0.36%

Impact Sensitivity, with 1 kg wt >90cm vs 48–50cm for RDX

Power by Ballistic Mortar – 109.1% (TNT=100)

Thermal Stability at 100° – not acid in 300 mins

Properties of compd melting at 211.5–213° as reported by Blomquist & Fiedorek are:

Hygroscopicity at 25° & RH 90% 0.2% and at RH 100% 0.95%

Impact Sensitivity, with 1 kg wt >90cm vs 48–50cm for RDX

Power by Ballistic Mortar – 90.4% TNT

Thermal Stability at 135° – not acidic in 300 mins

Vacuum Stability at 120° – 5.39cc/5g in 90 mins

Volatility at 75° – 0.1% loss in wt in 48 hrs

Note: When compd of mp 88.5–90° was treated with 98% nitric acid, a mixture of the following products was obtd:

1-Methyl-N'-(2-nitroxyethyl) -ethylenedinitramine, $(H_3C)N(NO_2).CH_2.CH_2.N(NO_2)-[CH_2.CH_2(ONO_2)]$; mw 253.18, N 22.64%; small glistening plates (from 50% acet), mp 90–92°; no expl props of this compd were detd (Ref 3)

1-Nitroxytrimethylene-3-nitramine or **N-(2-Nitroxyethyl)-methylnitramine;** called **2-(Methylnitramino)-ethanol Nitrate in CA,** abbr as **MeNENA,** $(O_2NO)CH_2.CH_2.N(NO_2)CH_3$; mw 165.11, N 25.45%, OB to CO_2 minus 43.6%; crysts, mp 39–40.5°. Besides obtg it by the method of Blomquist & Fiedorek outlined in Ref 3, it could be prepd by treating N-methylethanolamine, $H_3C.NH.CH_2.CH_2OH$, with nitric acid, acetic anydride and Zn chloride, as described in NDRC Div 8, IntRepts PT-5, Dec 15, 1942 to Jan 15, 1943, p26. See also other Refs on its prepn

Its properties, as given by Blatt (Ref 4) are as follows:
Explosion Test at 360° – did not expl
Impact Sensitivity by Bruceton No 3 Machine – 50% positive >90cm
Power by Ballistic Mortar 137% of TNT
Thermal Stability at 100° – not acid in 300 mins
Thermal Stability at 135° – acidic in 50 mins
Vacuum Stability at 100° – >12cc/5g in 90 min
Volatility at 75° – too great to permit detn by the International Test

Refs: 1) Beil, not found (Dimethyltriethylenetetramine 2) Beil, not found (Dimethyltriethylenetetranitramine) 3) A.T. Blomquist, OSRD **4134**(1944) & PB 18867(1944), 8–9, 68–9 & 78 4) A.H. Blatt, OSRD **2014** (1944) 5) A.T. Blomquist & F.T. Fiedorek, USP 2481283(1949) & CA **44**, 4925(1950) 6) A.T. Blomquist & F.T. Fiedorek, USP 2485855(1949) & CA **44**, 3516(1950) (Prepn & props of MeNENA) 7) W.A. Shroeder et al, AnalChem **23**, 1740(1951) & CA **46**, 5434(1952) (UV & visible absorption spectra in ethyl alcohol, MeNENA) 8) J. Sallé, Thérapic **8**, 565(1953) & CA **48**, 5369(1954) (Prepn of MeNENA & detn of its vasodilating props) 9) A.T. Blomquist & F.T. Fiedorek, USP 2678946(1954) & CA **49**, 4704(1955) (Prepn of MeNENA) 10) M. DeGroote, USP 2743253(1956) & CA **50**, 12551(1956)(Prepn of parent compd) 11) L. Médard & M. Thomas, MP **39**, 195 (1957) & CA **53**, 718(1959) (Heat of combustion of MeNENA) 12) M. DeGroote & K-T Shen, USP 2828276(1958) & CA **53**, 770(1959) Prepn of parent compd) 13) V.C. Chambers Jr & A.E. Oberth, USP 3026203(1962) & CA **57**, 1790(1962) (Prepn of parent compd) 14) Ibid, PhotSciEng **6**, 216(1962) & CA **57**, 9385(1962) [Prepn of parent compd by reduction of N,N'-ethylenebis(α-methylaminoacetamide) with $LiAlH_4$ in anhyd tetrahydrofuran]

Dimethylurea and Derivatives

N,N'-Dimethylurea; sym-Dimethylurea or *1,3-Dimethyl-urea*, $H_3C.NH.CO.NH.CH_3$; mw 72.11, N 38.85%; col prisms, sp gr 1.142, mp 106°, bp 268–70°; v sol in w & alc; practically insol in eth; can be prepd by heating urea with methylamino hydrochloride at 160–170° and by other methods (Ref 1)

Attempts by Shriner (Ref 3) to use it as an ingredient of expls and proplnts were unsuccessful and so were attempts to nitrate it

Recently, however, (Ref 4) the following nitrated compd was reported:

1,3-Dimethyl-1,3-dinitrourea,
$H_3C.N(NO_2)CO.N(NO_2).CH_3$, mw 162.11, N 34.56%; liq, cap sensitive but insensitive to shock & friction; was prepd by nitrating the parent compd below 30° with concd nitric acid or with a mixt of HNO_3 & Ac_2O

Its expl compns consist of: AN 60, NH_4ClO_4 30 & 1,3-Dimethyl-1,3-dinitrourea(I) 10% (more powerful in BM than TNT); or AN 82, I 16 & NC 2%. A colloidal plastic compn is comprised of I 31, NG 15, NC 4 & AN 50% or I 33, NC 5, AN 45 & Amm perchlorate 17%. A proplnt compn is I 43, NC 50 & Ethyl Centralite 7% (Ref 4) (Compare with N,N'-Dimethyl-N,N'-diphenylurea, called Centralite 2, described in Vol 2 of Encycl, pC137-L)

Refs: 1) Beil **4**, 65, (331), [568] & {135} (1,3-Dimethylurea) 2) Beil **4**, not found (1,3-Dimethyl-1,3-dinitrourea) 3) R.L. Shriner, OSRD **2054**(1943), pp14–15 4) M. Kurokawa, BritP 862407(1961) & CA **55**, 15933(1961) (1,3-Dimethyl-1,3-dinitrourea) 5) J.T. Hayes & W.W. Haden, JAgrFoodChem **1969**, 17(5), 1077 & CA **71**, 100870t(1969) (Prepn & nitrification of N,N'-Dimethylurea)

N,N-Dimethyl-xenylamine or **N,N-Dimethyl-p-biphenylamine.** See N,N-Dimethylaminobiphenyl in this Vol, p D1308-R

Dim Igniters. See under Dark and Dim Igniters in Vol 3 of Encycl, pD16-L

Diminution of Sensitivity of Dynamites. See under DYNAMITES in this Vol, p

Dina. Ger designation for Dinitronaphthaline. It was manufd during WWII, together with Trinitronaphthaline at Semitin Fabrk, Pardubice (Czeckoslovakia) and used in some composite expls
Ref: Fedoroff et al, PATR **2510**(1958), pp Ger 36 & 37)

DINA. Code name for Diethylol-nitramine Dinitrate or Di(nitroxyethyl)-nitramine, described under Diethylol-amine in this Vol, p D1240-R

Dinaftaliti. Russian nonpermissible expls, such as *Dinaftalit Zernënnyi* (Granular Naphthalite) listed in Vol 3 of Encycl, p C441-R, under COAL MINING EXPLOSIVES and in Gorst (1957), p104

Dinamitas. Spanish Dynamites. Nonpermissible types (Explosivos de no seguridad) are listed in Vol 3, pp C441-R & C442-L. Permissible types (Explosivos antgrisú) are listed on pp C454-R & C455-L

Dinamiti. Italian Dynamites. Nonpermissible types are listed in Vol 3, p C440-L, and permissible types on p C451-L. See also Giua, Trattato **6**(I)(1959), 338–45

Dinamity, Nepredokhranitel'nnyiye. Russian nonpermissible Dynamites are listed in Vol 3, pC441-R

Dinamity Predokhranitel'nnyiye. Rus permissible Dynamites are listed in Vol 3, pC454-R

Dinamoni 1° and S. Italian nonpermissible explosives listed in Vol 3 of Encycl, pp C439-L & C440-L, without giving their compn, except stating that accdg to Vettori they contain TNT & AN

On pC440-L is also given a Dinamon listed in Belgrano (1952), p163 as contg AN 69, $KClO_4$ 8, TNT 20 & Al 3%; its gap test value is 20mm at density 0.90g/cc, 45mm at d 1.10, 35mm at 1.20 and 30mm at 1.25

Dinamony. Russian nonpermissible explosives of which four formulations are listed in Vol 3, pC441-R and several other formulations are given in this Vol, under DYNAMMONS

Dinaphthylamine and Derivatives

Dinaphthylamine (β,β'), $C_{10}H_7.NH.C_{10}H_7$; mw 269.33, N 5.20%; lfts (from benz), mp 171–72°, bp 471°; insol in w; sl sol in hot alc; v sl sol in hot benz; prepn is given in Beil (Ref 1)

X,X,X,X-Tetranitro-β,β'-dinaphthylamine, $C_{20}H_{11}N(NO_2)_4$; mw 449.33, N 15.59%; yel cryst granules (from hot Nitrobenz), mp 285–86°; v sl sol in benz, cumol, alc & eth; sol in hot nitrobenzene; was prepd by nitrating the parent compd in acetic acid with concd nitric acid (Ref 2). No expl props are reported

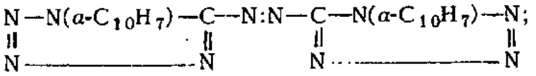
Hexanitro-β,β'-dinaphthylamine, $C_{10}H_4(NO_2)_3.NH.C_{10}H_4(NO_2)_3$; mw 539.33, N 18.18%; flakes, mp – not reported; readily sol in alc; sl sol in acet ac; insol in ether, cumol & nitrobenzene; prepd by nitrating the tetranitro deriv with an excess of fuming nitric acid (Ref 3)

No expl props are reported

Refs: 1) Beil **12**, 1278, (536) & [717] 2) Beil **12**, 1279 (Tetranitro) 3) Beil **12**, 1279 (Hexanitro)

Dinaphthylazotetrazole and Derivatives

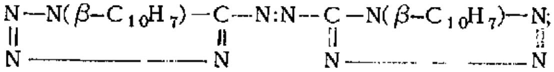
1',1-Di(α-naphthyl)-5',5-azotetrazole or Di[1-(α-naphthyl)-tetrazole-5]-diazene,



$$\begin{array}{l} N-N(\alpha\text{-}C_{10}H_7)-C-N{:}N-C-N(\alpha\text{-}C_{10}H_7)-N; \\ \| \qquad\qquad\qquad\quad \| \qquad\quad \| \qquad\qquad\qquad\quad \| \\ N\text{--------------}N \qquad N\text{--------------}N \end{array}$$

mw 418.42, N 33.48%; red ndls, mp – puffs off at ca 180°; insol in w; sl sol in acet; fairly sol in AcOH; v sl sol in alc & eth; can be prepd by warming an aqueous suspension of α-naphthyl-1-amino-5-tetrazole with a concd soln of Ca hypochlorite. It is considered as a mild explosive (Refs 3 & 4)

1′,1-Di (β-naphthyl)-5′,5-azotetrazole or Di[1-(β-naphthyl)-tetrazole-5]-diazene,



$$\begin{array}{l} N{-}N(\beta{-}C_{10}H_7){-}C{-}N{:}N{-}C{-}N(\beta{-}C_{10}H_7){-}N; \\ \| \qquad\qquad\qquad\quad \| \qquad\quad \| \qquad\qquad\qquad\quad \| \\ N{-\!-\!-\!-\!-\!-}N \qquad\qquad N{-\!-\!-\!-\!-\!-}N \end{array}$$

mw 418.42, N 33.48%; red ndls, mp – explodes at 204°; insol in w & eth; v sl sol in alc even on warming; sl sol in hot acet & AcOH; easily sol in hot benz; can be prepd by warming an aqueous suspension of β-naphthyl-1-amino-5-tetrazole with a concd soln of Ca hypochlorite. It is considered as a mild explosive (Refs 3 & 4)

Refs: 1) Beil, not found (Di-α-naphthyl.....) 2) Beil, not found (Di-β-naphthyl.....) 3) R. Stollé et al, JPraktChem **134**, 284, 307 & 308(1932) 4) F.R Benson, ChemRevs **41**, 15(1947)

Dinaphthyl-succinamide. See Bis(α-naphthyl)-succinamide in Vol 2, pB151-R

1,3-Di-1-naphthyltriazene, See Diazoamino-naphthalene in this Vol, p D1158-L

Ding-Dong. Name formerly applied to an Air Force air-to-air missile, now termed **Genie.** It utilizes a solid proplnt rocket and is capable of carrying a nuclear warhead

Ref: Glossary of Ord(1959), pp96-R & 135-R

Dinicotinic Acid and Derivatives

Dinicotinic Acid or *3,5-Pyridinedicarboxylic Acid*, $NC_5H_3(COOH)_2$; mw 167.12, N 8.38%; col crysts, mp 314°(Hackh's); 323°(decomp); bp – subl with decompn; v sl sol in w, alc or eth; sol in HCl). Prepn & other props in Beil (Ref 1)

Dinicotinic Acid Dizaide (Dinikotinsäure-diazid in Ger), $NC_5H_3(CO.N_3)_2$; mw 217.15, N 45.16%; pltlts (from eth), mp 83°, bp – explodes on rapidly heating; was obtd by reaction of the dimethyl ester of dinicotinic acid with hydrazine hydrate and treating the reaction product with K nitrite & dil HCl in the cold (Refs 2 & 3)

NOTE: No nitro derivs of Dinicotinic Acid were found in Beil nor in CA thru 1966 .

Refs: 1) Beil **22**, 160, (535) & [107] 2) Beil **22**, (535) 3) H. Meyer & H. Tropsch, Monatsh **35**, 211(1914) & CA **8**, 1575(1914)

Dinite No 1 Powder. It was a commercial explosive made in about 1920 by incorporating DNT with NC in a mixer. It is not used now

Refs: 1) G.D. Clift, PicArsn; private communication (about 1950) 2) C.S. Davis, PATR **12**(1931)

1′,3′-Di (2-nitramino-1,3-dinitroxy-propane)-2′,4′,6′-trinitrobenzene. See 1′,3-Bis-(1,3-dinitroxy-2-nitramino-propane)-2′,4′,6′-trinitrobenzene in Vol 1, pA252-L

1′,3′-Di(1-nitramino-2,3-dinitroxy-propane)-2′,4′,6′-trinitrobenzene. See 1′,3′-Bis-(2,3-dinitroxy-1-nitramino-propane)-2′,4′,6′-trinitrobenzene in Vol 1, pA252-R

Di(β-nitraminoethyl)-amine. See Bis(β-nitraminoethyl)-amine or 1,7-Dinitro-1,4,7-triazaheptane in Vol 2, pB128-R

Di(β-nitraminoethyl)-ammonium Nitrate. See under Bis(β-nitraminoethyl)-amine Nitrate in Vol 2, pB128-R

N,N′-Di(β-nitraminoethyl-nitramino)-ethane. See 1,2-Bis(2′-nitraminoethyl-nitramino)-ethane in Vol 2, pB129-R

N,N′-Di(β-nitraminoethyl)-N,N′-dinitrourea. See N,N′-Bis(β-nitraminoethyl)-N,N′-dinitrourea in Vol 2, pB130-R

1,2-Di(or Bis)(2-nitramino-2-imidazolin-1-yl)-ethane or **Bis-1(2-nitramino-2-imidazolynyl)-ethane.** See Vol 1, pA220-R under AMINOIMIDAZOLINE

2,2-Di(nitraminomethyl)-1,3-propanedinitramine. See 2,2-Bis(nitraminomethyl)-1,3-propanedinitramine in Vol 2, pB130-R

1,3-Dinitramino-2-propanol-nitrate. See under Diaminopropanol and Derivatives in this Vol, p D1143-R

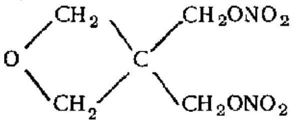
4,4′-Dinitramino-3,5,3′,5′-tetrabromo-benzaldehydedeazine. See Vol 2, pB36-L

4,6-Dinitramino-s-triazine-2-ol. See Dinitroammeline in Vol 1 of Encycl, pA274-L

1-(2,4-Dinitranilino)-benzene-4'-diazonium Nitrate. See Vol 1, pA421-R

3,3-Di(nitrotomethyl) -butene-1. See under Dimethyl-butene in this Vol, p D1331-L

3,3-Di(or Bis)-(nitratomethyl) -oxetane or **3,3-Oxetanedimethanol Dinitrate,**



mw 208.13, N 13.46%; wh cake, mp 88–90°; d 1.76, ignition temp 233°, thermal stability 1.12cc gas/g at 100° in 48 hrs; impact sensitivity 34cm with 2½ kg wt; was prepd when PETriN (Pentaerythritoltrinitrate) in abs alc was heated with KOH at 73° for 4½ hrs with stirring and the mixt cooled to 0° (Ref 2)

See also Refs 3 & 4

Refs: 1) Beil, not found 2) D.L. Kouba et al, Hercules Powd Co Monthly Progress Report (2 May 1952) (Navy Contract NOrd-11280, Task A) 3) D.E. Elrick et al, JACS **76**, 1374(1954) & CA **49**, 3811(1955) (Prepn) 4) A. Schrage, USP 3058994(1962) & CA **58**, 5638(1963) (Prepn)

1,2-Di(or Bis)-(2-nitrimino)-3-nitro-1-imidazolidyl)-ethane or **Bis-1-(2-nitrimino-3-nitroimidazolidinyl) -ethane.** See Vol 1, p A220-R under AMINOIMIDAZOLINE

DINITRO-, DINITROXY- AND DINITROSO- COMPOUNDS

Under these terms are known organic compds contg either two $-NO_2$, two $-ONO_2$ or two $-NO$ groups. As a rule, they are described in this Encyclopedia under corresponding parent compds. For example, Dinitrobenzenes are described under Benzene and Dinitrotoluenes are under Toluene. If a parent compd has no other nitrated derivatives besides di- (such as mono-, tri-, tetra-, etc — nitro- or nitroso-) or has no azido- or diazido- groups, the di- compd may be described as such in this Volume and not under its parent compd. Some dinitroxy- compds are described as dihydroxy-dinitrates

Following list gives location of explosive dinitro-, dinitroso- and dinitroxy- compounds not found in this Volume. Some non-expl compds, such as Dinitro-cumene, are included because they were proposed as ingredients of expl compositions:

Dinitroabietic Acid. See Vol 1 of Encycl, p A3-L

Dinitroacetophenones. See Vol 1, p A48-L

1,5-Dinitro-3-acetyl-1,3,5-triazine (Code name **TAX**). See Aceto-3,5-dinitro-1,3,5-triazacyclohexane in Vol 1, p A50-L

Dinitroaminobenzaldehyde. See Vol 1, pA186-L

Dinitroaminobenzamides. See Vol 1, pp A186-R & A187-L

Dinitroaminobenzene. See Dinitroanilines in Vol 1, pp A408-R & A409-L

Dinitro-1-aminobenzene-arsonic Acid. See 3,5-Dinitro-4-aminophenylarsonic Acid in Vol 1, p A245-R

Dinitroaminobenzoic Acid, See Vol 1, p A189

Dinitroaminocresols. See Vol 1, p A194

2,4-Dinitro-6-amino-3-cyano-phenol. See 2,6-Dinitro-4-amino-3-hydroxybenzonitrile in Vol 1, p A218-L

1,6-Dinitro-2-(aminoguanyl)-benzalaminoguanidinium-biguanidine benzalhydrazone. See Benzalaminoguanidinium-1,6-dinitro-2-(aminoguanyl) -biguanidine benzalhydrazone in Vol 1, p A215-L

1,6-Dinitro-2-(aminoguanyl) -biguanidine and its salts. See Vol 1, pp A214 & A215

1,6-Dinitro-2-(aminoguanyl)-biguanidine Nitrate. See Vol 1, p A214

2,6-Dinitro-4-amino-3-hydroxybenzonitrile or **2,4-Dinitro-6-amino-3-cyanophenol.** See Vol 1, p A218-L

Dinitroaminoimidazoline or **Dinitroaminodiazacyclopentane.** See 1-Nitro-2-nitramino-Δ^2-imidazoline in Vol 1, p A220-L

5,7-Dinitro-6-amino-indazole. See Vol 1, p A224-L

3,5-Dinitro-2-aminophenol. See Vol 1, p A241-R

4,6-Dinitro-2-aminophenol or **Picramic Acid.** See in Vol 1, p A241-R. Its salts are on pp A241 & A242

2,6-Dinitro-3-aminophenol. See Vol 1, p A243-L

2,3-Dinitro-4-aminophenol. See Vol 1, p A243-R

2,5-Dinitro-4-aminophenol. See Vol 1, p A243-R

2,6-Dinitro-4-aminophenol. See Vol 1, p A243-R

3,5-Dinitro-4-aminophenol. See Vol 1, p A244-L

3,5-Dinitro-4-aminophenylarsonic Acid. See Vol 1, p A245-R

2-(2',6'-Dinitro-4'-aminophenyl) -ethanol. See Vol 1, p A246-L

1,1-Dinitro-2-aminopropane; or **β,β-Dinitroisopropylamine.** See Vol 1, p A250-R

3,5-Dinitro-2-aminopyridine. See Vol 1, p A255-L

3,5-Dinitro-4-aminopyridine. See Vol 1, p A255-L

5,7-Dinitro-8-aminoquinoline. See Vol 1, p A256-L

3,6-Dinitro-4-aminoquinoline. See Vol 1, p A256-R

2,ω(or 3,ω)-Dinitro-4-amino-styrene. See Vol 1, p A257-R

Dinitroaminothiazole. See 2-Nitramino-5-nitrothiazole in Vol 1, p A263-R

Dinitroammeline. See Vol 1, pp A273-R & A274-L

Dinitroanilines. See Vol 1, pp A408-R & A409-L

2,4-Dinitroanilinoacetic Acid or **N-2,4-Dinitrophenyl)-glycine.** See Vol 1, p A420-L

1-(2,4-Dinitroanilino)-benzene-4'-diazonium Nitrate or **2',4'-Dinitrodiphenylamine-4-diazonium Nitrate.** See Vol 1, p A421-R

2-(2',4'-Dinitroaniline)-1,3-propanediol. See Vol 1, p A435-L

2,4-Dinitroanisole. See Vol 1, p A448-L

1,5-Dinitroanthraquinone-2,6-diazide. See 1,5-Dinitro-2,6-diazido-anthraquinone in Vol 1, p A460-L

Dinitroantipyrines. See Vol 1, p A472

3,3-Dinitroazetidine. See Vol 1, p A519-L

Dinitroazidobenzene or **Dinitrophenylazide.** See Azidodinitrobenzene in Vol 2, p B42-R under BENZENE AND DERIVATIVES

DINITROAZIDO COMPOUNDS are listed as Azidodinitro compds in Vol 1, pp A627-R to A643-L

Dinitroazoanisole. See Vol 1, p A646-L

Dinitroazobenzenes. See Vol 1, p A648

Dinitro-p,p'-azobenzoic Acid. See x,x'-Dinitroazobenzene-4,4'-dicarboxylic Acid in Vol 1, p A651-L

2,5-Dinitroazobisformamidine. See Azobis-(nitroformamidine) in Vol 1, p A652-L

Dinitroazonaphthalenes. See Vol 1, p A656

Dinitroazophenetole. See Vol 1, p A657-L

Dinitroazostilbenes or **Dinitro-distyrylazobenzenes.** See Vol 1, p A659

Dinitroazotoluenes or **Dinitrodimethylazobenzenes.** See Vol 1, p A661-L

Dinitroazoxyaniline. See 4,4′-Diamino-3,3′-dinitroazoxybenzene in Vol 1, p A665-R

Dinitroazoxyanisole. See Vol 1, p A665-R

Dinitroazoxybenzene. See Vol 1, p A667-L

5,5′-Dinitroazoxybenzene-3,3′-dicarboxylic Acid or **3,3′-Dinitro-5,5′-dicarboxyazoxybenzene.** See in Vol 1, p A669-L

Dinitroazoxynaphthalenes. See Vol 1, p A670

2,2′-Dinitro-2,2′-azoxypropane or **2,2′-Dinitro-2,2′-dimethylazoxyethane.** See in Vol 1, p A671-R and A672-L

Dinitroazoxytoluenes or **Dinitrodimethylazoxybenzenes.** See in Vol 1, p A672-R

Dinitrobenzaldehyde. See Vol 2, p B35-L

2,6-Dinitrobenzaldehyde-4-diazonium Chloride. See Vol 2, p B35-R

Dinitrobenzaldehyde-nitrophenylhydrazone. See Vol 2, p B37-R

2,6-Dinitrobenzaldehyde-picrylhydrazone. See 2,6-Dinitrobenzaldehyde-(2,4,6-trinitrophenyl)-hydrazone in Vol 2, p B38-L

Dinitrobenzamide. See Vol 2, p B39-L

3,5-Dinitrobenzamidine Picrate. See Vol 2, p B39-R

1,2-(or o-) Dinitrobenzene. See Vol 2, p B46-L

1,3-(or m-) Dinitrobenzene. See Vol 2, p B46-R

1,4-(or p-) Dinitrobenzene. See Vol 2, p B46-R

Dinitrobenzene, Commercial. See Vol 2, p B47-L

Dinitrobenzeneazonitronaphthol. See in Vol 2, p B54-L

3,5-Dinitro-benzeneazo-trinitromethane. See in Vol 2, p B53-R

2,4-Dinitrobenzene-1-diazonium Hydroxide. See Vol 2, p B55-R

4,6-Dinitrobenzene-2-diazo-1-oxide; 4,6-Dinitro-2-diazophenol; Diazodinitrophenol or **Dinol.** See Vol 2, p B59-L

2,6-Dinitrobenzene-4-diazo-1-oxide or **2,6-Dinitro-4-diazophenol.** See Vol 2, p B59-R

2,4-Dinitrobenzenesulfenamide. See Vol 2, p B60-R

2,4-Dinitrobenzenesulfenylchloride. See Vol 2, p B61-L

Dinitrobenzenesulfonic Acid. See Vol 2, p B62-L

Dinitrobenzidine. See Vol 2, p B64-L

Dinitrobenzidine Perchlorate. See in Vol 2, p B64-L

Dinitrobenzimidazole. See in Vol 2, p B65-R

Dinitrobenzodioxan. See in Vol 2, p B67-L

Dinitrobenzofuroxan. See Vol 2, p B68-R

Dinitrobenzoic Acids exist as six isomers. See in Vol 2, pp B71-R to B72-R

Dinitrobenzoic Acid Azide or **Dinitrobenzoyl Azide.** See in Vol 2, p B70-R

Dinitrobenzoic Acid Hydrazide or **Dinitrobenzoylhydrazide.** See in Vol 2, p B72-R

Dinitrobenzol. German and Russian name for Dinitrobenzene, described in Vol 2, pp B46-L to B47-R

This toxic explosive was used during WWII by the Germans as an extender for TNT and as a desensitizer for some expls, such as RDX. Its addition to some high melting HE's lowered their mp's, thus rendering them suitable for cast-loading [Fedoroff et al, PATR **2510**(1958), p Ger 36-R]

Dinitrobenzol was manufd in Russia before & during WWII by nitrating benzol with mixed nitric-sulfuric acid, washing the crude product with water, followed by purification with Na sulfite soln at 65°C. The yield of commercial product was 87%, mp 88–89° and sp gr 1.45–1.50. It was used quite extensively (in spite of its toxicity) either straight or in mixts with TNT, TNX, AN, PA, etc for filling shells, mines and bombs

Following mixtures contg DNB were used in Russia:

Belity: a) DNB 65 & AN 35%; b) AN 87 & DNB 13%; c) AN 80, TNX 12 & DNB 8%

K-1 Splav (K-1 Fusion): TNT 70 & TNB 30%. This was used for filling land mines made of cast iron. Although this mixt was less brisant than straight TNT, it was, nevertheless, so brisant that it broke some cast iron mines into fragments too small to be effective against personnel. The brisance of K-1 Splav was reduced by the insertion in its cast pieces of less brisant expl, such as *Schneiderite*, which consisted of AN 88 & DNN 12% [Fedoroff et al, PATR **2145**(1955), pp Rus 3, 4, 10 & 21]

Dinitrobenzonitrile. See Vol 2, p B77-L

1,1′-Di(p-nitrobenzoperoxy)-cyclohexane or **Peroxybenzoic Acid, p-Nitrocyclohexylidene Ester.** See 1,1-Bis(p-nitrobenzoyl-peroxy)-cyclohexane in Vol 2, p B135-L

2,5-Di(p-nitrobenzoperoxy)-2,5-dimethylhexane or **Peroxybenzoic Acid, p-Nitrol-1,1,4,4-tetramethyltetramethylene Ester.** See 2,5-Bis(p-nitrobenzoylperoxy)-2,5-dimethyl-hexane Ester in Vol 2, p B135-R

9,9′-Di(p-nitrobenzoperoxy)-fluorene or **Peroxybenzoic Acid, p-Nitrofluorenylidene Ester.** See 9,9′-Bis(p-nitrobenzoylperoxy)-fluorene in Vol 2, pp B135-R & B136-L

Dinitrobenzophenone. See Vol 2, p B77-R

Dinitrobenzophenyltriazole. See Vol 2, p B78-R

4,6-Dinitro-1,2-benzoquinone-2-chlorimine. See Vol 2, p B85-L

Dinitrobenzotriazole. See Vol 2, p B87-R

Dinitrobenzotrifluoride. See Vol 2, p B89-L

5,7-Dinitrobenz-oxadiazole. One of the names for Diazodinitrophenol, described in Vol 2, p B59-L as 2,6-Dinitrobenzene-2-diazo-1-oxide

Dinitrobenzoyl Azide. See Vol 2, p B70-R

Dinitrobenzoyl Chloride. See Vol 2, p B89-R

Dinitrobenzoyl Hydrazide. See Dinitrobenzoic Acid Azide in Vol 2, p B72-R

Dinitrobenzyl Alcohol. See Vol 2, p B92-L

Dinitrobenzyl Alcohol Nitrate. See Vol 2, p B92-L

Dinitrobenzylamine. See Vol 2, p B93-L

Dinitrobenzylazide. See Vol 2, pp B94-R & B95-L

Dinitrobenzylbenzoate. See Vol 2, p B95-L

Dinitrobenzylcyanide. See Vol 2, p B96-L

2,4-Dinitrobenzyl-1,4-dinitrophenyl Ether. See Vol 2, p B99-L

N,N'-Di(3-nitrobenzylideneamino)-guanidine. See N,N'-Bis(3-nitrobenzylideneamino)-guanidine in Vol 2, p B136-L

Dinitrobenzyl Nitrate. See in Vol 2, p B92-L. Its mw should be 244.13 (instead of 243.13); N 17.22% (instead of 17.28%) and OB to CO_2 −62.3% (instead of −62.5%)

2,4-Dinitrobenzyl-4-nitrophenyl Ether. See Vol 2, p B99-L

N,N'-Di(3-nitrobenzylideneamino)-guanidine. See N,N'-Bis(3-nitrobenzylideneamino)-guanidine in Vol 2, p B136-L

Dinitrobibenzyls. See Vol 2, p B110

3,7-Dinitro(bicyclo)-1,5-ethylidene-2,4,6,8-tetramethylene-1,3,5,7-tetramine; or **3,7-Dinitro-1,5-endoethylidene-1,3,5,7-tetraaza-2,4,6,8-cycloöctane;** CA name **3,7-Dinitro-9-methyl-1,3,5,7-tetra-azabicyclo [3.3.1] nonane.** See under 1,5-Endoethylidene-1,3,5,7-tetraaza-2,4,6,8-cycloöctane

3,7-Dinitro(bicyclo)-2,4,6,8,9,10-hexamethylene-1,3,5,7-tetramine or **3,7-Dinitro-1,5-endoethylene-1,3,5,7-tetraaza-2,4,6,8-cycloöctane.** CA name **3,7-Dinitro-1,3,5,7-tetraazabicyclo [3.3.2] decane** (Code name **homo-DPT**) See under 1,5-Endoethylene-1,3,5,7-tetraaza-2,4,6,8-cycloöctane

3,7-Dinitro(bicyclo)-2,4,6,8,9-pentamethylene-1,3,5,7-tetramine; Code named **DPT or DNPT** (abbr for Dinitropentamethylenetetramine); **3,7-Dinitro-1,5-endomethylene-1,3,5,7-tetraaza-2,4,6,8-cycloöctane;** CA name **3,7-Dinitro-1,3,5,7-tetraazabicyclo[3.3.2]-nonane.** See under 1,5-Endomethylene-1,3,5,7-tetraaza-2,4,6,8-cycloöctane

1,6-Dinitrobiguanidine. See Vol 2, p B115-L

1,4'-(or 1,5'-)-Dinitrobiimidazole. See Vol 2, p B115-L

3,3'-Dinitrobiphenyl-Lead Nitrate or **Di-(m-nitrophenyl) Lead Nitrate.** See Vol 2, p B123-R

Dinitrobis(ethylenediamine)-cobalt (III) Nitrates. See under Di(ethylenediamine) and Derivatives in this Volume, p D1231

N,N'-Dinitro-N,N'-bis(2,4,6-trinitrophenyl)-ethylenediamine; N,N'-Dipicrylethylenedinitramine; or **2,4,6,2',4',6'-Hexanitrodiphenylethylenediamine** (Code named **Bitetryl** or **Ditetryl** (US) and **Octyl** (British). See in Vol 2, p B131-R under Bis(anilino)-ethane and Derivatives

Dinitrobitolyl or **Dinitro-dimethylbiphenyl.** See Vol 2, p B163-R

1,5-Dinitrobiuret, Salts
1,5-Dinitrobiuret, $O_2N.NH.CO.NH.CO.NH.NO_2$, described in Vol 2, p B164-R forms salts, some of which are explosive. Several salts were prepd at PicArsn and described in two reports (Refs 3 & 4); both of which were conf at the time of publication of Vol 2. One of the salts, the *Potassium*, $K_2C_2HN_5O_5$, was mentioned in Encycl, while the *Lead dinitrobiuret*, $PbC_2HN_5O_5$, was not. This salt was prepd by Blankman and described in Ref 4. The Lead salt, shiny silvery plates, exploded violently when heated on a steel spatula with a match flame. In the 100° Heat Test, this salt exploded in the first 48 hrs

The Silver salt, $AgC_2H_3N_4O_4$, wh ndls (from w + alc) also exploded on heating (Ref 2)

Refs: 1) Beil **3**, 126 (K salt, $KC_2H_3N_4O_4$) 2) Beil **3**, 126 (Ag salt) 3) S. Helf, I. Minsky & B. Guida, PATR **1841**(1951) (Compounds of High Nitrogen Content in Propellant Compositions) 4) H.D. Blankman, PATR **1848**(1951) (Development of Improved Primers. Preparation and Study of Metal Salts of High Nitrogen Compounds)

Dinitrobromoaniline. See Vol 2, p B307-R

Dinitrobromoazobenzene. See Vol 2, p B308-L

Dinitrobromoazoxybenzene. See Vol 2, p B308-R

Dinitrobromobenzene. See Vol 2, p B309-L

Dinitrobromobenzoyl Azide. See Vol 2, p B311-L

1,1-Dinitrobromoethane. See Vol 2, p B311-R

Dinitrobromomethane. See Vol 2, p B312-R

Dinitrobromomethylaniline. See Vol 2, p B313-R

Dinitrobromophenol. See Vol 2, p B315-L

Dinitrobromoresorcinol. See Vol 2, p B316-L

Dinitrobromotoluene. See Vol 2, p B316-R

Dinitrobutane. See Vol 2, p B367-R

Dinitro-iso-butane. See Vol 2, p B368-R

1,4-Dinitro-2,3-butanediol. See Vol 2, p B369-R

Dinitrobutanol. See Vol 2, p B374-L

Dinitrobutene. See Vol 2, p B376-L

Dinitro-N-butylaniline. See Vol 2, p B378-R

Dinitrobutyric Acid. See Vol 2, p B392

Dinitrocarbanilide. See Vol 2, p C44-L

Dinitrocarbazoles. See Vol 2, pp C47-R & C48-L

Dinitrocellulose. An obsolete name for NC of N=11.11%, which corresponds to the formula $C_6H_8O_3(ONO_2)_2$, now written as $C_{24}H_{32}O_{12}(ONO_2)_8$ and sometimes called Octanitrocellulose. See Table in Vol 2, p C100-R

Dinitrocentralite 1. See Vol 2, p C135-L

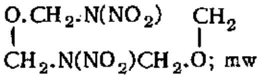
Dinitrocentralite 2. See Vol 2, p C139-L

Dinitrocresols. See Vol 3, p C557

Dinitrocumene. See Vol 3, p C574-L

Dinitrocymenes. See Vol 3, p C637-R

3,7-Dinitro-3,7-diaza-1,5-dioxacyclooctane

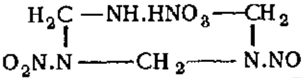


$$\begin{array}{ll} O.CH_2.N(NO_2) & CH_2 \\ | & | \\ CH_2.N(NO_2)CH_2.O & \end{array}$$

; mw 252.14, N 38.11%; crysts (from acet), mp 263–64°; sol in acet; insol in w; was isolated as one of the products of hydrolysis of hexamine; can be prepd by evaporation, in vacuo, of nitramide in formalin; and from 3,7-dinitro-1,5-endomethylene-1,3,5,7-tetraazacyclooctane, AN & nitric acid (Ref (2)

Refs: 1) Beil, not found 2) W.J. Chute et al, CanadJRes **27B**, 225 & 235–36(1949) & CA **43**, 8355(1949)

3,5-Dinitro-3,5-diazapiperidinium Nitrate (Code name **PCX**)



$$\begin{array}{lcl} H_2C & -NH.HNO_3- & CH_2 \\ | & & | \\ O_2N.N & -CH_2- & N.NO_2 \end{array}$$

; crysts, mp 98–103°; was isolated by Berman et al (Ref 4) in the nitrolysis of hexamine dinitrate with 97% nitric acid at −40° for 45 mins, and the reaction mixt diluted by adding ice. The product on treating with 70% nitric acid, heating to boiling, and diluting with water yielded Cyclonite (RDX). See also Refs 2 & 3

Refs: 1) Beil, not found 2) W.J. Chute et al, CanadJRes **27B**, 506(1949) 3) A.H. Vroom & C.A. Winkler, CanadJRes **28B**, 701(1950) 4) L. Berman et al, CanJChem **29**, 767 & 773(1951) & CA **46**, 2084(1952)

Dinitrochloroanilinopropanediols. See Vol 3, p C247-R

Dinitrochlorobenzenes. See Vol 3, p C249-R

Dinitrochlorobenzoic Acid. See Vol 3, p C252-L

Dinitrochlorobenzoic Acid Azide or **Dinitrochlorobenzoyl Azide.** See Vol 3, p C252-L

Dinitrochlorobenzonitrile. See Vol 3, p C252-R

α-Dinitrochlorohydrin. See 3-Chloropropane-1,2-diol-1,2-dinitrate in Vol 3, p C265-R

β-Dinitrochlorohydrin. See 2-Chloropropane-1,3-diol-1,2-dinitrate, Vol 3, p C266

Dinitrochloromethane. See Vol 3, p C259

Dinitrochloromethylaniline. See Vol 3, p C260-R

Dinitrochloropropane. See Vol 3, p C265-L

Dinitrochloropyridine. See Vol 3, p C267

1,5-Dinitro-2,6-diazido-anthraquinone. See Vol 1, p A460-L

4,6-Dinitro-1,3-diazido-benzene. See Vol 2, p B43-L

2',4'-Dinitro-4-diazo-diphenylamine. See 4-(2',4'-Dinitro-phenylimino)-1-diazonium-1,4-benzoquinone in Vol 2, p B82-R

Dinitrodiazophenol (Code named **Dinol**). See Vol 2, p B59-L

1,8-Dinitro-3,6-dicarbethoxy-3,6-diazaoctane. See 1,2-Bis(2'-nitroxyethyl-ethanediurethane in Vol 2, p B130-L

1,10-Dinitro-4,7-dicarbethoxy-1,4,7,10-tetrazadecane. See 1,2-Bis(2'-nitramino-ethyl) -ethanediurethane in Vol 2, p B129-R

3,3'-Dinitro-5,5'-dicarboxylazoxybenzene. See 5,5'-Dinitroazoxybenzene-3,3'-dicarboxylic Acid in Vol 1, p A669-L

1,7-Dinitrodiethylenetriamine. See Bis-(β-nitraminoethyl) -amine in Vol 2, p B128-R

Dinitrodiethylol-oxamide Dinitrate; Dinitro-diethanol-oxamide Dinitrate; or N,N'-Dinitro-di(β-nitroxyethyl) -oxamide (Code name **NENO**). See under Diethylol-oxamide and Derivatives in this Volume, p D1244-L

N,N'-Dinitro-N,N'-diethyl-oxamide. See under Diethyloxamide in this Vol, p D1246-L

N,N'-Dinitro-N,N'-diethyl-sulfamide or **N,N'-Dinitro-N,N'-diethyl-sulfonamide,** $H_5C_2.N(NO_2).SO_2.N(NO_2).C_2H_5$; mw 242.22, N 23.13%. Not found in the literature thru 1966

It evidently can be prepd by nitrating, with 95% nitric acid, its parent compd: *N,N'-Diethyl-sulfamide,* $H_5C_2.NH.SO_2.NH.C_2H_5$; mw 150.21, N 18.65%. Not found in the literature thru 1966

Nitration can be conducted by the same method as was used by Aaronson in prepn of N,N'-Dinitro-N,N'-dimethyl-sulfamide, described in this Vol under N,N'-Dimethyl-sulfamide

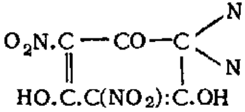
Aaronson recommended prepn of N,N'-Dinitro-N,N'-diethyl-sulfamide, expecting it to be less volatile and less sensitive than its dimethyl homologue and for this reason more suitable for military purposes

Refs: 1) Beil, not found (Parent) 2) Beil, not found (Dinitro) 3) H. Aaronson, PATR **1374**(1943), p 3

Dinitrodiglycol. Same as Diethyleneglycol-dinitrate, described under Diethyleneglycol

4,6-Dinitro-1,3-dihydrazinobenzene. See 1,3-Bis(hydrazino)-4,6-dinitrobenzene in Vol 2, p B143-R

4,6-Dinitro-3,5-dihydroxy-2-azo-benzoquinone (called by von Herz *Dinitro-azo-dihydroxy-quinonanhydrid),*



$$\begin{array}{l} O_2N.C{-}CO{-}C\langle^{N}_{N} \\ \quad\;\;\| \qquad\qquad | \\ HO.C.C(NO_2){:}C.OH \end{array}$$

; mw 242.11, N 23.14%. This compd and its expl salts were claimed to be prepd by von Herz and recommended for use in initiators. It can be prepd by treating mono- or di-nitroaminophloroglucinol with hot

strong sulfuric-nitric acid (Ref 2)
Refs: 1) Beil, not found 2) E. von Herz, BritP 207563(1923) & CA **18**, 1573–74(1924)

9,9′-Dinitro-9,9′-dihydroxy-10,10′-dihydroanthracene. See Bis(nitrohydroanthranol) in Vol 2, p B144-L

N,N′-Dinitro-N,N′-dihydroxyethyl-oxamide Dinitrate. See Dinitrodiethylol-oxamide Dinitrate under Diethylol-oxamide in this Volume, p D1244-L

3,6-Dinitro-2,5-dihydroxyquinone Diazide. See under Dihydroxybenzoquinone and Derivatives

1,4-Dinitro-3,6-diketopiperazine. See under Diketopiperazine in this Vol

Dinitrodimethylaniline. See under Dimethylaniline in this Vol

2,2′-Dinitro-2,2′-dimethylazoxyethane. See 2,2′-Dinitro-2,2′-azoxypropane in Vol 1, p A671-R

Dinitrodimethylbiphenyl. See Dinitrobitolyl in Vol 2, p B163-R

Dinitrodimethylbutane. See under Dimethylbutane in this Vol

Dinitrodimethyl-diketopiperazine. See under Dimethyl-diketopiperazine in this Vol

Dinitrodimethylglycoluril. See under Dimethylglycoluril p D1340-R

Dinitrodimethylhydantoin. See under Dimethylhydantoin in this Vol

Dinitrodimethylolbenzene. See under Dimethylolbenzene in this Vol

Dinitrodimethylol-oxamide or **Dinitroxydimethyl-oxamide.** See under Dimethylol-oxamide in this Vol

N,N′-Dinitrodimethyl-oxamide or **Dimethyl-dinitrooxamide** (Code name **MNO**). See under Dimethyloxamide in this Vol

4,6-Dinitro-2,5-dimethylphenyl-3-nitramine. See 3,5-Dinitro-2-nitramino-p-xylene in Vol 1, p A272-R

Dinitrodimethylpropane. See under Dimethylpropane in this Vol

Dinitrodimethylsulfamide. See under Dimethylsulfamide in this Vol

Dinitrodimethylsulfonamide. See under Dimethylsulfonamide in this Vol

Dinitrodimethyltartaramide Dinitrate. See under Dimethyltartaramide in this Vol

N,N′-Dinitro-N,N′-dimethyl-3,5,3′,5′-tetranitrobenzidine. See Vol 2, p B64-R

1,3-Dinitro-5,7-dimethyl-1,3,5,7-tetrazacyclooöctane. See under Dimethyl-tetrazacyclooöctane in this Vol

Dinitrodimethylurea. See under Dimethylurea in this Vol

Dinitro-dinitrosobenzenes. See Vol 2, p B45-R

Dinitro-di(β-nitroxyethyl)-oxamide (Code name **NENO**). See under Diethylol-oxamide in this Vol

Dinitro-di(β-nitroxyethyl)-sulfamide. See under Diethylol-sulfamide in this Vol

1,4-Dinitro-2,5-dioxa-3,6-dimethyl-piperazine. See 1,4-Dinitro-3,6-dimethyl-1,5-diketopiperazine under Dimethyldiketopiperazine in this Vol

Dinitrodiphenylamine. See under Diphenylamine in this Vol

2,4-Dinitro-diphenylamino-4-carboxyl Azide. See 4-Anilino-3,5-dinitrobenzoylazide in Vol 1, p A422-L

2′,4′-Dinitrodiphenylamino-4-diazonium Nitrate. See 1-(2,4-Dinitroanilino)-benzene-4-diazonium Nitrate in Vol 1, p A421-R

N,N′-Dinitro-N,N′-dipropyl-3,5,3′,5′-tetranitrobenzidine. See Vol 2, p B64-R

3,7-Dinitro-1,5-endoethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane. See under 1,5-Endoethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane (Code named **homo-DPT**)

3,7-Dinitro-1,5-endoethylidene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane. See under 1,5-Endoethylidene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane

3,7-Dinitro-1,5-endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane. See under 1,5-Endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane (Code named **DPT**)

Dinitroethane. It is described under Ethane

Dinitroethyleneglycol or **Nitroglycol (NGc).** See under Ethyleneglycol and Derivatives

Dinitroethyleneurea. See under Ethylene Urea and Derivatives

Dinitroethylolamine. See 2-Amino-2,2-dinitroethanol in Vol 1, p A201-R

Dinitroethyloxamide. See under Ethyloxamide and Derivatives

Dinitroformin or **Formyl-dinitroglycerin.** See under Formin or Formic Acid Esters

Dinitrofuran. See under Furan and Derivatives

Dinitrogen Pentoxide, N_2O_5
Dinitrogen Tetroxide, N_2O_4 } Described under NITROGEN OXIDES
Dinitrogen Trioxide, N_2O_3

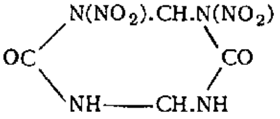
Dinitroglycerin or Glycerol Dinitrate and Trinitroglycerin or Nitroglycerin (NG). It is described under GLYCERIN AND DERIVATIVES

Dinitroglycerin Picrate or **Glycerin-2,4,6-trinitrophenylether Dinitrate.** See under Glycerin and Derivatives

Dinitroglycol; Ethyleneglycoldinitrate; or **Nitroglycol (NGc).** See under Ethyleneglycol and Derivatives

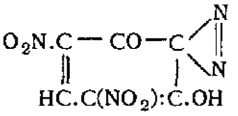
Dinitroglycoluril. Two isomers were described in Vol 1, p A65-R & A66-L, under Acetylenediurein

Blatt (Ref) described a **Dinitroglycoluril** of the probable formula



as a white solid which darkened at 305° without melting; its Ballistic Mortar Value is 101%; Sensitivity to Impact – comparable to PETN and Vacuum Stability – unsatisfactory. It was prepd by nitrating glycoluril with 98% nitric acid in the presence of acetic anhydride at a temp below 5°
Ref: A.H. Blatt, OSRD Rept **2014**(1944)

4,6-Dinitro-3-hydroxy-2-diazo-benzo-quinone, called by von Herz *Dinitroresorcindiazoanhydrid,*

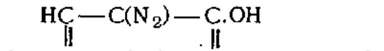


mw 226.11, N 24.78%; yel ndls; obtd as free diazonium compd by treating the K salt with concd sulfuric acid (Ref 1)

Its **Potassium Salt,** $C_6H(OK)(NO_2)_2(:N)(:O)$, yel ndls (from concd aq soln) was prepd by von Herz on treating with an excess of K nitrite the mononitroamino-resorcinol, $H_2N.C_6H_3(OH)_2$, dissolved in hot concd nitric-sulfuric acid. After prolonged boiling, the soln was cooled and this rendered fine, yel ndls of K salt. The crysts were washed with a small amt of cold water and dried. The salt was patented for use as a primary charge in compd detonators. For example a detonator contg 0.4g of PETN as a base charge required only 0.015g of K salt

Refs: 1) Beil **16**, 535 [The parent compd called in Ger 4.6-Dinitro-2-diazo-resorcin or 4.6-Dinitro-3-oxy-o-chinon-diazid-(2)] 2) E. von Herz, BritP 207563(1923) & CA **18**, 1573–74(1924)

3,5-Dinitro-2-hydroxy-1-diazo-benzo-quinone [called 2.6-Dinitro-4-diazo-resorcin; 3,5-Dinitro-2-oxy-p-chinon-diazid-(1); or 3.5-Dinitro-4-oxy-o-chinon-diazid-(1) in Ger],



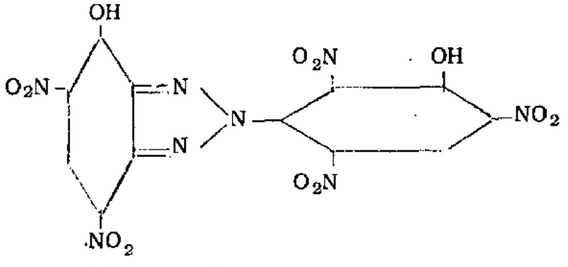
$$\begin{array}{l} HC—C(N_2)—C.OH \\ \|\qquad\qquad\quad\ \| \\ O_2N.C—C(:O)—C.NO_2 \end{array}$$

; mw 226.11, N 24.78%; golden yel pltlts, very expl compd; was obtd by dissolving 2,3,6-trinitro-4-diazophenol (2,3,5-trinitro-p-quinone-1-diazide) in boiling NaOAc, collecting and washing the orange Na salt, dissolving in water, and acidifying with aq HCl. The product combines, when moist, with β-naphthol (Refs 1 & 2)

Refs: 1) Beil **16**, 536 2) R. Meldola & J.G. Hay, JChemSoc **95**, 1384(1909) & CA **3**, 2971(1909)

3,5-Dinitro-2-hydroxy-1-methyl-benzene. See under Hydroxytoluene and Derivatives

5,7-Dinitro-4-hydroxy-2(2,4,6-trinitro-3-hydroxyphenyl)-benztriazole, called in German *5.7-Dinitro-4-oxy-2[2.4.6-trinitro-3-oxyphenyl]-benztriazol,*

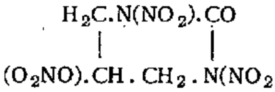


mw 452.22, N 24.78%; brn-amorphous powd, mp 176–180°(dec), bp – explodes at 275°; readily sol in alc, acet ac & acet; sl sol in hot w; almost insol in benz; was prepd by treating the Na salt of azopicric acid with NaSH in water (Refs 1 & 2)

Refs: 1) Beil **26**, [61] 2) K. Elbs & O.H. Schaaf, JPraktChem [2] **120**, 17(1929) & CA **22**, 4508(1928)

β,β-Dinitro-*iso*-propylamine. See **1,1-**Dinitro-2-aminopropane in Vol 1, p A250-R

1,3-Dinitro-2-keto-5-hydroxyhexahydropyrimidine Nitrate,



$$\begin{array}{l} \qquad H_2C.N(NO_2).CO \\ \qquad\quad\ |\qquad\qquad | \\ (O_2NO).CH.CH_2.N(NO_2) \end{array}$$

; mw 251.12, N 27.89%; OB to CO_2 minus 15.9%; solid; mp 112–113°; was prepd by nitrating with fuming nitric acid in the presence of acetic anhydride at below 15°, the product obtd by condensing (on heating) urea, $CO(NH_2)_2$, with 1,3-diamino-2-propanol, $H_2N.CH_2.CH(OH).CH_2.NH_2$. It is a very powerful expl (Trauzl Test Value = 135% PA), comparable in Impact Sensitivity to Tetryl (FI Value = 60% PA), but of unsatisfactory Thermal Stability (99° Heat Test 3 minutes; 100° Vacuum Test – more than 15cc in less than 16 hrs). It is hydrolyzed by water. See Refs 2 & 3

Refs: 1) A.H. Blatt, OSRD Rept **2014**(1944) 2) A.F. McKay & D.F. Manchester, JACS **71**, 1972(1949) & CA **43**, 9065(1949) 3) A.F. McKay et al, JACS **72**, 3205(1950) & CA **44**, 10704(1950)

Dinitromethane. It is described under Methane and Derivatives

Dinitromethoxyethoxyquinol-Nitric Acid, Potassium Salt of (Dinitromethoxyäthoxychinolnitrosäures Kalium in German), $C_9H_{10}N_3O_8K$ (its probable structural formula is given in Ref 1, p 290, lines 12 & 13); mw 327.29, N 12.84%; OB to CO_2 and K_2O minus 75.77%; red ndls; mp – not given. It was prepd by the action of ethyl ester of Picric Acid on K methylate (Refs 1 & 3)

It was proposed by Saigger (Ref 4) as a flash-reducing agent by placing it in propellant charges at the base of projectiles

This compd resembles some of the red-colored substances described by Jackson et al in Ref 2

Refs: 1) Beil **6**, 290 2) C.L. Jackson et

al, AmChemJ **20**, 444–45(1898); Ibid **23**, 376–96(1900); Ibid **29**, 89–120(1903)
3) J. Meisenheimer, Ann **323**, 242–44(1902)
4) E. Saigger (to Westfälisch-Anhaltische Sprengstoff-AG), GerP 636977(1937) & CA **31**, 3280–81(1937)

Di(3-nitro-5-methyl-4-hydroxyphenyl)-dimethylmethane or **Di(nitromethyl-hydroxyphenyl)-propane.** See 2,2-Bis(3-nitro-5-methyl-4-hydroxyphenyl)-propane in Vol 2, p B152-L

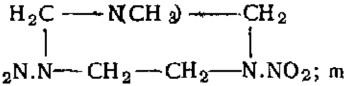
3,5-Dinitro-4-methyl-2-nitrimino-4-thiazoline. CA's name for compd listed in Vol 1, p A235-R as 3,5-Dinitro-4-methyl-2-nitrimino-Δ^4-thiazoline

Dinitromethyloxamide. It is described under Methyloxamide and Derivatives

1,2-Dinitro-2-methyl-propane. This compd was described in Vol 2, p B368-R as **Dinitro-isobutane**, but its expl props, such as given by A.H. Blatt in OSRD Rept **2014**(1944), were not included, because the rept was classified confidential at the time of publication of Vol 2 of Encycl. As the rept is now declassified, the following props can be reported:
Abel Heat Test at 82.2° – 4 mins
Gelatinizing Power for NC – molten material gelatinizes NC rapidly
Initiation, Sensitivity to. It is incomplete when primed with No 6 cap with Tetryl base
Power by Ballistic Mortar – 51% Blasting Gelatin
Power by Trauzl Test 250cc vs 300 for TNT (ca 83% TNT)
Thermal Stability at 120° – no brown fumes evolved after 5 hrs
Vacuum Stability at 100° – 2.05cc evolved in 40 hrs

3,7-Dinitro-9-methyl-1,3,5,7-tetraazabicyclo[3.3.1]-nonane. CA name for 3,7-Dinitro-1,5-endoethylene-1,3,5,7-tetraaza-2,4,6,8-cycloöctane described under 1,5-Endoethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane

3,6-Dinitro-1-methyl-1,3,6-triazacycloheptane (called 1,5-Dinitro-3-methyl-hexahydro-1-H-1,3,5-triazepine in CA Formula Index 1947–56, p 132F)



$$\begin{array}{l} H_2C - N(CH_3) - CH_2 \\ \quad\ | \qquad\qquad\qquad\quad | \\ O_2N.N - CH_2 - CH_2 - N.NO_2 \end{array}$$

; mw 205.18, N 34.14%; crysts, mp 159°; was prepd when ethylenedinitramine, $(CH_2NHNO_2)_2$, in 37% formalin was treated dropwise at 0° with 30% aq methylamine soln (Refs 1 & 2). An HCl salt, mp 138–40° was also prepd

Its UV absorption spectrum was detd by Jones & Thorn (Ref 2)
Refs: 1) G.S. Myers & G.F. Wright, CanJRes **27B**, 489(1949) & CA **43**, 9074(1949)
2) R.N. Jones & G.D. Thorn, CanJRes **27B**, 828(1949) & CA **44**, 2848(1950)

2-(2′,4′-Dinitronaphthylnitramino)-1,3-dinitroxypropane or **2-(2′,4′-Dinitronaphthylnitramino)-1,3-propanediol Dinitrate.** See Vol 1, p A252-L

3-(2′,4′-Dinitronaphthylnitramino)-1,2-dinitroxypropane or **3-(2′,4′-Dinitronaphthylnitramino)-1,2-propanediol Dinitrate.** See Vol 1, p A252-R
Note: This compd was erroneously listed as 3-(2′,4′-Dinitronaphthylnitramino)-1,3-propanediol Dinitrate

Dinitronitraminoanthraquinones. See Vol 1, p A183-R

Dinitronitraminobenzoic Acids. See Vol 1, p A189-R

Dinitronitraminotoluenes. See Vol 1, p A265-R

Dinitronitraminoxylenes. See Vol 1, p A272-R

3,5-Dinitro-4-(3′-nitrobenzamido)-phenol. See Vol 2, p B40-L

3,5-Dinitro-5-nitroso-anisole. See Vol 1, p A450-L

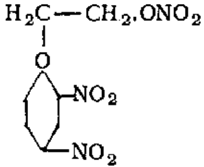
Dinitro-nitroso-benzene. See Vol 2, pp B44-R & B45-L

2,4-Dinitro-6-nitroso-benzoic Acid. See Vol 2, p B72-R

Dinitro-nitroso-bromobenzene. See Vol 2, p B309-R

Dinitro-N-nitroso-chloromethylaniline. See Vol 3, p C261-L

1-(2′,4′-Dinitrophenoxy)-2-nitroxy-ethylol or **β-(2,4-Dinitrophenoxy)-ethyl Nitrate,**



; mw 273.16, N 15.38%; crysts, mp 70°, dec at 145° & deflgr at 300°; was prepd by action of anhyd nitric acid on 2,4-dinitrophenoxyethanol (Ref 2). Médard & Thomas (Ref 3) detd its heat of combustion at const vol as 927.4kcal/mol & heat of formation 64.9kcal/mol; Tavernier & Lamouroux (Ref 4) reported values of 930.1 & 62.2kcal/g, respectively. A patent was issued (Ref 5) for the prepn of this compd by nitrating 2-(2,4-dinitrophenoxy) ethyl nitrate with concd sulfuric acid & 60% nitric acid at −10°

Blanksma & Fohr (Ref 2) reported prepn of the 2,4,6-trinitro deriv, mp 104°, bitter taste, by nitrating the dinitro compd with anhyd nitric acid & sulfuric acid at 0° for 30 mins

Refs: 1) Beil, not found 2) J.J. Blanksma & P.G. Fohr, RecTravChim **65**, 711(1946) (Engl) & CA **41**, 5484(1947) 3) L. Médard & M. Thomas, MP **35**, 172(1953) & CA **49**, 11284(1955) 4) P. Tavernier & M. Lamouroux, MP **39**, 354(1957) & CA **54**, 21974(1960) 5) S.W. Tinsley & J.T. Fitzpatrick, USP 3037057(1962) & CA **57**, 12382(1962)

2,6-Dinitrophenylamine-4-diazonium Nitrate. See 1-Anilino-2,6-dinitrobenzene-4-diazonium Nitrate in Vol 1, p A421-L

2′,4′-Dinitrophenylamine-4-diazonium Nitrate. See 1-(2,4-Dinitroanilino)-benzene-4-diazonium Nitrate in Vol 1, p A421-R

2-(2′,4′-Dinitrophenylamino)-1,3-dihydroxy-propane. See 2-(2′,4′-Dinitroanilino)-1,3-propanediol in Vol 1, p A435-L

2,4-Dinitro-phenylamino-ethylol. See 2-(2′,4′-Dinitroanilino)-ethanol in Vol 1, p A424-R

2,4-Dinitrophenyl-5-aminotetrazole. See under Phenyl-aminotetrazole

1,8-Di(4-nitrophenylazo)-2,7-dihydroxy-naphthalene. See 1,8-Bis(4-nitrophenylazo)-2,7-dihydroxy-naphthalene in Vol 2, p B153-L

N,N′-Di(4-nitrophenylazo)-glycine. See N,N′-Bis(4-nitrophenylazo)-glycine in Vol 2, p B153-L

2,4-Di(4-nitrophenylazo)-5-nitro-1-naphthol. See 2,4-Bis(4-nitrophenylazo)-5-nitro-1-naphthol in Vol 2, p B153-R

4,6-Dinitrophenyl-1,3-di(β-hydroxyethyl)-ether. One of the names for 1,3-Bis(2-hydroxyethoxy)-4,6-dinitrobenzene, described in Vol 2, p B145-R

2-(3′,5′-Dinitrophenyl)-1,3-dinitroxy-2-nitro-propane or **2-(3′,5′-Dinitrophenyl)-2-nitro-1,3-propanediol-dinitrate.** See under Phenyl-dihydroxypropane or Phenyl-propanediol and Derivatives

2,2′-(4,6-Dinitro-m-phenylenedioxy)-ethanol or **4,6-Dinitroresorcinol-bis(2-hydroxyethyl)-ether.** CA's names for 1,3-Bis(2-hydroxy-ethoxy)-4,6-dinitrobenzene described in Vol 2, p B145-R

N-(2,4-Dinitrophenyl) -glycine. See 2,4-Dinitroanilinoacetic Acid in Vol 1, p A420-L

2,4-Dinitrophenyl-hydrazine. See under Phenyl-hydrazine and Derivatives

4-(2′,4′-Dinitro-phenylimino)-1-diazonium-1,4-benzoquinone or **2,4-Dinitrophenyl-p-phenylenediazoimine.** See Vol 2, p B82-R

α-(2,4-Dinitrophenyl)-β-nitroethylol or **1-(2′,4′-Dinitrophenyl) -2-nitroethylol.** See under Phenyl-ethylol and Derivatives

3,5-Dinitrophenyl-nitromethane. See under Phenyl-methane and Derivatives

1-(2′,4′-Dinitrophenyl) -1-nitroxy-2-nitroethylol or **α-(2,4-Dinitrophenyl)-β-nitroethylnitrate.** See under Phenyl-ethylol or Phenylhydroxyethyl and Derivatives

Dinitropiperazine. See under Piperazine and Derivatives

Dinitropropanes. See under Propanes and Derivatives

Dinitropropylols. See under Propylols and Derivatives

2,2-Dinitropropyl-4,4,4-trinitrobutyrate (DNPTB), $H_3C.C(NO_2)_2.CH_2.O.CO.CH_2.CH.C(NO_2)_3$; mw 355.18, N 19.72%; OB to CO_2 minus 29%; wh crysts, sp gr 1.68; mp 11° (Form I), 95°(Form II) and 59°(Form III); bp exp at 300° in 5 secs. Three distinct modifications of DNPTB have been observed. Form I crystallizes from soln in carbon tetrachloride, chlf, acet, chlf-hexane, acet-w or methanol-w at RT. Prolonged standing of Form I at RT under the mother liquor promotes a transition to Form II. Under solidification of molten DNPTB, Form II is always observed. Form III can form from I. Both Forms I and III gave very erratic sensitivity values

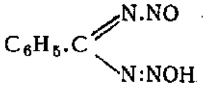
DNPTB was prepd by Hill (Refs 2 & 5) by slowly mixing dinitropropanol, $CH_3C(NO_2)_2CH_2OH$, trinitrobutyryl chloride, $Cl.CO.CH_2.CH_2.C(NO_2)_3$ and Al chloride in carbon tetrachloride at 60° and refluxing the mixture at 75° for 2 hrs. After the completion of reaction, the slurry was cooled and the crystalline product purified. It had a mp of 95° to 96°

DNPTB is one of the Navy's experimental expls. Its deton velocity was 7600m/sec and vacuum stability value 0.5ml at 100° in 40 hrs (Ref 5) (See also Refs 3, 4, & 6–9)

Refs: 1) Beil, not found 2) M.E. Hill, NAVORD Rept **2497**(1952) 3) W.B. Hewson, Hercules Rept on High Explosives, Navy Contract NOrd **11280**, Task A, 18 Oct,1954 4) J.R. Holden & J. Wenograd, NAVORD Rept **4427**(1956) 5) W.R. Tomlinson & O.E. Sheffield, PATR **1740**(1958); later edition **AMCP** (Army Materiel Command) **706–177**, Washington, DC, March 1967 6) D.J. Glover, Tetrahedron **19**, Suppl 1, 219(1963) & CA **59**, 13358(1963) (Spectrophotometric method for quantitative analysis of) 7) A. Solomonovici & S. Blumberg, IsraelJChem **3** (2) 63(1965 (Eng) & CA **63**, 6851(1965) (Prepn by Michael reaction of gem-dinitroalkanes & nitroform) 8) M.E. Hill, USP 3223725(1965) & CA **64**, 6570(1966) (Prepn with Friedel-Crafts catalyst) 9) M.E. Hill, USP 3230247(1966) & CA **64**, 9598(1966) (Prepn by transesterification)

4,6-Dinitroresorcinol-bis(or di)(2-hydroxyethyl)-ether. See 1,3-Bis(2-hydroxyethoxy)-4,6-dinitrobenzene in Vol 2, p B145-R

Dinitrosobenzamidine; Benzenyldioxytetrazolic Acid or **Dinitrosobenzenylamidine,** $C_6H_5.C(:N.NO).NH(NO)$ or



$$C_6H_5.C\begin{matrix}\nearrow N.NO \\ \searrow N{:}NOH\end{matrix}$$

mw 178.16, N 31.47%; OB to CO_2 minus 134.6%. It is an unstable expl compd prepd by treating its K salt with sulfuric acid and extracting with amyl alcohol. Its stable K salt can be prepd by treating an aq sol of benzamidine (benzenylamidine) hydrochloride, $C_6H_5.C(:NH).NH_2.HCl$, (described in Beil **9**, 280) with an aq soln of K nitrite, plus HCl of sp gr 1.2. The resulting substance was dissolved in alcohol and treated with K hydroxide and K acetate. The *Potassium Salt*, $KC_7H_5N_2O$, was recrystallized in the form of ndls or lflts, which could be exploded by heating or friction. It was patented by Rathsburg (Ref 3) as a primary ingredient in detonators

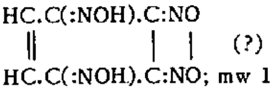
Other expl salts patented for the same purpose:

Ammonium Salt, $NH_4C_7H_5N_4O_2$; prisms deflagrating at 137°
Benzamidine Salt, $C_7H_8N_2+C_7H_6N_4O_2$; lfts (from alc); prisms (from w) deflagrating at 178° on rapid heating
Silver Salt, $AgC_7H_5N_4O_2$; solid, extremely sensitive to impact, friction and heat
Note: Salts of dinitrobenzamidine were prepd as early as 1899 by Lossen & Mierav (Ref 2), but the parent substance was not isolated until later
Refs: 1) Beil **9**, 331 2) W. Lossen & (?) Mierav Ber **32**, 1250(1899) 3) H. Rathsburg, BritP 185555(1923) & CA **17**, 1147 (1923) 4) A.H. Blatt, OSRD **2014**(1944)

Dinitrosobenzene. See Vol 2, pp B44-R & B45-L

Dinitrosobenzenylamidine. See Dinitrosobenzamidine in this Vol

2,3-Dinitrosobenzoquinone-1,4-dioxime.



$$\begin{array}{l} HC.C(:NOH).C:NO \\ \;\| \qquad\qquad\; | \;\; | \quad (?) \\ HC.C(:NOH).C:NO \end{array}$$

; mw 196.12, N 28.57%; dk yel ndls (from alc), mp 68°; sol in alkalies, which soln on exposure to air is oxidized to 1,2,3,4-tetranitrosobenzene; was prepd by adding dropwise nitric acid (d 1.40) to aq diquinoyl tetraoxime soln (Refs 1 & 2). No expl props are reported
Refs: 1) Beil **7**, 886 2) R. Nietzki & (?) Geese, Ber **32**, 506(1899) 3) No later refs found thru 1966
NOTE: See also Benzoquinone-dioxime and Derivatives in Vol 2 of Encycl, pp B83–B84

3,7-Dinitroso(bicyclo)-2,4,6,8,9-pentamethylene-1,3,5,7-tetramine; or **3,7-Dinitroso-1,5-endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane; 3,7-Dinitroso-1,3,5,7-tetraazabicyclo[3.3.1]nonane** (CA name). See under 1,5-Endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane and Derivatives

Dinitrosochloromethylaniline. See Vol 3, p C260-L

3,7-Dinitroso-1,5-endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane. See under 1,5-Endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane and Derivatives

3,7-Dinitrosopentamethylene-1,3,5,7-tetramine. See 3,7-Dinitroso-1,5-endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane under 1,5-Endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane and Derivatives

3,7-Dinitroso-1,3,5,7-tetraazabicyclo[3.3.2]-nonane. CA name for 3,7-Dinitroso-1,5-endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane, described under 1,5-Endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane and Derivatives

3,7-Dinitro-1,3,5,7-tetraazabicyclo[3.3.2]-decane. CA name for 3,7-Dinitro-1,5-endoethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane, described under 1,5-Endoethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane and Derivatives

3,7-Dinitro-1,3,5,7-tetraazabicyclo[3.3.1]-nonane. CA name for 3,7-Dinitro-1,5-endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane, described under 1,5-Endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane and Derivatives

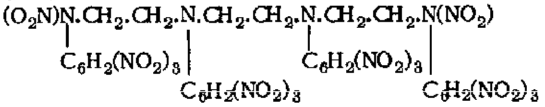
1,4-Dinitro-1,1,4,4-tetramethylolbutane Tetranitrate,
$[(O_2NO)CH_2]_2C(NO_2).CH_2.CH_2C(NO_2)\text{-}[CH_2(ONO_2)]_2$; mw 448.22, N 18.75%; solid, mp 131.5–134°; bp – explodes at higher temp; can be prepd by nitration of product obtd by condensation of 1,4-dinitrobutane with formaldehyde

It is an expl comparable in power and sensitivity to PETN
Refs: 1) Beil, not found 2) BurMinesHighExplResDivRept **No 25**, June 9, 1942
3) H.B. Haas, OSRD Rept **2016**(1943)
4) A.H. Blatt, OSRD **2014**(1944)

1,5-Dinitro-1,1,5,5-tetramethylolpentane Tetranitrate, $[(O_2NO)CH_2]_2C(NO_2).CH_2.CH_2.CH_2.C(NO_2)[CH_2(ONO_2)]_2$, mw 462.25, N 18.18%; solid, mp 84–85°; bp – expl at higher temp; can be prepd by nitration of product obtd by condensation of 1,5-dinitropentane with formaldehyde

It is an explosive more powerful than Tetryl and more sensitive than TNT
Refs: 1) Beil, not found 2) BurMines, HighExplsDivRept **No 25**, June 9, 1942
3) H.B. Haas, OSRD Rept **2016**(1943)
4) A.H. Blatt, OSRD **2014**(1944)

1,10-Dinitro-1,4,7,10-tetrapicrylhexamethylene-1,4,7,10-tetramine or **α,ω-Dinitrotetrapicryltriethylenetetramine;**



$(O_2N)\underset{C_6H_2(NO_2)_3}{N}.CH_2.CH_2.\underset{C_6H_2(NO_2)_3}{N}.CH_2.CH_2.\underset{C_6H_2(NO_2)_3}{N}.CH_2.CH_2.\underset{C_6H_2(NO_2)_3}{N}(NO_2)$

mw 1080.64, N 23.33%, OB to CO_2 minus 62.2%; solid, mp 159°(dec); bp – expl at higher temp; can be prepd by nitration of product obtd on condensation of triethylenetetramine with Picryl Chloride

It is an explosive comparable to Tetryl in power and in impact sensitivity
Refs: 1) Beil, not found 2) Canadian Explosive Project **CE-12**, covering the period from April 1 to June 1, 1942, Univ of Toronto, sponsored by the CNRC (Canadian National Research Council) 3) A.H. Blatt, OSRD **2014**(1944)

1,7-Dinitro-1,4,7,10-tetrazadecane. See 1,2-Bis(2^1-aminoethylnitramino)-ethane in Vol 2, p B129-L

Dinitrotoluenes. They aredescribed under TOLUENE and DERIVATIVES

2,4-Dinitro-1,3,5-triaminobenzene. See under Triaminobenzene and Derivatives

1,7-Dinitro-1,4,7-triazaheptane. See Bis-(β-nitraminoethyl) -amine in Vol 2, p B128-R

4,6-Dinitro-1,3,5-triazido-benzene. See Vol 2, p B43-R, under BENZENE AND DERIVATIVES

2,3-Dinitroxy-1-aminopropane. See 1-Amino-2,3-propanediol Dinitrate in Vol 1, p A251-L

β,β′-Dinitroxydiethyl-ammonium-hydroxide or **Bis(β-nitroxyethyl)-ammonium-hydroxide.** See under Diethylolamine and Derivatives, p D1242-L

β,β′-Dinitroxydiethyl-dinitroöxamide or **Bis(β-nitroxyethyl)-dinitroöxamide** (Code name **NENO**). See under Diethylol-oxamide, p D1244-L

β,β′-Dinitroxydiethyl-nitramine or **Bis-(β-nitroxyethyl)-nitramine** (Code name **DINA**). See under Diethylolamine, p D1240-R

β,β′-Dinitroxydiethyl-nitrosamine or **Bis (β-nitroxyethyl)-nitrosamine.** See under Diethylolamine, p D1240-L

Dinitroxydimethylnitramine or **Di(nitroimino)-methanol Dinitrate,**
$(O_2NO)H_2C.N(NO_2).CH_2(ONO_2)$; mw 212.08, N 26.41%; OB to CO_2 +15.1%; crysts, mp 59–60°; bp – expl at higher temp; sol in methanol, eth, et acet, acetone, NMe and doxane; sl sol in benz & chlf; insol in w; was obtd by Chute et al on treating with 99% nitric acid some of the products isolated on nitrolysis of hexamine

It is a powerful, impact-sensitive expl, which unfortunately decomposes on standing when exposed to air
Refs: 1) Beil, not found 2) W.J. Chute et al, CanadJRes **27B**, 235–37(1949) & CA **43**, 8354 (1949) 3) E. Aristoff et al, CanJRes **27B**, 543(1949) & CA **43**, 9075(1949)

1,5-Dinitroxy-2,4-dinitro-2,4-diazapentane or **2,4-Dinitro-2,4-diazapentanediol-1,5-dinitrate** (Code name **COX**). See under Dihydroxytrimethylenediamine

Di(β-nitroxyethyl)-nitramine. See under Diethylolamine, p D1240-R

Di(β-nitroxyethyl)-N,N′-dinitroöxamide or **N,N′-Dinitroethylol-oxamide** (Code name

NENO). See under Diethylol-oxamide, p D1244-L

Di(nitroxyethyl) -nitramine or **Diethylolnitramine Dinitrate**. See under Diethylolamine or Diethanolamine, p D1240-R

Dinitro-xylene; Dinitro-xylene-diazide and Dinitro-xylene-dinitrate. They are described under XYLENE and DERIVATIVES

Dinitroxynitraminodiethane or **Diethylolnitramine Dinitrate**. See under Diethylolamine, p D1240-R

1,9-Dinitroxy-1,3,5,7,9-pentamethylene-2,4,6,8-tetranitramine. See 1,9-Dinitroxy-2,4,6,8-tetranitro-2,4,6,8-tetrazanonane

1,8-Dinitroxy-1,3,5,6,8-pentamethylene-2,4,7-trinitramine. See 1,8-Dinitroxy-2,4,7-trinitro-2,4,7-triazaöctane

Dinitroxy(propaneamine). See under Aminopropanediols and Derivatives in Vol 1 of Encycl, p A251-L

Dinitroxy(propylamine). Same as above

Dinitroxy(propylamine) Nitrate, $H_2N.C_3H_5(ONO_2)_2+HNO_3$, is described in Vol 1, p A251-R as the:
Nitrate Salt of 1-Amino-2,3-propanediol Dinitrate

This compd seems to be identical with **Dihydroxy-propylamine Trinitrate**, patented in Germany before WWII for use in explosive compositions

Refs: 1) DynamitAG, BritP 358157(1930) & CA **26**, 6141(1932) 2) Clift & Fedoroff, **2**(1943), pp D7 & **4**(1946), 26

Di(nitroxypropyl)-amine or **Dinitroxydipropylamine**. See under Di(propylol)-amine and Derivatives

Dinitroxyquinone. See under Dihydroxybenzoquinone and Derivatives

1,7-Dinitroxy-1,3,5,7-tetramethylene-2,4,6-trinitramine or **1,7-Dinitroxy-2,4,6-trinitro-2,4,6-triazaheptane** (Code names **ATX** and **NBSX**). See under Dihydroxytetramethylenetriamine and Derivatives

1,9-Dinitroxy-2,4,6,8-tetranitro-2,4,6,8-tetraazanonane (Code designation **106**) or **1,9-Dinitroxypentamethylene-2,4,6,8-tetranitramine** (called in CA 2,4,6,8-Tetranitro-2,4,6,8-tetraazanonane-1,9-diol Dinitrate)
$(O_2NO)H_2C.N(NO_2).CH_2.N(NO_2).CH_2.N(NO_2).-CH_2.N(NO_2).CH_2(ONO_2)$; mw 434.21, N 32.26%, OB to CO_2 minus 3.7%; solid, mp 201° (on rapid heating), bp – expl at higher temp. It can be prepd either by nitration of 1,9-Diacetoxypentamethylene-2,4,6,8-tetranitramine (Code name AcAn) (described in this Vol as 1,9-Diacetoxy-2,4,6,8-tetranitro-2,4,6,8-tetrazanone) or by nitration of 2,6-Dinitro(bicyclo)-1,3,5,7,9-pentamethylene-2,4,6,8-tetramine (Code name DPT) also called 3,7-Dinitro-1,5-endomethylene-1,3,5,7-tetraaza-2,4,6,8-cycloöctane and described here under 1,5-Endomethylene-1,3,5,7-tetraaza-2,4,6,8-cycloöctane and derivatives

Its explosive properties are:
Impact Sensitivity – Bruceton No 3 Machine, 50% positive with 5kg wt at 14cm, vs 50cm for RDX; twelve times as sensitive as TNT
Power by Ballistic Mortar: 155% TNT
Power by Trauzl Test: 164% TNT
Thermal Stability – not given in Blatt
Velocity of Detonation: 7305m/sec at sp gr 1.265g/cc and 7895 at 1.35

Refs: 1) Beil, not found 2) A.H. Blatt, OSRD **2014**(1944) 3) A.F. McKay et al, CanJRes **27B**, 466(1949) & CA **43**, 9072 (1949) 4) R.N. Jones & G.D. Thom, CanJRes **27B**, 831, 835(1960) & CA **44**, 2848(1950) (UV absorption spectrum) 5) E. Lieber et al, AnalChem **23**, 1594(1951) & CA **46**, 3857 (1952) (IR absorption spectrum) 6) G.F. Wright & W.J. Chute, USP 2678927(1954) & CA **49**, 7606–07(1955) (Prepn)

1,5-Dinitroxytrimethylene-2,4-dinitramine (Code named **COX**). See under Dihydroxytrimethylenediamine

1,7-Dinitroxy-2,4,6-trinitro-2,4,6,-triazaheptane or **1,7-Dinitroxy-1,3,5,7-tetramethylene-2,4,6-trinitramine** (Code named **ATX** and **NBSX**). See under Dihydroxytetramethylenetriamine

1,8-Dinitroxy-2,4,7-trinitro-2,4,7-triazaöctane or **1,8-Dinitroxy-1,3,5,6,8-pentamethylene-2,4,7-trinitramine** (CA name 2,4,7-Trinitro-2,4,7-triazaöctane-1,8-diol Dinitrate), $(O_2NO)H_2C.N(NO_2).CH_2.N(NO_2).CH_2.CH_2.-N(NO_2).CH_2(ONO_2)$; mw 374.19, N 29.94%; wh solid, could not be purified from dioxane, CCl_4 or acet; unstable product which decompd after a few mins at RT; was obtd when 1,8-acetoxy-2,4,7-trinitro-2,4,7-triazaöctane was nitrated with 98% nitric acid at 0° for four mins and the soln kept at 0° for seven mins longer & drowned in 10 parts ice-water mixt. The crude material was reconverted to the acetoxy deriv by treatment with Na acetate in acetic acid at RT (Ref 2)

Refs: 1) Beil, not found 2) G.S. Myers, CanJRes **27B**, 499(1949) & CA **43**, 9074 (1949) 3) Not found in later Refs thru 1966

Dinitrüre. Accdg to Sidgwick (Ref), the name "dinitrure" applies to compds contg the group $>C(NO_2).C(NO_2)<$, which can be isolated among the products of reactions of nitrogen oxides with unsaturated hydrocarbons

For example, when stilbene, $C_6H_5CH:CH.C_6H_5$, is treated with nitrogen pentoxide, N_2O_5, the resulting compd is $C_6H_5.CH(NO_2).CH(NO_2).C_6H_5$, named 1,2-dinitro-1,2-diphenyl-ethane. The group, $-CH(NO_2).CH(NO_2)-$, is a "dinitrure"

Some "dinitrures" are explosive

Ref: N.V. Sidgwick, "Organic Chemistry of Nitrogen", OxfordUnivPress, London (1937), pp225 & 244

Dinitryl. Same as Glycerin α-2,4-dinitrophenylether Dinitrate or 1-(2,4-Dinitrophenoxy)-2,3-propane Dinitrate, described under GLYCERIN AND DERIVATIVES

Dinol. See 2,2,8,8-Tetranitro-4,6-dioxa-1,9-nonanediol which is listed under 4,6-Dioxa-1,9-nonanediol as a conf compd

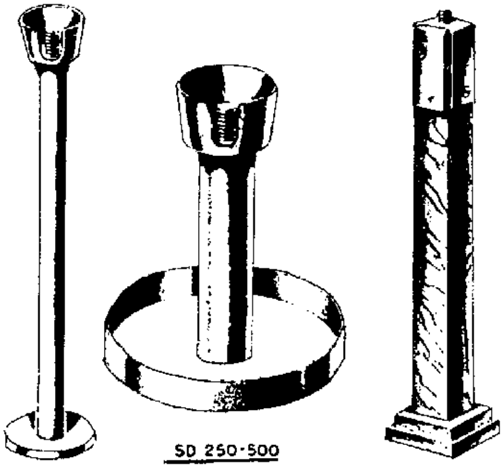
Dinol. Commercial name for Diazodinitrophenol, described under DINITROBENZENE-DIAZOÖXIDE in Vol 2, p B59-L

"Dinort" Rods. German WWII devices secured to the nose of A/P (antipersonnel) bombs designed to produce burst just above the ground, because fragments would be ineffective if the bomb had penetrated the soil prior to bursting

Dinort rods were also used with shaped charge bombs, acting in those cases as "stand-off" devices to improve the effectiveness of the charge

There were two types of Dinort rods: a) Drawn steel tubes (1.75 inch diam by 23.6 inches long or 2.75 x 14.8) and b) Square wooden sticks (2.25 x 2.25 and 22.6 inches long

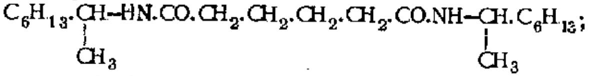
Ref: Fedoroff et al, PATR **2510**(1958), p Ger 37-L



Dinort Rods

Dioctyl Adipamide and Derivatives

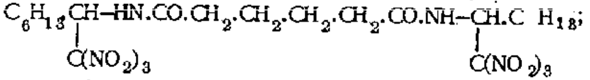
N,N'-Dioctyl-adipamide or *Bis(2-ethylhexyl)-adipamide,*



$$C_6H_{13}.\underset{\displaystyle CH_3}{\underset{|}{CH}}-HN.CO.CH_2.CH_2.CH_2.CH_2.CO.NH-\underset{\displaystyle CH_3}{\underset{|}{CH}}.C_6H_{13};$$

mw 368.60, N 7.60%; may be considered as the parent compd of its hexanitro deriv, although not used to prep it:

N,N'-Di(1,1,1-trinitro-2-octyl) -adipamide or **1,1,1,1',1',1'-Hexanitro-2,2 -dioctyl-adipamide,**



$$C_6H_{13}\underset{\displaystyle C(NO_2)_3}{\underset{|}{CH}}-HN.CO.CH_2.CH_2.CH_2.CH_2.CO.NH-\underset{\displaystyle C(NO_2)_3}{\underset{|}{CH}}.C\ H_{18};$$

mw 638.59, N 17.55%; wh solid, mp 140°(dec), burns with a flash & can be detonated with diffc; was obtd by reaction of nitroform, n-heptaldehyde & adipamide in Skellysolve B under reflux for several hrs (Ref 3)

Refs: 1) Beil, not found (Parent) 2) Beil, not found (Hexanitro) 3) P.O. Tawney, USRubberCo QuarterlyProgressRept **No 15**, Nord **10129**(1951), p7

Dioctyl Adipate (DOA) or **Bis(2-octyl)-adipate.** Same as **Di-(2-ethylhexyl)-adipate,** described earlier in this Vol, without giving US Specification requirements and tests. This compd has been used in polyisobutylene binders intended for expl and proplnt compns. One of the rocket proplnts, **Unit No 547**, using DOA is described in conf "Propellant Manual SP1A/M2 (1969)

US Military requirements for DOA are given in Specification MIL-D-13383(Ord), 15 April 1954 entitled DI-(2-ETHYLHEXYL)-ADIPATE and in Engineering Order EOPA-52740-2, 3 June 1968, which requires changing Acid Number from 0.07 to 0.10

Requirements:

Property	**Required Value**
1) Color	No darker than color std of 4.4.1
2) Sp Gr at 20°/20°C	0.927±0.002g/c
3) Refractive Index at 20°C	1.4470±0.0010
4) Fl Pt, min	385°F
5) Fire Pt, min	420°F
6) Acid Pt, max	0.10
7) Viscosity at 100°F	8.46±0.30 centistokes
8) Saponification Number	302±2

Lot. Unless otherwise specified, a lot shall consist of 50000 pounds, maximum. However, when shipment is made in tank cars the contents of each car shall constitute a lot

Sampling in Containers. Remove at least 3 and not more than 10 containers from a lot, mix the liquid in each container and remove ca 8 oz from each to an air-tight glass bottle, having a glass stopper or cork stopper covered with a metal foil. Label the bottles (with these primary samples) and remove from each an equal amt into a similar 8-oz bottle, which is labeled as "composite", giving the name of the material, manufacturer, plant, contract or order number, lot number and its size

Sampling in Tank Cars. Attach to the neck of a quart-size, clean, narrow-neck bottle, a piece of cord and fill the bottle by lowering and raising it thru the liquid in the tank. Discard the rinsing and repeat the operation by uniformly lowering and raising the bottle. The bottle must just be filled as it reaches the surface of the liquid. Transfer the collected liquid to an air-tight bottle provided with a glass-stopper or a cork stopper covered with metal foil. Label the bottle as described above

Inspection. It shall be conducted as described in Specification MIL-A-2550A, Amend 5 (Feb 1969): The supplier is responsible for the performance of all inspection requirements. He may utilize his own or any other inspection facilities. When reqd, the supplier shall submit samples to a Government approved laboratory for examination & tests. The samples will be inspected for all the requirements set forth in the detail specification (See above)

Tests.

4.4.1. *Color.* Prepare the standard color solution by introducing 1.00±0.01g of CP iodine and 5.0±0.1g of CP KI to a 150-ml beaker, contg ca 25ml of distilled water as solvent. Transfer the soln quantitatively to 1000-ml volumetric flask, dilute to the mark with distd w and mix thoroughly. This soln shall not be used after 24hrs. For the test, fill a clean 50-ml Nessler tube to the mark with above color soln and fill the other 50-ml tube with the sample. Hold the two tubes side by side and compare their colors using transmitted light. Consider the sample satisfactory if its color is not darker than that of the standard

4.4.2. *Specific Gravity.* Determine sp gr of the sample at 20/20°C by means of a suitable pycnometer (such as described in Vol 3 of Encycl, p D69)

4.4.3. *Refractive Index.* Determine RI at 20°C using an accurate refractometer such as Abbé (See SGA Scientific Inc, Bloomfield, NJ Catalog (1972), p 742 or other suppliers

4.4.4. *Flash Point.* Determine it in accordance with method 1103.7 of Federal Test Method Std 791B (Jan 1969) (ASTM Method D92–66). The Cleveland Open Cup is filled to a specified level with sample. The temp is increased rapidly at first and then at a slow constant rate as the flash point is approached. At specified intervals a small test flame is passed across the cup. The lowest temp at which the flame causes vapors above the liq to ignite is taken as the flash point [For details see ASTM Standards, Part II (1971), pp66–92]

4.4.5. *Fire Point.* It shall be determined in accordance with method 1103.7 of Federal Test Method Std 791B (Jan 1969) (ASTM Method D92–66). See under Flash Point above. To det flash point, the test is continued until application of test flame causes the DOA to ignite and burn for 5 secs [For details see ASTM Standard, Part II (1971), pp 66–92]

4.4.6. *Acid Number.* Transfer ca 100ml of a soln consisting of equal parts, by volume, of 95% ethanol and reagent grade benzene, to a 300-ml Erlenmeyer flask and heat it on a steam bath to incipient boiling of its contents. Add 2–3 drops of ca 1% phenolphthalein indicator and titrate with appr 0.01N std KOH soln, to a faint pink color. Add, exactly, 50ml of sample, and continue titration from the same KOH buret to a faint pink color of soln in the flask. Calculate the acid number (milligrams of KOH soln required to neutralize 1 g of the sample), as follows:

$$\text{Acid Number} = \frac{56.1(AN)}{Bd}$$

where: A = ml of KOH soln required to titrate the sample
N = normality of KOH soln (usually 0.01N)
B = volume of sample titrated (50ml)
d = specific gravity of the material (See 4.4.2)

4.4.7. *Viscosity.* It shall be detd in accordance with method 305.6 of Federal Test Method Std 791B (Jan 1969) (ASTM Method D445–65). The time is measured for a fixed vol of the liq to flow thru a calibrated Ubbelohde capillary viscometer under an accurately reproducible head and at a closely controlled temp. The kinetic viscosity is then calculated from the measured time flow and the calibration constant of the viscometer. Kinetic viscosity is a measure of the time for a fixed vol of liq to flow by gravity thru a capillary. It is usually expressed in centistokes, cSt, so that 1 St = 100 cSt. The dynamic viscosity is the product of kinematic viscosity & density of the liq, both at same temp. The unit is poise, P, in g/cm/sec [For details see ASTM Standard, Part 17(1971), pp178–83]

4.4.8. *Saponification Number.* Transfer an accurately weighed portion of ca 2g of the sample to a 300-ml ground-glass Erlenmeyer-, or round-bottom flask. Add 50ml of appr 0.5N alcoholic KOH soln and connect the flask to an air condenser, ca 250mm in length. Reflux for 3hrs on a steam bath at such a rate that the major portion of the alcohol will condense in the lower half of the condenser. After allowing the flask with contents to cool to RT, disconnect the condenser and rinse it inside with three 10-ml portions of neutralized 95% alcohol, collecting the washings in the flask. Remove the condenser and add 2–3 drops of 1% phenolphthalein indicator to the flask contents, and titrate with appr 0.5N hydrochloric acid to the first permanent faint pink color. Make a blank detn on 50ml of the alcoholic 0.5N KOH soln and titrate with HCl as above. Calculate the saponification number as follows:

$$\text{Saponification Number} = \frac{56.1(A-B)N}{W}$$

where: A = ml of HCl required to titrate the sample

B = ml of HCl required for blank titration

N = normality of hydrochloric acid soln

W = weight of the sample (ca 2g)

Dioctyl Azelate (DOZ). See Di(2-ethylhexyl) azelate in this Vol, p D1237-L

N,N'-Dioctyl-p-phenylenediamine (DOPDA) or **Bis(2-ethylhexyl)-p-phenylenediamine,** $(H_2N)_2C_6H_2[COOCH_2CH(C_2H_5)C_4H_9]_2$; mw 420.58, N 6.66%; description of compd & props are given below under specification requirements

E.J. Bicek [USP 320930 (1965)& CA **64**, 3415(1966)]prepd this compd by passing H and a soln of $p\text{-}O_2NC_6H_4NH_2$ in $C_2H_5COC_5H_{11}$, downward over a sulfided-Pt-catalyst in a jacketed reaction tube at the rate of 100cc/hr at 850lbs/sq inch at 267°F

DOPDA has been used as an additive to rubber compositions at the time of manufacture for the purpose of providing ozone resistance to elastometers. Mixtures of DOPDA with solvents such as acetone (usually in 50/50 ratio) are flammable and toxic, causing skin irritation. The material covered by US Military Specification MIL-D-50000A(MR), July 1966 is intended for use as an externally applied (brush or dip) solution to rubber items, particularly tires. This chemical functions as an antiozonant, preventing cracking of stressed, vulcanized rubber items in outdoor storage

Requirements and tests covered by the above Spec are as follows:

1) *Color* shall be reddish, characteristic of this compound. It is detd by visual examination of a sample contained in a test tube

2) *Refractive Index at 20°* – 1.507 to 1.511 when determined in accordance with method 4371 of Fed Test Method Std No 141

3) *Specific Gravity* at 60/60° F 0.900 to 0.915 when detd in accordance with method 4183 of Fed Test Method Std No 141

4) *Solubility in Acetone* shall be complete when one volume of DOPDA is mixed with one vol of acetone

5) *Pour Point.* Fill a test tube about half-full, store it for 24hrs at a temp no higher than 42°F and then turn the tube upside down. The material shall be capable of being poured

Dioctyl Phthalate (DOP) or **Bis(2-ethylhexyl)-phthalate,** $C_6H_4[COOCH_2CH(C_2H_5)C_4H_9]_2$; mw 390.54; lt col liq, sp gr 0.9861 at 20/20°, n_D 1.4836 to 1.485 at 20°; fr p minus 50 to 55°, bp 231° at 5mm, bp 385° at 760mm, flash p 410°F, vapor pressure 1.3mm Hg at 200°, viscosity 81.4cps at 20°; insol in w, miscible with mineral oil; can be prepd by interaction of 2-ethylhexyl alcohol with phthalic anhydride (Refs 5 & 6)

It is used as a plasticizer for many resins and synthetic rubbers; was used in quantities up to 5% in British "Casting Powder No 1". It has been used as a fuel-binder in some US propellants (such as Unit Nos 1024, 1026, 1058, 1059 & 1067 described in ConfPropInt-Manual SPIA/M2(1969)

US Armed Forces requirements are covered by Specification MIL-D-13796(Ord), (Nov 1954)

1) *DOP Content* – 99.5% by wt, min. Transfer a 1.0g portion accurately weighed to a 250ml Erlenmeyer flask having a standard taper ground glass neck. Add exactly 25.00ml of 0.5N alcoholic KOH soln, attach a reflux condenser and heat the flask on a steam bath until a ppt is formed and then 10 mins longer. Add 15ml of distd w to dissolve the pptd K phthalate and reflux for an addnl 45–60 mins. Cool the flask to RT; disconnect the reflux condenser, lift it slightly above the ground glass joint and rinse them with ca 25ml of distd w into the flask. Titrate the excess of KOH in the flask with 0.3N HCl in presence of 10 drops of phpht indicator until disappearance of pink color. Run a blank concurrently with the sample

$$\%DOP = \frac{19.53\,(A-B)N}{W}$$

where: A = ml of HCl used to titrate the blank

B = ml of HCl used to titrate the sample

N = normality of HCl

W = weight of sample

2) *Specific Gravity at 20/20°* shall be 0.9840 to 0.9865 when detd by means of a pycnometer (See Vol 3 of Encycl, p D69)

3) *Acidity* (as phthalic acid) shall be 0.01% by wt, max. Place 100ml of ethanol in a 250ml Erlen flask, add 3 drops of phpht indicator and neutralize to a faint pink color with 0.1N NaOH soln. Add a 50.0ml portion of sample to alc

and titrate with 0.1N NaOH soln to a faint pink color persisting for at least 15 secs

$$\%\text{Acidity (as phthalic acid)} = \frac{0.166AN}{G}$$

where: A = ml of NaOH soln required for titration
N = normality of NaOH soln
G = specific gravity of sample

4) *Water.* DOP shall be miscible without turbidity with 9 volumes of 60° API gasoline, when detd in the following manner. Pipet a 5ml portion of sample into a 100-ml glass-stoppered graduate, add 5ml of above gasoline and shake. If no turbidity is formed add another 5-ml portion of gasoline, shake and if no turbidity forms, continue addition in 5ml portions until a total of 45ml of gasoline will be used
Note: It is stated on p1 of the Spec that DOP shall be miscible without turbidity with 9 volumes of gasoline. If the sample is 5ml, the total vol of gasoline shall be 45ml and not 95ml as stated on p3 of the Spec

5) *Color as Received* shall not be darker than that of the 50-ppm Pt-Co standard, when detemined as described below

The APHA (American Public Health Association) color standard (stock solution) is prepd by dissolving 1.245g K_2PtCl_6 (contains 500mg Pt) & 1.000g $CoCl_2.6H_2O$ (contains 248mg Co) in distd w contg 100ml cp HCl and diluting with w to 1000ml. This soln has a color intensity equivalent to 500ppm of platinic cobalt chloride
Note: Accdg to Bofors laboratory manual (Ref 6, p30) this soln, known as APHA500 contains 500mg Pt and by diluting its 10ml with 90ml of w in 100ml Nessler tube, the APHA50 is obtd

For detn of color, fill to the mark a 100ml Nessler tube with the sample as received and compare the intensity of color with that of APHA50 contd in another Nessler tube by looking vertically downward thru the two solns against a white background

6) *Heat Test Color* shall not be darker than that of the 75-ppm platinum cobalt standard when detd as described below:

Prepare the APHA75 standard by diluting 15ml of APHA50 to 100 in a Nessler tube and compare the color with that of the sample which was previously heated in an oil bath at 220±5°C for 2hrs and allowed to cool during 30 mins to RT

7) *Suspended Matter* shall be absent when a sample transferred to a Nessler tube is observed vertically against a white background

8) *Odor* shall be slight and mild when several mls are poured in several places on clean filter paper, and the odor noted at once

9) *Ash Content* shall be not greater than 0.005%. Transfer a 10g portion of sample into an accurately tared porcelain crucible, evaporate to dryness on a hot plate, and ignite the residue at red heat, cool in a desiccator and weigh. Repeat incineration to be assured that constant wt was achieved

$$\%\text{ Ash} = \frac{100A}{W}$$

where: A = increase in wt of crucible
W = wt of sample

DOP has also been used in Swedish expls and proplnts. Their specification requirements and tests are described in Ref 5

1) *Appearance and Color.* Pour the sample into a Nessler tube and inspect for transparency and absence of mechanical impurities. Compare its pale-yel color with APHA standards in the same manner as described under US specification tests. The APHA500 contains 500mg Pt per liter

2) *Refractive Index* — 1.485 to 1.490 at 20°

3) *Ester Content, as DOP.* Transfer ca 2g sample weighed on analytical balance, into a 250ml extraction flask and dissolve in 100ml of ethanol. Add from a buret 50.00ml of 0.5N NaOH soln, connect a reflux condenser to the flask and heat until complete saponification of sample (2 hours). Cool to RT and titrate the excess of NaOH soln with 0.5N sulfuric acid soln using phpht as an indicator until discoloration of soln

Run a blank detn at the same time

$$\%\text{ Dioctylphthalate} = \frac{19.52(b-a)n}{w}$$

where: a = ml of 0.5N sulfuric acid soln used for the sample
b = ml of 0.5N sulfuric acid soln used for the blank
n = normality of sulfuric acid soln
w = weight of sample

Note: The above tests are the same as those used for Diamylphthalate and Dibutylphthalate, described on pp82–3 of Ref 5, except that the formulas for calcn are different, namely

$$\%\text{ Diamylphthalate} = \frac{15.13(b-a)n}{w} \quad \text{and}$$

$$\% \text{ Dibutylphthalate} = \frac{13.92(b-a)n}{w}$$

We failed to describe the tests given in Bofors book under our description in this volume of Diamylphthalate and Dibutylphthalate, pp D1150 & D1197, respectively

Diamylphthalate and Dibutylphthalate have been used in Swedish proplnts as moderants and plasticizers for NC

4) *Acidity.* Use the same method as described for Diamylphthalate and Dibutylphthalate on pp 82–3 of Ref 5

Weigh out 10g sample into 300ml Erlenmeyer and dissolve in 100ml of previously neutralized (with NaOH soln) ethanol. Titrate with 0.2N NaOH soln using phpht indicator (using a microburet) until the appearance of faint pink coloration

$$\% \text{ Phthalic Acid} = \frac{8.3an}{w}$$

where: a = ml of 0.2N NaOH soln used
n = normality of NaOH soln
w = weight of sample

Same formulas are used for Diamylphthalate and Dibutylphthalate

5) *Water Content.* Use the Karl Fischer method described in detail on pp15–18 of Ref 5

Refs: 1) Beil, not found 2) A.H. Castelli et al, PATR **2222**(1956) 3) L. Marvillet, MP **40**, 273 (1958) 4) L. Marvillet, MP **42**, 249(1960) 5) Anon, "Analytical Methods for Powders and Explosives", ABBofors-Nobelkrut, Bofors, Sweden (1960) 6) CondChemDict (1961), 378-R 7) Proplnt Manual, SPIA/M2(1969) (Conf)

Dioctyl Sebacate (DOS). See Di(2-ethylhexyl)-sebacate in this Vol and the Conf Proplnt Manual SPIA/M2 (1962) in which are described rocket proplnts Units 511 & 611 using DOS as a plasticizer

US Military requirements for DOS are covered by Spec MIL-D-10692(Ord)(1954), entitled Di(2-ethylhexyl)sebacate (without mentioning that the compd is identical with commonly used name Dioctyl Sebacate)

The requirements and tests are as follows:

1) *Color* must be colorless to amber

Prepare the standard color solution by transferring an accurately weighed 0.202g sample of CP iodine and 1.000g of CP K iodide to a 1000ml volumetric flask contg sufficient distd w to dissolve both substances. Fill the flask to the mark, mix thoroughly and fill one of the 50ml Nessler tubes to the mark. Fill another Nessler tube with sample, place both tubes side by side and compare their colors by transmitted light

2) *Specific Gravity at 20/20°* – 0.913±0.002. Determine by a pycnometer as described in Vol 3 of Encycl, p D69

3) *Refractive Index at 20°* – 1.4495±0.0010. Determine it using an Abbé Refractometer

4) *Flash Point* – 400°F, minimum. Use Method No 110.3 of Spec VV-L-791

5) *Fire Point* – 490°F, minimum. Use the same apparatus as in Flash Point

6) *Acid Number* – 0.1, maximum. Heat a 50/50 mixt (by vol) of 95% alc and CP benzene placed in a 300ml Erlenmeyer flask to incipient boiling, add 3 drops of 1% phpht indicator and neutralize the soln by adding 0.01N KOH to a faint pink color. Add 50ml of sample and titrate with 0.01N KOH to a faint pink color

$$\text{Acid Number} = \frac{56.1AN}{VG}$$

where: A = ml of KOH soln required for the sample
N = normality of KOH soln
V = volume of sample
G = sp gr of sample

6) *Viscosity at 100°F* – 0.1153±0.0010 poises. Use a calibrated Ubbelohde Viscosimeter in accordance with Method A of Method 30.5 of Spec VV-L-791

7) *Saponification Number* – 261±2. Transfer a 2g accurately weighed portion of sample to a 300ml flask, provided with a ground glass neck and add 50ml of ca 0.5N alcoholic KOH soln. Connect the flask to an air condenser ca 250mm in length and reflux for 3 hours on a steam bath at such a rate that the major portion of the alcohol will condense in the lower half of the condenser. Cool the ensemble to RT, lift the condenser and, holding it over the neck, rinse with three 10ml portions of neutralized 95% alcohol collecting the washings in the flask. Add 3 drops of 1% phpht indicator and titrate with 0.5N HCl soln to disappearance of practically all pink coloration. Titrate a blank consisting of 50ml of 0.5N alc KOH soln refluxed in the same manner as the sample

$$\text{Saponification Number} = \frac{56.1(A-B)N}{W}$$

where: A = ml of HCl soln required for blank
B = ml of HCl soln required for sample
N = normality of HCl soln
W = wt of sample

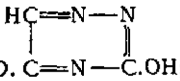
-dioic. A suffix indicating two-COOH groups, as for example ethanedioic HOOC.COOH, which is the same as oxalic acid
Ref: Hackh's Dictionary (1944), pp278-L & 604-L

Di-Oil. One of the names for crude DNT (Dinitrotoluene), $CH_3.C_6H_3(NO_2)_2$ which will be described under TOLUENE AND DERIVATIVES

-diol. A suffix indicating the presence of two hydroxyl groups in an organic compound of aliphatic series. Such compds are known as **diols**, as for example ethandiol (or glycol), propandiol, butandiol, etc
Ref: Hackh's Dictionary (1944), pp278-L & 383-L

Diolefine. An unsaturated aliphatic hydrocarbon with the general formula C_nH_{2n-2} and two double bonds. Eg: diallyl (1,5-hexadiene), $CH_2{:}CH.CH_2.CH_2.CH{:}CH_2$
Ref: Hackh's Dict (1944), p278-L

3,5-Diol-*as*-triazine,



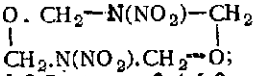
mw 113.08, N 37.16%; wh compd (sublimation in vacuo, 200°, 1mm), mp 277.79°; was prepd when 3,5-dihydroxy-1,2,4-triazine-6-carboxylic acid was heated in diphenyl ether for 30 mins at 190–210°, mixt cooled and ppt washed with diethyl ether (Ref 3). This compd was prepd also by Seibert by a different method. No expl props are reported
Refs: 1) Beil, not found 2) W. Seibert, ChemBer **80**, 494(1947) & CA **43**, 123(1949)
3) E.A. Falco et al, JACS **78**, 1940(1956) & CA **50**, 13952(1956)

-dione. A suffix indicating the presence of two keto-groups: –OC.CO–, such as in compds known as diketones: 2,3-butandione(2,3-butanone) or diacetyl, $H_3C.CO.CO.CH_3$; or 2,4-pentadidione(2,4-pentanone) or actylacetone, $H_3C.CO.CH_2.CO.CH_3$, etc
Ref: Hackh's Dict (1944), pp278-L (dione) & 274 (diketone)

Diorrexine. A mining expl, invented by Pancera of Austria and patented in England in 1881 and later tried in the USA. Its composition was: K or Na nitrate, sawdust & sulfur. To these could be added charcoal, Picric Acid (ca 1.5%) & water
Ref: Daniel's Dict (1902), p206

Dioxa. In the nomenclature which seems to originate in Canada, the word "*dioxa*" indicates the presence of two separate oxygens as a part of the chain, just as the word *diaza* indicates the presence of two nitrogens in the chain. For example, the hypothetical compd, R–O–NH–CH_2–NH–O–R, contains a dioxa and a diaza group. The name *oxa* indicates one oxygen and the name *trioxa* three oxygens in the chain. Correspondently, *aza* indicates one nitrogen, *triaza* three, *tetraza* four, *pentaza* five, etc

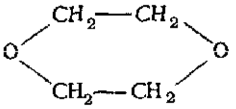
Following are examples: **3,7-Dinitro-3,7-diaza-1,5-dioxo-cyclooctane,**



1,9-Dinitroxy-2,4,6,8-tetranitro-2,4,6,8-tetrazanonane, $(O_2NO)H_2C.N(NO_2).CH_2.N(NO_2).CH_2-N(NO_2).CH_2.N(NO_2).CH_2(ONO_2)$;
2,10-Dioxa-4,6,8-triaza-hendecane,
$H_3C.O.CH_2.NH.CH_2.NH.CH_2.NH.CH_2.O.CH_3$

1,3-Dioxalane. See 1,3-Dioxolane

Dioxane or Dioxan-1,4. *(Diethylene-1,4-dioxide),*



mw 88.10; col, very flammable liq, sp gr 1.034

at 20/4°, fr p 11.8°, bp 101.4°, n_D 1.4221 at 20°, fl p 18°(65°F)(ASTM open cup), vapor pressure 29.0mm Hg at 20°; viscosity 0.0131 poise at 20°, heat of combstn 581 kcal/mole; miscible with w & alc; sol in eth and in most org solvents, and is an excellent solvent for resins, waxes and oils. Its unusual solvent power is due to the presence of two ether-oxygen atoms (Refs 11, 12, 13 & 14)

Vapors of dioxane are of pleasant odor but extremely toxic to inhale. In the book of Curme & Johnston (Ref 13) is cited a case of nearly simultaneous deaths in 1933 of five Brit workers after they inhaled nearly saturated vapors of dioxane during five 12-hr shifts. Inhaling air with 1000ppm dioxane repeatedly for 1½ hrs a day might cause kidney and liver injury. When taken orally by small animals, L_{50} were from 4 to 6ml per kg. Contact with skin should be avoided, not only because it is unpleasant, but also because dioxane is absorbed thru the skin

Dioxane was first prepd in 1863 by Lourenco (Refs 1 & 2) by heating ethyleneglycol with ethylene dibromide in sealed tubes for several days at 160°. In the same year Wurtz (Ref 3) prepd it independently by treating dioxane dibromide (obtd by him in 1862) with hydrogen sulfide or Hg at RT. Several industrial methods of prepn are listed in the book of Curme & Johnston (Ref 13). Among them may be mentioned a method patented by scientist of IG Farbenind (Ref 4), consisting of dehydration of ethyleneglycol, polyethyleneglycol or their ethers using catalysts such as sulfuric or hydrochloric acid, Zn chloride, etc. Other methods are in Ref 9

Accdg to Dreyfus (Refs 8 & 10) dioxane and its homologues can be obtd by passing formaldehyde or acetaldehyde over catalysts favoring esterification reactions, such as sulfuric acid or ferric chloride

In CondChemDict (Ref 14) are listed two methods of prepn: a) Treating ethyleneglycol with acids and b) Treating β,β'-dichloroethyl ether $(ClH_2C.CH_2Cl)_2O$ with alkali (See also Refs 5 & 7)

Chemical reactions of dioxane are described in Ref 13, pp121–22, but the description of tests and analytical procedures are not there

Dioxane is extensively used as a solvent for cellulose acetate, fats, greases, etc (Ref 14)

It was tried on the laboratory scale by Aaronson at PicArsn (Ref 6) as a plasticizer for NC's and found to be a fairly good solvent for Collodion Cotton and Pyrocellulose, but rather poor solvent for Guncotton and High Nitrogen NC (14% N). The resulting jels were very toxic and their dioxane solvent was very difficult to remove by evaporation. This made dioxane an undesirable solvent for NC's, much inferior to acetone and propylene oxide

Dioxane has been used by the US Armed Forces as a solvent and the requirements and tests were listed in Specification MIL-D-11323, which is now cancelled

Refs: 1) Beil **19**, 3, (609) & [4] 2) A. Laurenco, AnnChimPhys (3), **67**, 288(1863) 3) A. Wurtz, Ibid (3), **69**, 324(1863) 4) IG FarbenindustrieAG, USP 1681861(1928) & 1939189(1933) 5) J. van Alphen, Rec **49**, 1040–44(1930) 6) H.A. Aaronson, PATR **274**(1932) (Preliminary Study of the Nitration of Dioxane 7) C.W. Jones et al, IEC **25**, 1283–86(1933) 8) H. Dreyfus, FrP 772154 (1934) 9) IG FarbenindAG, GerP 597496 (1934) & 598952(1934) 10) H. Dreyfus, USP 2072101(1937) 11) T.H. Durrans, "Solvents", Reinhold, NY (1950), 159 12) I. Mellan, "Industrial Solvents", Reinhold, NY (1950), 674 13) G. O. Curme, Jr & F. Johnston, "Glycols", Reinhold, NY (1952), 119–22 (Numerous refs) 14) CondChemDict (1961), 403–R & 404–L 15) Kirk & Othmer, 2nd edit, **10**(1966), p643 (Under Glycols)

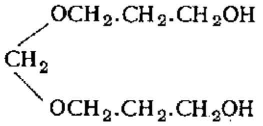
Dioxane, Nitration of. H.A. Aaronson [PATR **274** (Sept 1932)] attempted nitration of 1,4-dioxane with mixed acid and found a) it forms ethylene glycol dinitrate b) oxidation results in formation of an insol wh compd or of oxalic acid and c) it is not possible to oxidize to a mono- or poly-hydroxy deriv and to nitrate the product

NOTE: No nitrated derivs of Dioxane were found in the literature thru 1966

Dioxanes, Monocyclic are described by Elderfield **6**(1957), pp 1–58

4,6-Dioxa-1,9-nonanediol and Derivatives

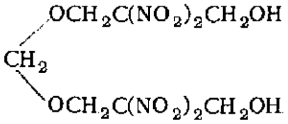
4,6-Dioxa-1,9-nonanediol,



$$CH_2\begin{cases} OCH_2.CH_2.CH_2OH \\ OCH_2.CH_2.CH_2OH \end{cases}$$

mw 164.20, O 38.98%; may be considered as the parent compd of its nitrated deriv, although not used to prep it:

Its **2,2,8,8-Tetranitroderivative** is described by T.N. Hall & K.G. Shipp in confidential report **NOLTR 61–6** (March 1961)



$$CH_2\begin{cases} OCH_2C(NO_2)_2CH_2OH \\ OCH_2C(NO_2)_2CH_2OH \end{cases}$$

mw 294.20, N 19.05%. This compd, designated as **DINOL**, is described in detail, with its method of prepn in the above report

Dioxatetraazapentadecane and Derivatives

3,13-Dioxa-5,7,9,11-tetraazapentadecane, $H_3C.CH_2.O.CH_2.NH.CH_2.NH.CH_2.NH.CH_2.NH-CH_2.O.CH_2.CH_3$; mw 220.31, N 25.89%; may be considered as the parent compd of its tetranitro deriv, although not used to prep it:

NOTE: Dinitro, $C_9H_{22}N_6O_6$, and Trinitro, $C_9H_{21}N_7O_8$, derivs were not found in the literature thru 1966

3,13-Dioxa-5,7,9,11-tetranitro-5,7,9,11-tetraazapentadecane, (called 1,9-Diethoxy-2,4,6,8-tetranitro-2,4,6,8-tetrazanonane by Chute et al), $H_3C.CH_2.O.CH_2.N(NO_2).CH_2.N(NO_2).CH_2-N(NO_2).CH_2.N(NO_2).CH_2.O.CH_2.CH_3$; mw 400.31, N 27.98%. This compd is described in this Volume, p D1224-L, under 1,9-Diethoxypentamethylene-2,4,6,8-tetranitramine

3,13-Dioxa-5,7,9,11-tetranitro-5,7,9,11-tetraazapentadecane-1,15-diol, $HOCH_2.CH_2O[CH_2.N(NO_2)]_4.CH_2O.CH_2.CH_2OH$; mw 432.31, N 25.92%; no props are given in the patent abstract; was prepd by treating the tetranitro linear amine having terminal nitroxy or halogen groups with dialcohols with or without $CaCO_3$ (Ref 2). The product forms polymers with diisocyanates & difunctional acids which, when mixed with Ammonium Perchlorate & C black form a solid rocket fuel

Refs: 1) Beil, not found 2) W.A. Gey & R. Reed Jr, USP 3121748(1964) & CA **60**, 11897g (1964)

Dioxatetraazatridecane and Derivatives

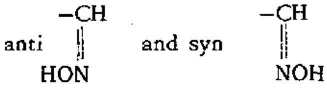
2,12-Dioxa-4,6,8,10-Tetraazatridecane, $H_3C.O.CH_2.NH.CH_2.NH.CH_2.NH.CH_2.NH.CH_2-O.CH_3$; mw 192.27, N 29.14%; its tetranitro deriv: **2,12-Dioxa-4,6,8,10-tetranitro-4,6,8,10-tetraazatridecane,** $H_3C.O.CH_2.N(NO_2).CH_2.N(NO_2)-CH_2.N(NO_2).CH_2.N(NO_2).CH_2.O.CH_3$, mw 372.26, N 30.01%; is described in this Volume, p D1304-L, under 1,9-Dimethoxy-2,4,6,8-tetranitro-2,4,6,8-tetraazanonane

Dioxatriazahendecane or Dioxatriazaundecane and Derivatives

2,10-Dioxa-4,6,8-triaza-hendecane; $H_3C.O.CH_2.NH.CH_2.NH.CH_2.NH.CH_2.O.CH_3$; mw 163.22, N 25.75%

Its trinitro deriv: **2,10-Dioxa-4,6,8-trinitro-4,6,8-triaza-hendecane,** or **2,10-Dioxa-4,6,8-trinitro-4,6,8-triaza-undecane,** $H_3C.O.CH_2.N(NO_2).CH\ .N(NO_2).CH_2.N(NO_2)-CH_2.O.CH_3$; mw 298.22, N 28.18%; is described in this Volume, p D1305-L, under 1,7-Dimethoxy-2,4,6-trinitro-2,4,6-triazaheptane

Dioxime. The monovalent –HC:NOH radical occuring in two isomeric forms:

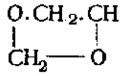


$$\text{anti}\ \begin{matrix}-CH\\ \|\\ HON\end{matrix}\qquad \text{and syn}\ \begin{matrix}-CH\\ \|\\ NOH\end{matrix}$$

Dioximes. A group of compds contg two dioxime radicals: *alpha* – Glyoximes; *beta* – Glyoxime Peroxides and *gamma* – Compounds in which the two oxime radicals are separated by the ethylene radical, such as in succinaldehydedioxime, $HON:CH.CH_2.CH_2.CH:NOH$

1,3-Dioxolane or Glycolmethylene Ether,

(Spelled 1,3-Dioxalane in CA and by Elderfield) or **Glycolformal**,



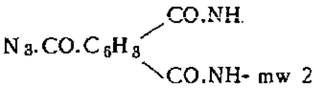
$O.CH_2.CH_2$ / CH_2—O ; mw 74.08; flammable col liq, sp gr 1.060 at 20/4°, bp 75–76°, flash p 35°F, vap press 70mm Hg at 20°; stable under neutral or sl alkaline condition; sol in w; can be prepd by warming ethyleneglycol with polyoxymethylene in the presence of phosphoric acid in a tube at 100°(Refs 1 & 2)

It is suitable as a low-boiling solvent and extractant for oils, fats, waxes and cellulose derivatives

Refs: 1) Beil **19**, 2, (609) & [3] 2) H.T. Clarke, JChemSoc **101**, 1804(1912) & CA **7**, 5904(1913) 3) Elderfield **5**(1957), pp1–44 4) CondChemDict (1961), 404-L

Dioxopiperazine. See Diketopiperazine

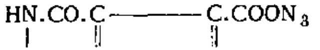
1,4-Dioxo-1,2,3,4-tetrahydrophthalazin-5-carbonyl Azide [called 1.4-Dioxo-1.2.3.4-tetrahydro-phthalazin-carbonsäure-(5) Azid in Ger],



$N_3.CO.C_6H_3\langle^{CO.NH}_{CO.NH}$- mw 231.17, N 30.30%; powd, mp – explodes on heating; insol in ether; was prepd from the hydrazide, $C_9H_8N_4O_3$, by reaction with $NaNO_2$ in dil nitric acid with cooling

Refs: 1) Beil **25**, (591) 2) T. Curtius, JPraktChem **91**(2), 91(1917) 3) Not found in later Refs thru 1962

3,6-Dioxo-1,2,3,6-tetrahydro-[pyrazolo-3',4',4,5-pyridazin]-5-carbonyl Azide [called 3.6-Dioxo-1.2.3.6-tetrahydro-[pyrazolo-3 .4 .4.5-pyridazin]-carbonsäure-(5) Azid in Ger],

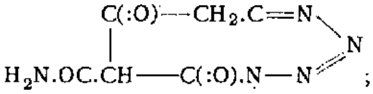


$HN.CO.C$———$C.COON_3$ / $HN.CO.C$—NH—N ; mw 221.14, N 44.34%; yel ppt, mp – explodes on heating; readily sol in alkali; insol in cold w; was prepd by nitrating pyrazole-3,4,5-tricarbonic acid trihydrazide with $NaNO_2$ in HNO_3 at 0°

Refs: 1) Beil **26**, (186) 2) T. Curtius, JPraktChem **91**(2), 72, 81(1915) 3) Not found in later Refs thru 1962

Dioxotetramethylenotetrazolecarboxamide and Derivatives

1,3-Dioxotetramethylenotetrazole-2-carboxamide or *5,7-Dioxo-5,6,7,8-tetrahydro-pyridotetrazole-6-carboxamide* (CA name),



$H_2N.OC.CH$——$C(:O).N$—N / $C(:O)$—$CH_2.C{=}N$—N ; mw 195.14, N 35.89%; no description of compd is given in abstract; was prepd from the 2-nitrile deriv by treating with concd sulfuric acid at 80–90°. On merely heating with w it splits off 2 moles of water, giving C-acetonyltetrazole, $H_3C.CO.CH_2\underline{C:N.N:N.NH}$, which does not undergo acid or ketone cleavage. The amide deriv of parent compd forms an ether, $(C_6H_4N_5O_2)_2O$, on merely boiling in alcohol

1,3-Dioxotetramethylenetetrazole-2-nitrile has no pronounced physiological action, the amide has a marked beneficial action on the heart, while neither the C-acetonyltetrazole nor its N-methyl ether has any physiological effect (Ref 2)

The carboxylic deriv, obtd from parent compd & concd sulfuric acid, decomp at 195° with evolution of carbon dioxide

Refs: 1) Beil, not found 2) G. Schroeter & E. Fink, Ber **71B**, 671–84(1938) & CA **32**, 4164–65(1938) 3) No later refs found thru 1966

Dioxy–. A German prefix indicating the presence of two hydroxyl groups (OH) in an organic compound

Dioxy–. An Amer or Brit prefix indicating the presence of two additional oxygen atoms in an organic compound

Dioxyaminoethylene Perchlorate. See Ethylenedioxyamine Perchlorate (Abbrn EDAP)

Dioxybenzene. One of the names for Benzoquinone, which is described in Vol 2, pB79-R

Dioxydiphenyl. A Ger name for Biphenol, which is described in Vol 2, pB122

1,1'-Dioxy-[ditetrazolyl-(5,5')]. A Ger name for 1,1'-Dihydroxy-(5,5'-bis-tetrazole), described in Vol 2, pB148-L

DIPAM. Navy code name for 3,3′-Diamino-2,4,6,2′,4′,6′-hexanitrobiphenyl described in this Vol, pD1133-R. See also S.A. Moses, Ordn **56,** 357 (1972)

Dipenta. Code name for Dipentaerythritol Hexanitrate, abbr as DPEHN

Dipentaborylmethane, $B_5H_8CH_2B_5H_8$; mw 138.35; obtd in a yield of less than 1% by reaction of pentaborane with dichloromethane in the presence of $AlCl_3$. None of the desired product was isolated

Boron hydrides (See Vol 2, pB253-R) are of interest as high-energy fuels

Its prepn by Friedel-Crafts reaction is described by A.C. Antoine in the National Advisory Committee for Aeronautics Report RM **E58A14a,** April (1958)

DIPENTAERYTHRITOL AND DERIVATIVES

Dipentaerythritol (DPE), $(HO.H_2C)_3C.CH_2.O.CH_2.C(CH_2OH)_3$. It is actually a dehydrated combination of two mols of PE (pentaerythritol),

$(HO.H_2C)_3C.CH_2O\boxed{H.HO}H_2C.C(CH_2OH)_3$;

mw 254.28; white crysts, sp gr 1.365 at 15°, mp 221°; insol in acet, benz, paraffin oils and fats; soly in w – 0.12g in 100g at 15° and 11.64g at 100°; soly in alc – 0.07g at 0° and 0.32g at 100°; sl sol in glycerin (Refs 1–17). Its heat of combstn at const vol and at 18° 1315.8kcal/mol and heat of formation at const vol 371.8kcal/mol and at const pressure 380.2kcal/mol (Ref 19)

DPE can be obtd as a by-product in the manuf of PE, $C(CH_2OH)_4$, by condensation of formaldehyde with acetaldehyde in the presence of Ca hydroxide

The following reactions take place:

$$CH_3CHO + 3HCHO \rightarrow C(CH_2OH)_3CHO$$

$$C(CH_2OH)_3CHO + HCHO + \tfrac{1}{2}Ca(OH)_2 \rightarrow C(CH_2OH)_4 + \tfrac{1}{2}Ca(HCOO)_2$$

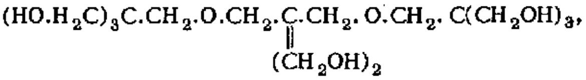
The highest yield (ca 15%) of DPE can be obtd if 3 mols of formaldehyde are reacted with 1 mol of acetaldehyde and 0.5 mol of Ca hydroxide

The procedure of prepn of PE contg DPE with small quantities of polypentaerythritols, such as TPE (tripentaerythritol) are essentially as follows:

In a stainless steel "reactor" are introduced 3 mols of formaldehyde (of 37% strength) and about 25% of its volume of water. After cooling the soln to 15–20° (with the aid of a jacket), are added gradually, with stirring, 0.5 mole of Ca hydroxide in an aq soln. Then, under the surface of liquid, is introduced 1 mole acetaldehyde, while vigorously stirring and maintaining the temp at 20–25°. After addition of all acetaldehyde and allowing the mixt to remain for several hours at RT, the temp is raised to 60° and maintained until the free acetaldehyde content drops to ca 1%

Then the liquid is transferred from the "reactor" to "neutralizer" into which is introduced gradually, with stirring, such an amount of sulfuric acid that the bulk of Ca ions precipitates as Ca sulfate. As this compd is slightly sol in water, a small amount of oxalic acid is added to complete the pptn of Ca ion. After this the slurry is filtered, the filtrate evaporated to a sp gr 1.270 and is chilled to cause crystallization of impure PE. Then the slurry is transferred to a "Nutsch" or to a centrifuge (wringer) to separate the crysts of impure PE from the mother liquor, which must be saved because it contains DPE and other PolyPE's such as TPE (tripentaerythritol) and TePE (tetrapentaerythritol)

The material left in the Nutsch or in a wringer known as "filter cake" is removed, dried and its mp determined. It is usually ca 180° which is much lower than that of pure PE (mp 262–263°), because this filter cake contains ca 15% of PolyPE's of DPE which is the principal product, while **TPE** (tripentaerythritol) is in smaller proportion. This product was isolated by Burrell (Ref 9). Its formula is

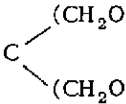


$$(HO.H_2C)_3C.CH_2.O.CH_2.\underset{\underset{(CH_2OH)_2}{\|}}{C}.CH_2.O.CH_2.C(CH_2OH)_3,$$

white crysts, mp 242–248°. It gives on nitration TPEON (Tripentaerythritol Octanitrate), $(O_2NO.H_2C)_3.C.CH_2.O.CH_2.C(CH_2.ONO_2)_2-CH_2.C(CH_2ONO_2)_3$

Crude PE (filter cake) can be used (without purification) for manuf of resins, plastics, etc; in its nitrated form it can be used for manuf of blasting explosives

Crude PE cannot be used for manuf of expls suitable for military purposes, such as PETN (Pentaerythritol Tetranitrate), $C(CH_2ONO_2)_4$, or PETrN (Pentaerythritol Trinitrate), called **Petrin**,



$C(CH_2OH)(CH_2ONO_2)_3$. These expls require a PE of at least 99% purity

Note: In our opinion, abbr PETN should be used for Pentaerythritol Trinitrate, while for Pentaerythritol Tetranitrate it is more correct to use PETeN

Purification of "filter cake" (crude PE) can be accomplished by dissolving it in an equal weight of hot water contg some HCl, adding some activated charcoal and filtering, while still hot. Cooling of filtrate produces crysts of pure PETN (1st crop) which are separated by filtration. The resulting filtrate is concentrated and then cooled. This produced the 2nd crop of crysts. In a similar manner a 3rd crop of crysts could be obtd

The "mother liquor", obtd after filtering in the Nutsch or in the wringer the slurry to separate the crude PE in the form of "filter cake", is transferred to the "recovery apparatus". Here the liquor is condensed to a thin syrup and cooled to cause crystallization of DPE and of other PolyPE's

DPE was first prepd and investigated in Germany by Friederich and von Brün (Refs 2 & 3), who found that on nitration of this product an explosive which was less powerful but brisant as PETN was obtd

Beginning about 1940 DPE and other PolyPE's were prepd in the US and GtBritain. It was found that they are useful not only in nitrated form, but also straight in manuf of resins, plastics and for drying oils (to replace the imported tung oil) in paints and varnishes

Following is a list of papers published during and after WWII on prepn and props of DPE and of other Polypentaerythritols:

Wyler & Wernett (Refs 4 & 5) investigated the props of commercial PE contg up to 15% of DPE as impurity and found that PE and DPE can form at least two double compounds in aqueous solns. One of these (compn is not given) is unstable and decomposes by water at 65–70°, while the other, consisting of ca 30% DPE and 70% PE is very stable and does not decomp in boiling water. It melts at 185–190°

For prepn of stable complex, 1 part of crude PE (known as "filter cake") was mixed with 2 parts of warm water and stirred with a small amount of activated charcoal. After filtering off the charcoal, the clear filtrate was evaporated at 65–70° until a relatively thick mush of nearly pure PE crystals precipitated. The slurry was discharged into a centrifuge heated at 70° and whizzed to a low moisture content. After removing the mother liquor and washing the crysts of PE in the centrifuge with warm w or with propanol, they were dried and tested (mp above 250°). Then the mother liquor was cooled until the elongated crysts of double-compound formed. After separating the crysts by filtration, followed by washing with warm w and drying, the product was analyzed and proved to correspond to the formula:

$[(CH_2OH)_3C.CH_2.O.CH_2.C(CH_2OH)_3] + [4C(CH_2OH)_4]$

This stable compd was found to be suitable for prepn of esters and resinous compositions for plastics, or it could be nitrated for conversion into a stable explosive of definite composition, which is described here under Dipentaerythritol Hexanitrate

Bried (Ref 6) described his method of isolation of DPE from mixts with PE

Walker (Ref 7) described prepn and props of DPE

Wyler (Ref 8) patented a method of separation of DPE from PE based on a change in the solubility of PE and DPE in a syrupy liquid obtd in the prepn of PE from CH_3CHO, HCHO and Ca hydroxide. On dehydrating this liquor by adding butyl alcohol, followed by distilling off the azeotropic mixture of $BuOH+H_2O$, a thick syrup was obtd. On cooling it, a large crop of fairly pure crysts of PE separated out. The mother liquor was evaporated to remove BuOH and this left a syrup consisting of DPE (and of other PolyPE's) and a small amt of PE. This syrup can be used as such for manuf of lacquers, resins, etc, or it can be nitrated to produce expls suitable for blasting purposes

Burrell (Ref 9) stated that technical DPE was manufd in the USA by Heyden Chem Corp, Garfield, New Jersey, by the alkaline condensation of formaldehyde and acetaldehyde, modifying the proportions and conditions of reaction so as to obtain a maximum yield of DPE with as little as possible of other PolyPE's and of PE. Their product with trade name **Dipentek** contained ca 10% of impurities. Its properties were accdg to Ref 17: free flowing powder of slightly off-white color; sp gr 1.33 at 25/4°, bulk density 0.55g/cc, mp 212–220°, ash content 0.1%, moisture 0.5%; stable in air and does not seem to be a hazard to health; used in protective coatings

Burrell prepd, in addition to DPE of mp 219–222°, TPE of mp 242–248°, a product of mp 230–240°, which proved to be a mixture of DPE & TPE with probably TePE (tetrapentaerythritol). He named this mixture the **Pleopentaerythritol**. Esterification of these technical Polypentaerythritols, (called by Heyden Co the **Polypenteks**) by heating with fatty acids (such as linseed) in the presence of alcohols (such as erythritol) produced drying oils, suitable as a replacement of tung oil in paints and varnishes

Wyler (Ref 10) patented the product obtd by nitration of PolyPE's for use in commercial expls

Burrell & Neidig (Ref 11) described various uses of pentaerythritols

Remensnyder et al (Ref 12) described prepn of a mixture contg 38.5% DPE, 29.9% TPE & 10.2% PE (based on acetaldehyde) by adding gradually during 1 hour at 40–43° a mixture consisting of 627 parts of 43.1% HCHO soln and 88 parts of liquid acetaldehyde to a mixt of 69.7ps of 43.1% HCHO soln, 432ps of 32% NaOH soln and 272ps of PE. After heating the mixt for 2hrs at 58°, it was cooled to 25°, neutralized to pH 8 with formic acid and adjusted to sp gr of 1.100. The resulting slurry was filtered and the 1st crop of crysts was collected. The filtrate was concentrated to sp gr 1.270, cooled and filtered to obtain the 2nd crop of crysts

The same investigators (Ref 13) patented a modification of previous process, intended to increase the yield of PolyPE and to decrease the amount of PE

Wyler (Ref 14) patented a method of DPE nitration

Marrian (Ref 15) stated that there is no record in the literature of direct synthesis of DPE and that its only known method of prepn is as a by-product of the manuf of PE

Marrian & McLean (Ref 16) patented in GtBritain a method of prepn of DPE from PE

Berlow et al (Ref 20) described prepn and some props of DPE

Heyden Chem Corp (Ref 17) described props of their technical DPE called *Dipentek*

Médard & Thomas (Ref 19) listed for DPE heat of combustion and heat of formation (See above)

CondChemDict (Ref 21) gives a brief description of Dipentek

Urbański (Ref 22) discusses the prepn & props of DPE, its hexanitro deriv and its eutectic mixt with PE

Walker (Ref 18) briefly mentions DPE, it eutectic with PE, and a process for separating PE from mixts with DPE, developed by Wyler & Wernett

Accdg to information obtd from the late Mr George D. Clift, the Trojan Powder Co of Allentown, Pennsylvania used to manuf technical DPE which contained small amounts of PE and TPE. The Co also used to manuf PolyPE, which consisted essentially of TPE with a small amt of DPE

Mixtures of DPE and PE have the following melting points: 100/0–DPE/PE 221°, 90/10 214°, 80/20 209°, 70/30 203°, 60/40 197°, 50/50 194°, 40/60 191.5°, 30/70 189°, 20/80 211°, 10/90 235° and 0/100 260°. By plotting these values on a graph, it is possible to obtain a chart suitable for approximate estimation of compn of mixtures on knowing their mp's

DPE gives on nitration a powerful expl DPEHN (Dipentaerythritol Hexanitrate), which is described below

Refs on DPE: 1) Beil, not found 2) W. Von Brün, "Ueber den Pentaerythrit", W. Greven, Krefeld, Germany (1930) (A book of 90pp) 3) W. Friederich & W. von Brun, Ber **63**, 2681 (1930) 4) J.A. Wyler & E.A. Wernett, USP 2251236(1941) & CA **35**, 7195(1941) 5) Ibid, USP 2299048(1942) & CA **37**, 1725(1943) 6) E.A. Bried, USP 2325589(1943) & CA **38**, 381(1944) 7) J.F. Walker, "Formaldehyde", Reinhold, NY (1944), pp115 & 151–52 8) J.A. Wyler, USP 2360186(1944) & CA **39**, 948(1945) 9) H. Burrell, IEC **37**, 86–9 (1945) 10) J.A. Wyler, USP 2389228 (1945) & CA **40**, 1036(1946) 11) H. Burrell & C.P. Neidig, "Pentaerythritol, Uses", Burrell & Neidig, NY(1946) [A book of 443pp, reviewed in C&EN **25**, 1147(1947)] 12) J.P. Remensnyder, P.I. Bowman & R.H. Barth, USP 2441597(1948) & CA **42**, 6844(1948) 13) Ibid USP 2441944(1948) & CA **42**, 6845(1948) 14) J.A. Wyler, USP 2448566(1948) & CA **43**, 1435(1949) 15) S.F. Marrian, ChemRevs **42**, 197–98(1948) 16) S.F. Marrian & A. McLean, BritP 615370(1949) & CA **43**, 5036(1949) 17) Heyden Chemical Corp (now called Heyden Newport Chemical Corp, Heyden Chemical Div, New York, NY 10017 Tech Bull **PE-2**(March 1950) 18) J.F. Walker, "Formaldehyde", Reinhold, NY, 2nd edit (1953), pp221–22 & 473 19) L. Médard & M. Thomas, MP **38**, 47–48(1956) 20) Evelyn Berlow, R.H. Barth & J.E. Snow, "The Pentaerythritols", Reinhold, NY(1958) 5–6, 16–17, 33–34, 39, 126 & others 21) CondChemDict (1961), 404-R 22) T. Urbański, "Chemistry and Technology of Explosives, Macmillan, NY, Vol **2** (1965), 195–96

Dipentaerythritol, Analytical Procedures

Wyler (Ref 3) described determination of DPE in PE based on crystallizing DPE from aq soln of both compds under controlled conditions. The procedure is as follows:

a) Weigh exactly 2.500g of sample (in a tared Al dish) and 2.500g of pure DPE

b) Transfer the sample to weighed (nearest 0.1g) 125-cc Erlenmeyer flask, add 60g of distilled water, shake, and heat until solution takes place

c) Allow the flask to stand for ½ hr, transfer it to a water bath at 18°C and allow it to remain overnight

d) Check to be certain that 60±0.1g of water are present

e) Place flask in a water bath at 25°, gently shake, remove stopper, and stir by means of a glass rod, the separated DPE crysts, keeping the temp at 25°

f) When the crysts are broken and the mixt has been kept at 25° for at least 10 mins, transfer the crysts to a weighed 15-ml Gooch-type, fritted glass crucible of medium porosity

g) Transfer should be made by using 40ml of denatured alcohol from a wash bottle

h) Wash the crysts with 10ml of ethyl ether, dry at 110° for 1–2 hrs, or to constant wt

i) Calculation % DPE:

$$\% \text{ DPE} = \frac{A-2.500+B}{2.500} \times 100$$

where: A = wt of dry crysts obtd
B = solubility correction, or 0.145gm

A summary of analytical methods for DPE & TPE is found in Ref 5

Refs — Analytical Procedures for DPE:

1) J.F. Walker, "Formaldehyde", Reinhold, NY(1944) 2) H. Burrell & C.P. Neidig, "Pentaerythritol, Uses", Burrell & Neidig, NY(1946) 3) J.A. Wyler, IEC (Anal Ed), **18**, 777–78(1946) & CA **41**, 863(1946) 4) J.H. Jaffe & S. Pinchas, AnalChem **23**, 1164–65(1951) & CA **46**, 384(1952) (Detn of DPE in presence of PE by IR spectroscopy) 5) K. Namba et al, JIndExplsSoc, Japan **15**, 282–89(1954) & CA **49**, 11282–83(1955) (Quantitative analysis of PETN in mixts with DPE by benzal method) 6) Evelyn Berlow, R.H. Barth & J.E. Snow, "The Pentaerythritols", Reinhold, NY(1958), 43–44 7) StdMethodsChemAnalysis **2B**, 6th edit (1963), not found

Dipentaerythritol Hexanitrate (DPEHN) or Nitrodipenta,

$[(O_2NO)H_2C]_3C.CH_2.O.CH_2.C[CH_2(ONO_2)]_3$; mw 524.28, N 16.03%, OB to CO_2 minus 27.5%; wh crysts, sp gr of crysts 1.63 at 15°, density of cast 1.613 at 15°, loading density at 3000–4000psi 1.59, and of pressed at 2000kg/sq cm 1.51; mp 73.7–75°; Davis gave 72°; bp – explodes above 250°; was first prepd in 1930 by Friederich & von Brün (Refs 2–4). Nitration of DPE was conducted by using concd nitric acid and by mixed nitric-sulfuric acid

Stettbacher, in the course of his extensive work on prepn of PE and PETN, isolated DPE of mp 221° and prepd DPEHN of mp 75°. This procedure is described in his book (Ref 5, pp 173–75). He also detd some of the expl props of DPEHN such as: that its impact and friction sensitivities are lower than those of PETN and its explosion temp is about 100° higher; its detonation velocity is about 1000m/sec lower than that of PETN and power ca 20% lower than that of PETN (Ref 5, p 363)

A brief description of DPEHN is given in the books listed here as Refs 6, 10, 11, 13, 15, 18, 21, 22 & 23. Explosive properties are listed in reports of Blatt & Whitmore (Ref 9), Blatt (Ref 12) and Livingston (Ref 14). Heats of combustion and of formation are listed by Médard & Thomas (Ref 20)

Wyler (Ref 7) patented an explosive prepd by nitrating with strong nitric or mixed acid a mixture consisting of 1 part DPE and 4 parts of PE

Wyler & Wemett (Ref 8) patented the **Nitrated Double Compound of DPE and PE,** $[(O_2NO.H_2C)_3C.CH_2.O.CH_2.C(CH_2ONO_2)_3]$-$[4C(CH_2ONO_2)_4]$, mw 1788.86, N 17.23%; white solid, prepd by nitrating with strong nitric or mixed acid a double compound described in Refs 4 & 5 under Dipentaerythritol

This mixed explosive has properties intermediate between PETN and DPEHN and has the advantage of a more or less constant and definite composition

As a rule, a small quantity of DPEHN present in PETN improves its stability and sensitivity to impact and friction, while large quantities decrease the stability slightly (Ref 2)

Following mp's were reported by von Brün (Ref 2) for mixtures DPEHN/PETN:100/0 – 75°, 80/20 – 98°, 60/40 – 114°, 40/60 – 126°, 20/80 – 134°, 10/90 – 137°, 5/95 – 139° and 0/100 – 141°

If DPE is isolated by the method described under Dipentaerythritol, nitration to DPEHN can be conducted by the method patented by Wyler (Ref 16). He introduced, gradually, with stirring, 1 part of finely divided DPE into 15 parts of mixed acid consisting of sulfuric acid 55.6, nitric acid 34.0 and water 10.4, previously cooled to 10°. After stirring for 30 mins, the slurry was drowned in 4 volumes of cold water, filtered and the residue on filter washed with water and then stirred for 4 hours with 5 parts of 1% aq ammonia soln. After washing it again with w and drying, the material was weighed. The yield was 2.06 parts of DPEHN for 1 part DPE

DPEHN can also be obtd by nitrating crude PE rich in DPE using either concd nitric acid alone or a mixed nitric-sulfuric acid, followed by extraction of DPEHN by acetone

Following method of nitration of PE was used (according to the late George D. Clift) during WWII by the Hercules Powder Co at the Radford Ordnance Works, Virginia:

Into a chrome-steel, jacketed nitrator, provided with mechanical agitation, was added 460lbs of 98% nitric acid and cooled to 10° (50°F) by circulating brine thru the jacket. While the acid was stirred, 100lbs of pulverized crude PE was introduced slowly by means of a vibrator feeder, while maintaining the temp at 10°. The addition took about 50 mins and resulted in complete dissolution of PE. While continuing to stir and cool, 250 lbs of concd sulfuric acid (92–93% strength) were slowly added. Since PETN is insol in sulfuric acid, it was completely precipitated. The resulting slurry was transferred into a "Nutsch" made of acid-resisting material (chemical ware or stainless steel funnel, provided with a stainless steel perforated plate and a fine mesh screen made of chrome steel or fiberglas). When most of the acid was drained by gravity, a vacuum was applied for 5–10 mins. The spent acid was run into storage tanks for subsequent transfer to the "Acid Recovery House", while the "filter cake" was removed by an operator (wearing a gas mask) using a nonsparking metal shovel, into the "Wash Tank" contg cold water, kept in constant agitation. When all the cake was transferred, the agitation was stopped to allow the slurry to settle and the supernatant acid-water run thru a stainless steel Nutsch provided with a fiberglas filter cloth. Then the tank was refilled with water, 10 lbs of Na carbonate added, the slurry agitated for about 5 mins and the ensemble transferred to the same Nutsch. After allowing the bulk of liq to drain by gravity, suction was applied until the water content in the cake dropped to ca 18%. Then the cake was broken by means of aluminum scoops and transferred to rubber-lined and rubber-tired buggies which were wheeled to the "Neutralizer and Crystallizer House"

The "Neutralizer" of this house consisted of a closed stainless steel vessel, provided with a jacket, agitator and water-cooled reflux condenser installed above the kettle to prevent the loss of acetone

After pouring into kettle 1120 lbs of commercial acetone (98% purity), two charges of crude PETN (total ca 460 lbs) were introduced thru an opening on the top of the kettle, together with 5 lbs of Na carbonate to insure complete neutralization. After closing the opening, the content of kettle was heated to 50° (122°F) (by means of hot water circulating thru the jacket). Then the temp was raised to

59°(138°F), and immediately the soln was run rapidly thru two closed 200-mesh screens (in order to filter off any grit or other foreign matter) into the "Crystallizer", where PETN was pptd by the addition of cold water

Before removal of DPEHN from this material, it was necessary to dry it. DPEHN dissolves in acetone more readily than PETN, and this is the process used to separate these two nitrates. The crude product is treated with an equal portion (by wt) of acetone. Water is added carefully causing the residual PETN to ppt. The soln separates into two layers:
1) the upper composed of fairly hydrated acetone contg a small amt of DEPHN and
2) the lower consisting of a saturated soln of DEPHN in acetone contg a little water. The bottom layer is removed and treated with an excess of alcohol. Almost pure DEPHN is pptd (Ref 23)

It is possible to convert DPEHN to DPE by treating its soln in acetone with boiling alkaline sulfide soln in alc (Ref 23)

Médard & Thomas (Ref 20) reported that on nitration of technical DPE previously purified with acetone, they obtd DPEHN of mp 68.3°. Treatment of this product with 79% nitric acid raised the mp to 73.7°, but they were not able to prep the compound with mp 75° as was reported by Friederich & von Brün (Ref 3)

Following are explosive and some other properties of DPEHN as reported by Stettbacher (Ref 5), Blatt & Whitmore (Ref 9), Davis (Ref 11), Blatt (Ref 12), Livingston (Ref 14), Médard & Thomas (Ref 20) and Tomlinson & Sheffield (Ref 24):
Brisance by Kast Formula: 148730 vs 198940 for PETN (Ref 2)
Brisance by Sand Test: 0.4g sample initiated by Lead Azide in 200g sand bomb crushed 57.4g sand vs 62.7g crushed by PETN (Ref 24)
Detonation Rate: 7410m/sec at sp gr 1.59 vs 8340m/sec at sp gr 1.71 for PETN (Ref 5, p175 and Ref 12)
Explosion Temperature: 255°(in 5 secs) and 300°(in 1 sec), vs 225° & 244° for PETN (Ref 24)
Friction Sensitivity by Friction Pendulum Test — crackles by steel shoe and unaffected by fiber shoe; same as for PETN (Ref 24)
Heat of Combustion at C_v: 1275kcal/mol (Ref 5a); 1258.4kcal/mol (Ref 20) and 2260cal/g (Ref 24)
Heat of Detonation: 1092cal/g vs 1485 for PETN (Ref 2)
Heat of Formation: 226.7kcal/mol at C_v and 238.6kcal/mol at C_p (Ref 20)
Heat Test at 100°: % loss in first 48hrs 0.11, in 2nd 48hrs 0.10 and no expln in 100hrs (Ref 24)
Hygroscopicity — % gain 0.03 at RT (Ref 24)
Impact Sensitivity: Bruceton No 3 Machine: 50% positive 37cm vs 20cm for PETN; 2 kg Kast Apparatus 0/6 shots at 20cm and 1/6 shots at 22cm vs 10 and 12cm for PETN (Ref 12); BurMinesApparatus, 2kg wt 14cm; PicatinnyArsenalApp, 2kg wt 4 inches for 10mg sample (Ref 24)
Initiation Sensitivity — less sensitive to initiation by LA or MF than PETN or Tetryl, especially at high pressure of loading, such as 1000kg/sq cm; it is more sensitive than Tetryl at low pressures of loading such as 250 to 500kg/sq cm (Ref 24)
Power by Ballistic Mortar: 144% TNT (Ref 12) & 142% TNT (Ref 24)
Power by Trauzl Test: 380cc vs 295cc for TNT and 475cc for PETN (Ref 5); 128% TNT (Refs 12 & 23)
Solubilities: v sl sol in w; more sol in acetone than PETN; more sol in concd nitric acid than PETN; insol in concd sulfuric acid
Temperature of Explosion (Detonation): 3240° vs 3920° for PETN (Ref 2)
Thermal Stability at 135° — satisfactory (Ref 12)
Vacuum Stability at 100°: 3.7cc/40hrs
Vacuum Stability at 120°: 11+cc/40hrs
Velocity of Detonation. See Detonation Rate

It is stored in dry condition and is used as an ingredient of priming compositions (Ref 24)

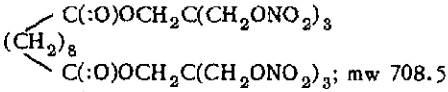
Refs for DPEHN: 1) Beil, not found 2) W. von Brün, "Ueber den Dipentaerythrit", W. Greven, Krefeld, Germany (1930) (A book of 90pp) 3) W. Friederich & W. von Brün, Ber **63**, 2681(1930) 4) Ibid, SS **27**, 73–6, 125–27 & 156–58(1932) (Explosive properties of DPEHN in comparison with PETN) 5) Stettbacher (1933), 173–75 & 363 (DPEHN) 5a) A. Schmidt, SS **29**, 262(1934) (Heat of combustion at C_v) 6) Pepin Lehalleur (1935) 218 (Hexanitrate de dipentaerythrite) 7) J.A. Wyler, USP 2086146(1937) & CA **31**, 6100 (1937) 8) J.A. Wyler & E.A. Wernett, USP 2251236(1941) & CA **35**, 7195(1941) (Nitrated double compound of DPE and PE) 9) A.H.

Blatt & F.C. Whitmore, OSRD **1085**(1942), p32 10) Clift & Fedoroff, Vol **2**(1943), p D10 11) Davis (1943), 282–83 12) A.H. Blatt, OSRD **2014**(1944) 13) Pérez Ara (1945), 307 (Hexanitrato de dipentaeritrita) 14) S. Livingston, PATR **1561**(1945) (Characteristics of Explosives HMX and DPEHN) 15) Vivas, Feigenspan & Ladreda **2**(1946), 69 (Hexanitrato de dipentaeritrita) 16) J.A. Wyler, USP 2437582(1948) & CA **42**, 4350 (1948) 17) S. Fujii, RevPhysChemJapan, ShinkichiHoribaCommem Vol **1946**, 153–57 & CA **44**, 1906(1950) 18) J.F. Walker, "Formaldehyde", Reinhold, NY (1953), 473 19) K. Namba et al, JIndExplsSocJapan **15**, 282–89(1954) & CA **49**, 11283(1955) 20) L. Médard & M. Thomas, MP **38**, 48(1956) 21) Evelyn Burlow, R.H. Barth & J.E. Snow, "The Pentaerythritols", Reinhold, NY (1958), pp55, 59 & 64 22) Ye.Yu. Orlova, "Khimiya i Tekhnologiya Brizantnykh Vzryvchatykh Veshchestv", GosIzdatOboronProm, Moskva (1960), Engl Translation "Chemistry and Technology of High Explosives", by Technical Documentation Office, MCLTD, WP-AFB, Ohio, MCL-844/1+2 (1961), Pt **11**, pp 641–42 23) T. Urbański, "Chemistry and Technology of Explosives", Macmillan, NY, Vol **2** (1965), p195–96 24) W.R. Tomlinson, Jr & O.E. Sheffield, "Properties of Explosives of Military Interest", PATR **1740**, reissued in revised form as AMCP (Army Materiel Command Pamphlet) **706–177**, March 1967, pp119–21 & **Jan 1971**, pp119–21 25) Anon, "Military Explosives", **TM 9–1300–214** & **TO 11A–1–34**, November 1967 (DPEHN is not listed)

Dipentaerythritol Hexanitrate
Analytical Procedures and Physical Tests. There are no US Specifications describing procedures and tests for analyzing DPEHN. It is suggested that physical tests described in Ref 1 and in Vol 1 of Encycl, pp VIIff be used when necessary to study DPEHN

Refs – Analytical Procedures for DPEHN: 1) L.C. Smith & E.C. Eyster, "Physical Testing of Explosives", Part III-Miscellaneous Sensitivity Tests; Performance Tests, OSRD Rept **5746**, Dec 1945 2) P. Aubertein & H. Pascal, MP **40**, 123(1958) 3) W.E. Fredericks & Frank Pristera, "Infrared Method for Determining Small Amounts of Dipentaerythritol Hexanitrate in Pentaerythritol Tetranitrate", Picatinny Arsenal, FREL Technical Notes **30**, April 1959, Project TB 3-0115B and 5A04-10-006 4) StdMethodsChemAnalysis **2B**, 6th edit (1963), not found

Dipentaerythritol Trinitrate Sebacate,



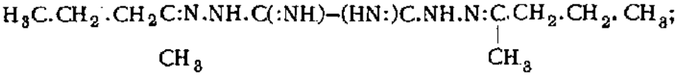
; mw 708.51, N 11.86%; viscous clear syrup; was obtd when sebacic acid chloride & PE trinitrate were mixed and heated to 50°. There was a fairly vigorous evolution of HCl. Reaction mixt was then heated 4 hrs at 50–60° and allowed to stand overnight. The material was dissolved in acetone & repptd with water as an oil which was separated, dissolved in methylene chloride, and dried over calcium sulfate. Pentane was added and the soln alternately chilled & warmed but no cryst product was obtd. Finally, the soln was evaporated under a stream of air (Ref 2)

This deriv of PE may have application in the field of expls

Refs: 1) Beil, not found 2) D.L. Kouba et al, Hercules Monthly Prog Rept, "High Explosives", 4 Jan 1952 (Navy Contract NOrd-11280, Task A) 3) Not found in later refs thru 1966

Di(pentafluorosulfur)-peroxide. See Bis(pentafluorosulfur)-peroxide in Vol 2, pB152-R

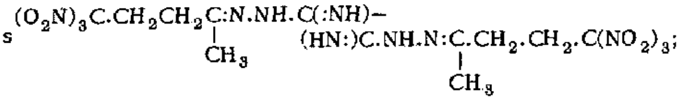
Di(2-pentanone)-cyanohydrazone and Derivatives
Di(2-pentanone)-cyanohydrazone or *Bis(methylpropylketone)-cyanohydrazone,*



$$H_3C.CH_2.CH_2\underset{CH_3}{C}:N.NH.C(:NH)\text{–}(HN:)C.NH.N:\underset{CH_3}{C}.CH_2.CH_2.CH_3;$$

mw 252.36, N 33.30%; may be considered as the parent compd of its hexanitro deriv, although not used to prep it:

Di(or Bis)(5,5,5-trinitro-2-pentanone)-cyanohydrazone or **Diiminooxalic Acid Bis(5,5,5-trinitro-2-pentylidene Dihydrazide,**



$$(O_2N)_3C.CH_2CH_2\underset{CH_3}{C}:N.NH.C(:NH)\text{–}(HN:)C.NH.N:\underset{CH_3}{C}.CH_2.CH_2.C(NO_2)_3;$$

mw 522.36, N 32.18%; pale-yel solid, mp 112–18°, burns with a flash when ignited, detonated by impact of a hammer; was prepd when a soln of diiminooxalic acid dihydrazide, $[NH_2NHC(:NH).C(:NH).NH.NH_2]$, in hot water was added to a soln of 5,5,5-trinitro-2-pentanone, $[(O_2N)_3C.CH_2.CH_2C(:O).CH_3]$, in absolute alcohol and the suspension cooled, filtered and washed with abs alc (Ref 3)
Refs: 1) Beil, not found (Parent) 2) Beil, not found (hexanitro deriv) 3) I.J. Schaffner, 5th USRubberRept, "Synthesis of New Explosives and Propellants", 1 Oct 1948 – 1 Feb 1949 (Contract Nord 10129), p43

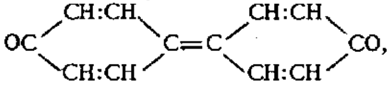
Dipenthrit. One of the names for DiPEHN

Diphenic (or Biphenic) Acid. See Biphenyldicarboxylic Acid in Vol 2, pB125-R

Diphenol or Dihydroxybiphenyl. See Biphenol in Vol 2, pB122-L

Diphenoquinone or Bibenzenone and Derivatives

4,4'-Diphenoquinone,



mw 184.18; golden-yel ndls or brick-red ndls (from benz), both modifications dec at 165°, nonvolatile & odorless; insol in cold w; v sl sol in alc & chlf; almost insol in hot benz; sol in warm nitrobenz giving a deep yel color; other props & methods of prepn in Ref 1

Tetraazido-4,4-diphenoquinone,
$O:C_6H_2(N_3)_2{=}C_6H_2(N_3)_2:O$, mw 318.25, N 51.21%; bk violet solid which exploded at 30°; explodes also by friction and impact. Was prepd by agitating during 18 hours a suspension of tetrabromodiphenoxyquinone in AcOH with an excess of Na nitrite. The resulting product, presumed to be tetrazido compd, was washed with AcOH, followed by alc and ether until the filtrate became colorless
Refs: 1) Beil **7**, 740 & {3766} (Parent compd) 2) Beil **7**, [664] (Tetraazido) 3) A. Korczyński & St. Namyslowski, BullFr [4] **35**, 1186–94(1924) & CA **19**, 644(1925)

1,2-Diphenoxy-ethane. See Bis (phenyl)-ethyleneglycol Ether in Vol 2, p B153-R
Insert on p B154-L after Ref 4:
5) W.B. Whalley, JCS **1950**, 2242 & CA **45**, 3347(1951)

Diphenyl. Same as Biphenyl described in Vol 2 of Encycl, p B122-R

N,N'-Diphenylacetamide. Same as *Acetyldiphenylamine* listed in Vol 1, pp A57-R to A58-L, but without giving its: mw 211.25; rhombic crysts, mp 103°, bp – sublimes; sl sol in w & in eth; sol in alc (Lange)

Its azido-, mononitro-, dinitro-, trinitro-, tetranitro- and hexanitro- derivatives are described on pp A58-L to A58-R of Vol 1

Diphenylacetamidine and Derivatives
N,N'-Diphenylacetamidine or *Ethenyldiphenylamidine*, $CH_3C(NH.C_6H_5):N.C_6H_5$, mw 210.27, N 13.32%; ndls (from alc), mp 132–33°; insol in w; sol in eth & hot alc. It can be prepd by treating a cold mixt of 6 moles aniline & 3 moles acetic acid with 2 moles of PCl_3 and heating to 160°. This compd forms crystalline org & inorg salts (Ref 1). See also Ref 6 for prepn

Azido, $C_{14}H_{13}N_5$, and *Diazido*, $C_{14}H_{12}N_8$, derivs were not found in the literature thru 1966

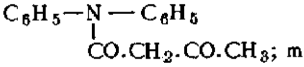
N,N'-Bis(4-nitrophenyl)-acetamidine or **N,N'-Di(p-nitrophenyl) -acetamidine,**
$O_2N.C_6H_4.N:C(CH_3).NH.C_6H_4.NO_2$; mw 300.27, N 18.66%; yel ndls (from petr eth), mp 131° (Ref 2) & (from alc), mp 262–64° (Ref 6); sol in hot w; was obtd with other products when 4-nitroaniline hydrochloride was heated with diacetamide at 200–10° (Refs 2 & 3). See Refs 4 & 6

It forms a *Nitrate* salt, $C_{14}H_{12}N_4O_4{+}HNO_3$, powd which dec at 182° without melting; insol in w, alc, acids & alkalis
Refs: 1) Beil **12**, 248, (195) & [144] (Parent compd) 2) Beil **12**, 720 & [391] (Dinitro deriv) 3) K. Brunner et al, Monatsh **48**, 123(1927) & CA **22**, 222(1928) 4) W. Bradley & I. Wright, JChemSoc **1956**, 646 & CA **50**, 13806(1956) [Rept yel ndls (from alc), mp 261–62° (softens

at 252°); prepd when p-nitroacetanilide was heated 2 hrs at 120° with PCl_5 and p-nitroaniline added and heated at 140° for 3 hrs] 5) R.H. DeWolfe & J.R. Keefe, JOrgChem **27**, 494(1962) & CA **57**, 727(1962) (Acid-catalyzed hydrolysis in 20% dioxane soln) 6) E.C. Taylor & W.A. Ehrhart, JOrgChem **28**, 1111–12 (1963) & CA **58**, 11270–71(1963) (Prepn by refluxing for 2 hrs at 140° a mixt of ethyl orthoacetate, pyridine, p-nitroaniline & glacial acetic acid)

NOTE: No higher nitrated derivs of N,N'-Bis (4-nitrophenyl)-acetamidine were found in the literature thru 1966

N,N-Diphenylacetoacetamide or **Acetoacetyldiphenylamine,**



$C_6H_5—\underset{CO.CH_2.CO.CH_3}{\underset{|}{N}}—C_6H_5$; mw 253.29, N 5.53%; col crysts (turning brown in the light), mp 81.5°; very sol in acet, et acet, benz, chlf & CS_2; sl sol in alc and gives a whitish emulsion with w. It can be prepd by interaction of diphenylamine, $C_6H_5.NH.C_6H_5$, and acetylketene, $CH_3COCH{:}CO$ (Ref 2)

Its heat of combstn at C_v 1965.06kcal/mol and at C_p 1966.36; heat of formation at C_v 53.25 and C_p 58.57 (Ref 3)

Was suggested by Tavernier & Lamouroux as an ingredient of propellants (Ref 3)

Refs: 1) Beil, not found 2) R.N. Lacey & E.E. Connolly, BritP 715896(1954) & CA **49**, 13290(1955) 3) P. Tavernier & M. Lamouroux, MP **38**, 76 & 84(1956)

Di(or Bis)phenyl-acetone and Derivatives

a,a'-Di(or Bis)-phenyl-acetone or *Dibenzylketone,* $C_6H_5.CH_2.CO.CH_2.C_6H_5$, mw 210.26; crysts (from aq alc), mp 34–35°, bp 330.6°; sol in petr eth, alc & carbon tetrachloride; can be prepd by heating α-phenyl-α'-benzyl-ethyleneglycol with aq sulfuric acid, or by heating α-phenyl-α'-benzyl-ethyleneoxide with either 50% sulfuric acid or with zinc chloride (Refs 1 & 5)

a,a'-**Di(or Bis)-(4-nitrophenyl)-acetone** or **Bis-(4-nitrobenzyl)-ketone,** $O_2N.C_6H_4.CH_2.CO.CH_2.C_6H_4.NO_2$; mw 300.26, N 9.33%; yel crysts (from glacial acet acid), mp – dec 205–06°; sol in benz; v sl sol in methanol, ethanol & petr eth; was prepd by heating 4,6-dioxo-3,5-bis(4-nitrophenyl)-2-(4-nitrobenzyl)-5,6-dihydro-γ-pyran with 20% KOH in a tube at 160°. Its phenylhydrozone deriv dec at 110–12° (Ref 2)

a,a-**Di(or Bis)-[2,4-dinitrophenyl]-acetone,** $[(O_2N)_2C_6H_3]_2CH.CO.CH_3$; mw 390.26, N 14.36%; yel ndls (from ethyl acetate), mp – dec 183°; sl sol in alc, glac acet ac & chlf; mod sol in hot ethyl acet; sol in warm alc NaOH with an intensive blue-violet color; was prepd by reaction of 2,4-dinitrophenylacetone, Na ethylate & 4-bromo-1,3-dinitrobenzene in alc (Refs 3 & 4)

Refs: 1) Beil **7**, 445, (238), [382] & {2160} 2) Beil **7**, 447 & (238) 3) Beil **7**, 448 4) W. Borsche, Ber **42**, 1316(1909) 5) J. Lévy & F. Gombinska, CR **188**, 713(1929) & CA **23**, 2708(1929) 6) No tetranitro derivs found in later refs thru 1966

Diphenylacetonitrile and Derivatives

Diphenylacetonitrile or *Dephenatrile,* $(C_6H_5)_2CH.CN$, mw 193.24, N 7.25%; yel crysts, mp 73–76°; insol in w; v sol in alc; can be prepd by reaction of borofluoride with a mixt of benzaldehyde, HCN & benz (Refs 1 & 3). Used as an herbicide (Ref 4)

p-Nitrophenyl-phenylacetonitrile, $O_2N.C_6H_4.CH.CN.C_6H_5$; mw 283.24, N 11.76%; crysts (from alc), mp 70–72°; was prepd by reaction of phenylacetonitrile with 4-chloro-1-nitrobenzene & KOH in pyridine with exclusion of air (Ref 2). No expl props are reported

Nitro-diphenylacetonitrile, $(C_6H_5)_2C(NO_2).CN$; mw 283.24, N 11.76%; crysts (from MeOH), mp 44–46°, dec on heating above 70°; was prepd by treating a soln of tetraphenylsuccinic acid dinitrile in chlf with nitrogen dioxide under anhydrous conditions (Ref 2)

NOTE: No higher nitrated derivs of Diphenylacetonitrile were found in the literature thru 1966

Refs: 1) Beil **9**, 674, (282), [469] & {3304} 2) Beil **9**, {3314} 3) J. Mills, USP 2447429 (1948) & CA **43**, 6666(1949) 4) CondChemDict (1961), 405-R

Diphenylacetylene and Derivatives

Diphenylacetylene or *Tolane*, $C_6H_5.C{\vdots}C.C_6H_5$; mw 178.22; monocl prisms, sp gr 0.966 at 100°/4, mp 60–62°, bp 300°; insol in w; v sol in eth; v sl sol in hot alc; can be prepd by heating β,β-dichloro-α,α-diphenylethylene with 2 moles of Na methylate, and by other methods (Ref 1)

Dinitrodiphenylacetylene, $C_{14}H_8N_2O_4$; mw 268.22, N 10.45%. The following isomers are found in the literature:

2,2'-Dinitro-diphenylacetylene, $O_2N.C_6H_4.C{\vdots}C.C_6H_4.NO_2$; yel ndls (from alc or glac acet ac), mp 189–93°; sol in alc, benz, pyridine & acet; mod sol in glac acet acid & aromatic hydrocarbons; sl sol in hot alc & petr eth (Ref 2, p330)

3,4'-Dinitro-diphenylacetylene, $O_2N.C_6H_4.C{\vdots}C.C_6H_4.NO_2$; crysts (from alc), mp 173–174°, sol in hot benz, nitrobenz, acet & ethyl acetate; sl sol in glac acet acid, chlf & alc; v sl sol in CCl_4, CS_2, eth & petr eth (Ref 2, p569)

4,4'-Dinitro-diphenylacetylene, $O_2N.C_6H_4.C{\vdots}C.C_6H_4.NO_2$; yel pltlts or ndls (from glac acet ac or alc), mp 207–11°; sol in concd sulfuric acid with a yel soln; sl sol in hot alc & glac acet acid; v sl sol in eth (Ref 2, pp 657 & 320). See Ref 3

2,4-Dinitrophenyl-phenyl-acetylene, $C_6H_5.C{\vdots}C.C_6H_3(NO_2)_2$; yel ndls (from glac acet ac), mp 112–112.5°; sol in pyridine, chlf, benz, hot glac acet ac & hot alc; its soln in concd sulfuric acid gives a blue-violet color (Ref 2, p320). See Ref 5

X,X-Dinitro-diphenylacetylene, $C_{14}H_8N_2O_4$, crysts (from glac acet ac), mp 234°(Ref 2, p569)

3,3'-Dinitro-diphenylacetylene or *Bis(m-nitrophenyl)-acetylene*, $O_2N.C_6H_4.C{\vdots}C.C_6H_4.NO_2$; no description of compd is given in abstract (Ref 4)

Other props & methods of prepg the Dinitro derivs are found in the Refs. No expl props are reported

NOTE: No higher nitrated derivs of Diphenylacetylene were found in the literature thru 1966

Refs: 1) Beil **5**, 656, (319) & [568] 2) Beil **5**, 657, (320) & [569] 3) L.M. Litvinenko et al, ZhObshKhim **28**, 2046(1958) & CA **53**, 2154 (1959) (Prepn of 4,4'-Dinitro deriv, mp 214°) 4) H. Sinn, MonatshChem **96**(3), 1036(1965) & CA **63**, 8150(1965) 5) J.J. Pappas & E. Gancher, JOrgChem **31**(4), 1289(1966) & CA **65**, 3899(1966) [Prepn of 2,4-Dinitrophenylphenylacetylene, crysts (from MeOH), mp 117–18°]

DIPHENYLAMINE AND DERIVATIVES

Diphenylamine (DPhA or DPA) or Phenylaniline, $C_6H_5.NH.C_6H_5$, mw 169.22, N 8.28%, OB to CO_2 minus 27.9%; col monocl crysts (from ligroin) with an aromatic odor and bitter taste; sp gr 1.160 at 20° & 1.054 at 61°; mp 52.9°, bp 302°; sl sol in w; sol in alc, eth, benz & ligroin. Being slightly basic, DPhA forms salts with acids. It is very toxic, acting both thru the skin and thru the organs of respiration & digestion (Refs 16, 22 & 31). Its 1% soln in concd sulfuric acid serves as a sensitive colorimetric reagent for nitrates. The test is described as item E in Vol 3 of Encycl, pp C405-R and C406-L

Vapor pressures of DPhA at various temps are listed in Ref 28

Preparation and properties of DPhA are described in Refs 1, 3, 9, 12, 13, 16, 19, 20, 21, 21a, 22, 28, 30 & 33

Here is a brief description of several methods of preparation

Accdg to Refs 1 & 22, DPhA was first prepd in 1864 by A.W. Hofmann on heating rosaniline (fuchsin carbinol base), $(CH_3.C_6H_3.NH_2)COH(C_6H_4NH_2)_2$. Better results were obtd by the condensation of aniline and phenol in the presence of Zn chloride catalyst in an autoclave at 200–230°:

$$C_6H_5NH\boxed{H+HO}C_6H_5+(ZnCl_2) \rightarrow$$
$$H_2O+C_6H_5NH.C_6H_5+(ZnCl_2)$$

Lab method of prepn, described by Pérez Ara (Ref 22, p581), consists of heating a mixt of 70g aniline and 70g of aniline hydrochloride in an autoclave at 200° for several hours, followed by cooling. The resulting mass was mixed with 100ml of distd w and 100ml HCl, boiled for 30mins, cooled and decanted. Purification was done by washing with hot w, followed by 5% Na carbonate soln, once more with w and then dried. Chemically pure product can be obtd by distillation in vacuo. Another method of purification consists of 4–5 successive recrystallizations from a mixture of ethanol & ligroin, 1:12 by volume, followed by drying in vacuo over phosphorus pentoxide for 5hrs

CondChemDict (Ref 30) lists the autoclave heating of equal formula weights of aniline and aniline hydrochloride, followed by boiling the crude product with dilute HCl to remove the unaltered aniline and then distilling the residue in vacuo

An industrial method of prepn consisting of heating 100kg aniline with 105kg aniline hydrochloride in an autoclave at 230° for 12 hrs is described by Pérez Ara (Ref 22, p581)

Wester et al (Ref 24) patented an industrial method of preparation of diarylamine, and more particularly of DPhA. Essentially the process comprises heating aniline in an autoclave at pressures betw 100 and 200psi, in the presence of ferric chloride catalyst in an amount betw 0.3% to 8.0% by wt of the aniline used. Yields as high as 58.8% were reported after heating for only 8 hrs at 160psi

DPhA is a non-expl compd possessing the property of absorbing and reacting with nitrogen oxides. As such N gases are forming during the storage of propellants contg NC, DPhA has found wide application in quantities of about 1% as a stabilizer in such proplnts. Accdg to Stettbacher (Ref 13, p197), A. Nobel proposed using DPhA in German proplnts as early as 1889. Then it was adopted in other European countries and also in South America. The US Army did not adopt it until 1908. Germany used it until a new group of stabilizers was developed beginning in 1906 in the laboratory of the Zentrallstelle für Wissenschaftlich-technische Untersuchungen zu Neubabelsberg (near Berlin) (Ref 9). These compds were named "Centralites" and are described in Vol 2 of Encycl, pp C126ff. Other countries than Germany have been using, to a certain extent, Centralites, but the use of DPhA has not been abandoned

Snelling (Ref 11) proposed using DPhA as a sensitizer for AN in mining expls

Action of DPhA on NC and on other organic nitrates was examined in France by several investigators such as Demougin & Landon (Ref 14), Muraour (Ref 15) and others. Muraour found that NC is attacked in storage by NC and by other nitric esters (such as PETN) with formation of mono- and poly-nitroderivatives of DPhA. When DPhA was replaced by Centralite 1-(N,N$'$-diethyl-N,N$'$-diphenylurea), which has no mobile H, no reaction occurred, and for this reason Muraour preferred Centralite to DPhA. Reactions of DPhA with nitric esters in presence of acetic acid were examined by Parpaillon (Ref 27)

Muraour (Ref 15) showed that pure NC is comparatively stable, but if, for some reason (for instance by action of heat), the decompn starts, the reaction is further accelerated by the products of decompn (N oxides). The role of a stabilizer, like DPhA, is to maintain the rate of decompn within certain limits and to prevent its acceleration. If a stabilizer is not properly chosen or if it is not well mixed in proplnt, it might form compds with NC, which instead of arresting the decompn will accelerate it. Marqueyrol's tests of proplnts with various stabilizers have shown that deceptive results may be obtd when different stabilizers are compared just at one temp. He recommends that proplnts be tested at two different temps

Dalbert (Ref 18) examined the stabilizing action of DPhA in Poudre B (see Vol 2 of Encycl, p B 1-L) and also made comparison (after storage for 16 months at 50°) of a double-base proplnt stabilized with 8.75% Centralite with two other proplnts in which 2% of Centr was replaced with either DPhA or Carbazole. Results of tests showed that Centralite alone was a better stabilizer than its mixts with DPhA & Crbz

Stability of French proplnts using DPhA was also examined by Leclercq (Ref 26a)

Pauling (Ref 23) examined changes in the percentages of DPhA in single and double-base proplnts, which have been subjected to vacuum stability tests at 80, 90, 100 and 110°

Reactions and transformation of DPhA during storage and aging of smokeless proplnts using it as a stabilizer have been studied by many investigators and results are reported in Refs 2, 4, 5, 6, 7, 8, 15, 17, 20, 21 and 22

It seems that the most probable changes in DPhA are as outlined below:

It was observed by Desmaroux (Ref 4), Marqueyrol & Muraour (Ref 5) and Marqueyrol & Loriette (Ref 6) during their studies of DPhA derivatives which impart a dark color to old proplnts, that the color is produced by impurities in ether (used in manuf of proplnt), or by the oxidizing action of air during drying and storage. Their presence is not an evidence that the proplnt has decomposed, but indicates that a certain amt of DPhA has been consumed and that, consequently, less of it remains available for use as a stabilizer

The first change which takes place in storage of DPhA contg proplnts is the formation of *Diphenyl-N-nitrosamine*, $C_6H_5.N(NO).C_6H_5$. The presence of this compd (which is as good a stabilizer as DPhA itself) was proven in all proplnts which were not too old or which had not suffered profound decompn. If any mineral acid is present it undergoes a tautomeric change with the formation of *p-Nitrosodiphenylamine*, $C_6H_5.NH.C_6H_4.NO$. This compd, a stabilizer itself, reacts with nitric acid and oxygen with the formation of *2,4'- and 4,4'-Dinitrodiphenylamines*, $(O_2N)C_6H_4.NH.C_6H_4(NO_2)$, both of which were isolated by Davis and Ashdown during and after WWI (Refs 2, 7 & 8). Finally, the last stage of change is the formation of *2,4,4'-Trinitrodiphenylamine*, $(O_2N)_2C_6H_3.NH.C_6H_4(NO_2)$

Besides the above compds, the presence of the following were also reported, especially when proplnts were stored at elevated temps, such as at 100°:

4-Nitrodiphenylamine, $(O_2N).C_6H_4.NH.C_6H_5$

4-Nitrodiphenyl-N-nitrosamine, $(O_2N).C_6H_4.N(NO).C_6H_5$

4,4'-Dinitrodiphenyl-N-nitrosamine, $(O_2N).C_6H_4.N(NO).C_6H_4(NO_2)$

2-Nitrodiphenylamine, $(O_2N)C_6H_4.NH.C_6H_5$

2-Nitrodiphenyl-N-nitrosamine, $(O_2N)C_6H_4.N(NO).C_6H_5$

2,4'-Dinitrodiphenyl-N-nitrosamine, $(O_2N)C_6H_4.N(NO).C_6H_4(NO_2)$

All of these compds were detected colorimetrically and more recently their presence was verified by the Chromatographic Method of Tswett, which is described under CHROMATOGRAPHY in Vol 3 of Encycl, pp C289-R to C298-L

Accdg to Davis and Asdown (Refs 2 & 7), the proplnt is usually nearing the end of its stable life when a *Dinitrodiphenylamine* is present and the proplnt is practically worthless (or soon becomes so) when a *Trinitrodiphenylamine* is present

The use of DPhA in proplnts contg NG is not recommended on account of the formation of tarry oxidation products (Ref 8) but, accdg to Mr E.F. Reese, formerly of PicArsn, some double-base proplnts examined in his lab contd DPhA

Owing to the basic character of DPhA, it must not be used in large quantities (Ref 19). It was proven by Marqueyrol (Ref 10) and also by Demougin & Landon (Ref 14) that DPhA attacks NC slightly even at a temp as low as 40°, but at 75° and higher temps the reaction is quite perceptible

It should be noted that DPhA used in smokeless propellants must be fairly pure and practically free of primary amines, because they attack NC more readily than DPhA

Presence of DPhA may be detected by any colorimetric tests described under Analytical Procedures (See next item)

Refs for DPhA: 1) Beil **12**, 174, (163) 2) T.L. Davis & A.A. Ashdown, IEC **7**, 674 (1915) 3) Marshall **2**(1917), 697 4) J. Desmaroux, MP **21**, 238(1924) 5) M. Marqueyrol & H. Muraour, MP **21**, 250 & 272(1924) 6) M. Marqueyrol & P. Loriette, MP **21**, 277 (1924) 7) T.L. Davis & A.A. Ashdown, JACS **46**, 1051(1924) 8) Ibid, IEC **17**, 674(1925) 9) Brunswig, Proplnts (1926), 315 10) M. Marqueyrol, MP **23**, 158–77(1928) & CA **23**, 5043(1929) 11) W.O. Snelling & J.A. Wyler, USP 1827675(1931) & CA **26**, 601(1932) 12) Marshall **3**(1932), 227 13) Stettbacher (1933), 197 14) P. Demougin & M. Landon, MP **26**, 273(1934–1935) & CA **30**, 7851(1936) 14a) G.C. Hale, ArmyOrdn **5**, 674–75(1935) 15) H. Muraour, Bull(Fr), [5] **3**, 2240–51 (1936) & CA **31**, 3697(1937) 16) Robert P. Dervillée & (?). Collet, AnnMédLegaleCriminolPoliceSci **17**, 968–74(1937) & CA **32**, 4786(1938) 17) F. Becker & G. Hunold, SS **33**, 213 & 244(1938) 18) R. Dalbert, MP **28**, 119–26 & 147–55(1938); CA **33**, 7569–70 (1939) 19) Thorpe **4**(1940), 520 20) Clift & Fedoroff **2**(1943), pp D9 to D11 21) Davis (1943), 308–13 21a) Kast-Metz(1944), 163–65 22) Pérez Ara(1945), 437 & 580–82 23) L. Pauling, "Changes in the Percentages of Diphenylamine in Single and Double Base Powders Which Had Been Subjected to Vacuum Stability Tests at 110°, 100°, 90° and 80°C", OSRD **5957** (Nov 1945) 24) A.B. Wester, L.A. Burrows & W.B. Johnson, USP 2447044 (1948) & CA **42**, 8212(1948) 25) J.G. Norvall et al, Analyst **73**, 389–93(1948) & CA **42**, 7984(1948) (Colorimetric detn of DPhA in NC powders) 26) D.A.F. Montenegro et al, Actas y Trabajos CongrSudamerQuím, 5° Congr(Lima, Peru) **1**, 391–95(1951) (in Portuguese) & CA **49**, 15241(1955) (Stabilization of NC with DPhA) 26a) R. Leclercq, MAF **25**, 635–49(1951) 27) M. Parpaillon, MP **36**, 177(1954) & CA **50**, 2173(1956) 28) T.E. Jordan, "Vapor Pressure of Organic Compounds", Interscience, NY (1954), Chap 7, pp 187 & 197; plate 14 29) J. Barlot, MP **36**, 199–207 (1954) & CA **50**, 730(1956)(Colorimetric detn of DPhA in LB, 7T & LB, and TU Powders) 30) CondChemDict (1961), p 405-R 31) Sax (1963), p 761-R 32) Kirk & Othmer, 2nd edit **2**(1963), p420 33) Urbański **3**(1967), p559 (DPA as stabilizing agent in proplnts)

Diphenylamine, Analytical Procedures and US Military Specification Requirements and Tests

The tests of Refs 1 to 9 are only listed but not described here. The tests of Buisson are briefly described in Ref 10

Davis (Ref 11, p312 gave the following qualitative test for DPhA and its products of decompn. A strip of filter paper on which an alcoholic extract of propellant is allowed to evaporate is colored blue by a drop of saturated soln of Amm persulfate in the presence of DPhA. Likewise the extract itself can be tested directly by adding several drops of Amm persulfate soln. Diphenylnitrosamine gives no color with Amm persulfate, but it imparts an intense blue coloration to a few ml of cold concd sulfuric acid. DPhNsoA gives no color with a cold 1% alcoholic soln of α-naphthylamine, but an orange color if the soln is heated. None of the other DPhA derivs which occur in smokeless proplnts gives these tests.

Pérez Ara (Ref 19, p438) describes identical tests and states that if a proplnt originally stabilized with DPhA gives negative results for the presence of both DPhA and DPhNitrosoamine, such a proplnt should be considered unfit to use because it contains no stabilizer

Many tests are described in Ref 14 and following is the abstract of some tests:

Qualitative Tests. Pulverize the proplnt to test, extract it with ether on methylene chloride (as described in MIL-STD-286A, Method 104.1 or in Vol 2 of Encycl, p C131-L) and evaporate the solvent using a stream of dry air. Place ca 1 mg of this residue on a spot plate and add a few drops of the reagent, such as described in the tests listed below

Test 1. Color Test with Concentrated Sulfuric and Dilute Nitric Acids. Add to 1 mg residue on a spot plate 3 drops of concd sulfuric acid and 1 drop of weak nitric acid (prepd by dissolving 6 drops of concd acid in 100ml dist w). The appearance of blue color indicates the presence of DPhA. The same color is obtd in presence of N-nitroso DPhA. If stronger nitric is used as indicated above, the color turns purple

Test 2. Color Test with Concentrated Sulfuric Acid and $K_2Cr_2O_7$. Add to 1 mg residue 3 drops of concd sulfuric acid and 1 mg of finely divided K dichromate. A blue color indicates the presence of DPhA or of N-nitroso DPhA

Note: If Centralite is present, tests 1 & 2 give a purple color. DPhUrethane likewise gives a purple color, but the color is only transient, remaining for 10–15 secs

Test 3. Color Test with Selenious Acid in Concentrated HCl. This test was developed at PicArsn (Ref 13) to make it suitable for testing foreign proplnts which contain stabilizers interfering in tests 1 & 2. Test 3 is rather specific for DPhA because Acardite, Centralite, DPhUrethane, EtPhUrethane, NG, DEGDN and DNT do not interfere

Add to 1 mg residue on a spot plate 3 drops of 10% selenious acid soln in concd hydrochloric acid and if the color developed is blue, DPhA and/or its nitroso is present. Dissolution of selenious acid in HCl is facilitated by heating on a water bath

Test 4. Detection of DPhA by Precipitation as the DPhA-dichloride. Treat an amt of ether extract residue, estimated to contain ca 10 mg DPhA, with HCl gas, generated in apparatus used for quantitative detn of DPhA (See Test 4). A white crystalline ppt of dichloride, having mp of 179°, is obtd when DPhA is present

Quantitative Methods.

Test 1. DPhA Nitration Method. Cut a sample of proplnt into small pieces, as described in Vol 2 of Encycl, p C131-L, under "Preparation of Sample", and weigh ca 5g, within 0.2mg, in a tared 250ml lipped beaker. Add a mixture of 10ml glac AcOH and 20ml nitric acid of density 1.42 and heat the beaker on a steam bath at 95° for 1½ hours. Cool the beaker with contents but do not agitate. Pour the liquid into 75ml of distd w cooled to 15° in a 250ml glass-stoppered Erlen flask and wash the beaker with an amt of w that will dilute the soln to 120ml. Shake the flask for 2 mins and allow to stand overnight. Filter the resulting *Hexanitrodiphenylamine* thru a Gooch prepd by washing with 10% nitric acid and igniting. Wash the ppt with 6 or 7 portions of 1% nitric acid soln, dry at 100–105° for 1 hr, cool in a desiccator and weigh. Run thru the crucible several portions of acetone until complete dissolution of nitro-compds. Dry the crucible at 100–105° for 1 hr and weigh within 0.2mg

$$\% \text{ DPhA} = \frac{\text{Wt of ppt on crucible x } 42.59}{\text{Wt of sample (ca 5g)}}$$

Note 1: The quantities of acids must be strictly adhered to

Note 2: In order to convert DPhA to its hexanitro-derivative, NC must be present in sample in quantity 1 g per each 10mg of DPhA. If NC is absent, use 20ml of fuming nitric acid in lieu of acid of sp gr 1.42

Note 3: Centralites, diphenylurea and diphenylurethane interfere with nitration test

Test 2: Gravimetric Bromination Method. The procedure of Pamphlet listed here as Ref 14 is also described in MIL-STD-286A, Method 201.2.3 (Ref 25). It is used for detg DPhA content of newly manufd proplnts where the solvent extract contains no other brominatable material (such as centralites, phenols, salicylates, etc)

Prepare the sample as described in MIL-STD-286A, Method 509.3, or in Vol 2 of Encycl, p C131-L and use a 5g portion weighed to within 0.2mg. Extract it with ether or methylene chloride using the Method 104.1 of the MIL-STD-286A (Ref 25), which is also described in Vol 2 of Encycl, p C131-L. Check for the completeness of extraction by disassembling the extraction app just before the solvent siphons out of the extractor and transfer ca 10ml into a 50-ml beaker. Evaporate the solvent using a stream of dry air and dissolve the residue (if any) in ca 5 drops of acetone. Place this soln in the indenture of a white spot plate and add 2–3 drops of Na nitrate soln (1% in sulfuric acid). The immediate appearance of a deep blue color indicates the presence of DPhA, and, therefore, incomplete extraction

When the extraction is complete, dissolve the dried residue in the extraction flask in ca 50ml of ether and quantitatively transfer this soln to a 250ml beaker (using ether for rinsing the extraction flask)

Place the beaker on the steam bath (maintained at ca 75° on the surface) and add, drop by drop, enough bromine to assure a slight excess over the amt necessary to convert all the DPhA to tetrabromdiphenylamine (0.6ml of Br for each percent of DPhA in proplnt). A slight excess of Br is indicated by the persistence of a reddish light-brown color

Swirl the contents of the beaker, place on the steam bath and bring the soln to a boil. Remove the beaker with boiling soln from the bath and pass a current of dry air until the odor of Br and of ether disappears, but not longer because prolonged evapn after reaching dryness will yield low results

Add to the residue in the beaker 40ml of 95% alcohol and heat to boiling. Note the time, add 40ml of cold distd w and allow the beaker to remain on the steam bath for exactly 10 mins. Then filter the hot slurry thru the tared (to within 0.2mg) filtering crucible (of sintered glass) and wash the ppt of tetrabromoDPhA, first with ca 80ml of boiling 47.5% alc and then with hot distd w

Dry the crucible with ppt for 1 hour at 105±2°, cool in a desiccator for 2 hrs and weigh to within 0.2mg. Replace it into the desiccator and reweigh after 2 hrs. If the difference in weighings is less than 0.5mg, consider the drying as completed

$$\% \text{ DPhA} = \frac{34.90A}{W}$$

where: A = increase in wt of crucible and
W = wt of sample (ca 5g) corrected for total volatiles

Note: A modification of "gravimetric bromination method", devised by Apatoff and Cohen of Frankford Arsenal is described in Vol 2 of Encycl, p C132 and also in Ref 29, pp5–6

Test 3. Volumetric Bromination Method. The procedure of Pamphlet listed here as Ref 14 is also described in specification MIL-STD-286A, Method 201.1.3. It is used for detg the DPhA content of newly manufd single-base and double-base proplnts contg no other brominatable material (such as salicylates, phenol, or centralites)

Prepare the sample and extract it as listed under previous test (Test 2)

Dissolve the dried residue in the extraction flask in ca 10ml of glac AcOH and, if the nominal content of DPhA in the proplnt is less than 0.5%, transfer the soln to the 250ml iodine titration flask. Rinse the extraction flask with four 10ml portions of glac AcOH, transferring the rinsings to the iodine flask

Note 1: If the nominal DPhA content of the proplnt is betw 0.5 & 1%, transfer the above soln to the 100ml volumetric flask and make to the mark with glac AcOH. Pipet out a 50ml aliquot of this soln to the 250ml iodine flask. Proceed further as described below

Note 2: If the DPhA content is above 1%, pipet out a 25ml aliquot of the 100ml in the volumetric flask to the iodine flask and add 25ml of glac AcOH. Proceed further as described below

To the 50ml soln in the iodine flask, add 25ml of 0.2N standard potassium bromate-bromide soln (prepd as described in Method 605.1 of MIL-STD-286A), accurately measured to within 0.01ml

Moisten the stopper of the iodine flask with a drop of 15% KI soln and add 5ml of 38% HCl. Observe the time and stopper the flask immediately; swirl it for a few seconds and after 1 min of bromination, counting from the time of addg HCl, add 10ml of 15% KI soln and swirl the flask. If an oil separates, add 25ml of carbon tetrachloride

Wash down the gutter and walls of the flask with distd w and titrate the contents immediately with standard 0.1N Na thiosulfate soln (prepd as described in Method 602.1 of MIL-STD-286A) until the soln assumes a light yel color. Add 5ml of starch indicator soln (prepd as described in Method 701.1), and continue the titration carefully until the blue color of the soln disappears

Make a blank determination, using 50ml of glac AcOH, and exactly the same vol of the potassium bromate-bromide soln

$$\%\ \text{DPhA} = \frac{2.115(A-B)N}{W}$$

where: A = Na thiosulfate soln required for the blank, ml

B = Na thiosulfate required to titrate the liberated iodine in the iodine flask, ml

N = Normality of Na thiosulfate

W = Weight of sample (ca 5g), corrected for total volatiles

Many other methods are described in Ref 14 besides the qualitative and quantitative tests mentioned above

Following is the method described on p13 of Ref 14, but not described in Ref 25

Test 4. Precipitation as Diphenylaminedichloride. This method permits a quantitative separation of DPhA from NG, DEGDN, Centralites, DPhUrethane and EtPhUrethane

The apparatus used for this test is shown in the Figure. It consists of items:

A. HCl Gas Generator

- 1-A 60-ml separatory funnel contg concd HCl
- 2-A 125-ml suction filtering flask contg concd sulfuric acid
- 3-Glass tubing, 5mm OD, serving as pressure equalizer

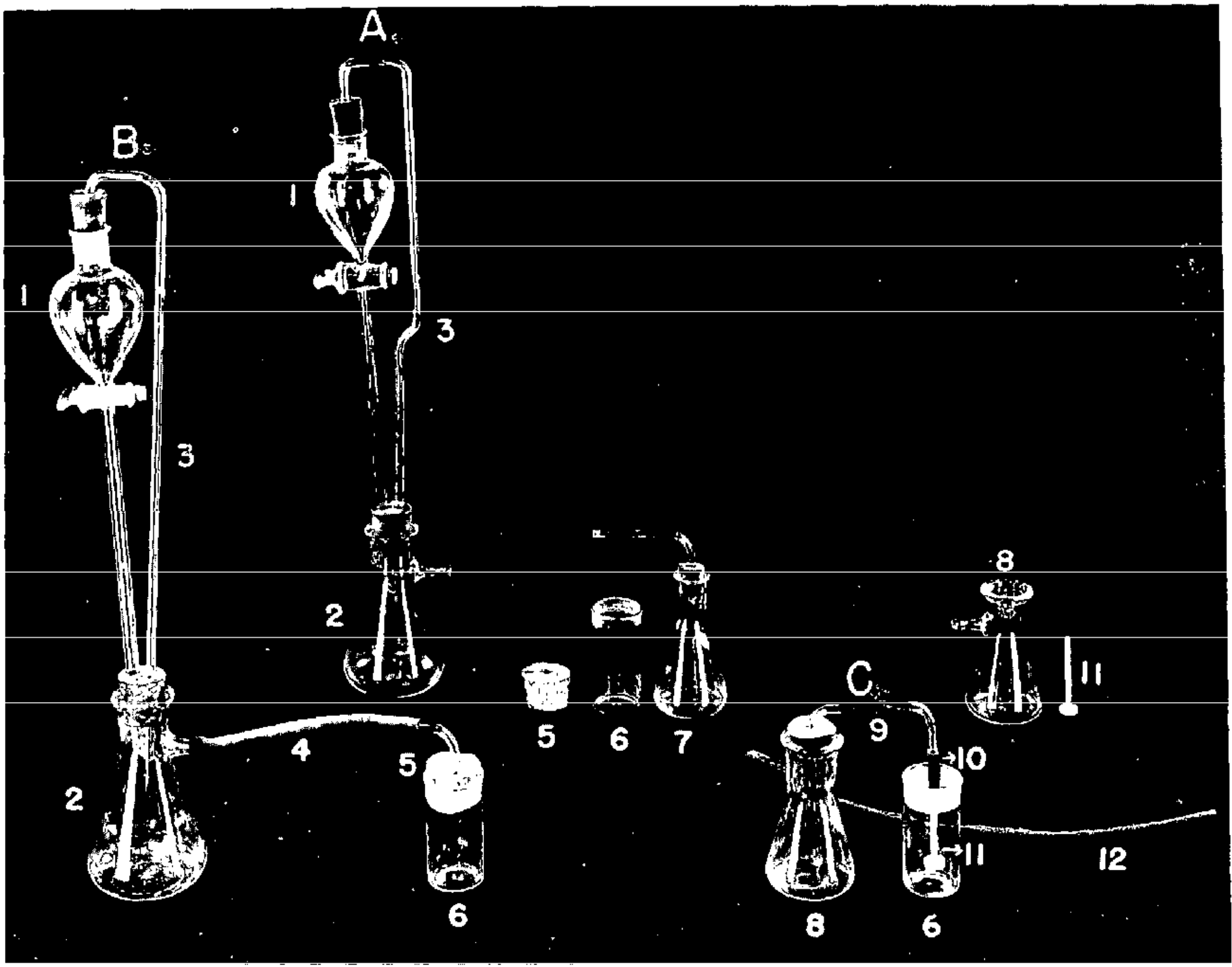
B. Assembly for Precipitation DPhA.2HCl

- 4-Rubber tubing
- 5-One-hole cork stopper, slotted
- 6-Precipitation receptacle, 60mm high, 30mm ID
- 7-A 50-ml Erlenmeyer with bent glass tubing and 1-hole rubber stopper, slotted

C. Assembly for Removing Supernatant Liquid from DPhA.2HCl

- 8-A 50-ml suction filtering flask
- 9-Bent glass tubing, 5mm diameter
- 10-Rubber tubing
- 11-Selas filter stick, 50mm high; diam of disc 11mm
- 12-Suction hose

Weigh accurately the precipitation receptacle alone (6) and also together with filtering stick (11). Remove the stick and transfer into (6) ca 50mg of residue (obtd on extraction of proplnt by the method indicated in Quantitative Test 2). Reweigh (6) and the difference in wt is the wt of the sample. Add 5ml of anhydrous ether, stopper with cork (5) and connect by means of rubber hose (4) to the apparatus shown as B. Allow 4–5 drops of concd HCl to drop from funnel (1) to flask (2) onto the surface of concd sulfuric acid and after HCl gas enters receptacle (6), agitate it by swirling. After 10 mins add 2–3 drops from (1) to (2) and swirl again the (6). After allowing to stand for another 10 mins, disconnect (6) from rubber tubing (4). Pour 10ml of anhyd ether into flask (7), attach it to (4) and saturate the ether with HCl gas by frequent agitation over an interval of 10 mins



APPARATUS FOR PRECIPITATION OF DIPHENYLAMINE DIHYDROCHLORIDE

Arrange the app as shown in C, attach the flask (8) to suction pump and remove the supernatant liquid in (6) by suction. Rinse the contents of (6) with three 3-ml portions of ether satd with HCl from flask (7). Dry the stick and the flask first by suction and then in vacuum desiccator to constant weight. The difference in weights between (6)+(11) (with ppt) and (6)+(11) (without ppt) gives the weight of DPhA dihydrochloride (W_2). If the wt of sample is W_1 (ca 5g), then:

$$\%\ \mathrm{DPhA} = \frac{82.3W_2}{W_1}$$

Note: If the sample contains a large amt of Nitrosodiphenylamine, a small amt of it ppts with DPhA. Nitro-substitution products of DPhA do not interfere with the test

Test 5. Chromatographic Determination. This test is described on pp15–17 of Ref 14

Preparation of Chromatographic Column.

Seal a glass cylinder, ca 2.5cm in diam and 15cm long, to a glass tube of 0.7cm in diam & 2.5cm long. Insert the tube in a perforated rubber stopper of a 125ml suction flask and put a perforated porcelain disc into the cylinder so that it is supported on the shoulders of its restricted end. Cover the disc with a layer of glass wool and pour into the column, while under suction, a previously prepd homogeneous mixture of Merck's CP silicic acid and Johns-Manville's Hyflo Supercel, to a height of 2cm from the top. Pack column uniformly by gently tapping the sides and finish by smoothing and levelling the top

with a spatula (See Fig on p C290-R of Vol 3).
Procedure:

Activate the column by prewashing with ca 40ml of petr ether and remove the bulk of eth by suction before transferring the sample to the column. Prepare the sample by dissolving in 5ml of dry benzene the dry residue obtd on extraction of 5g of proplnt, as indicated in Quantitative Test 2. After the sample has dissolved in benz, add 20ml of petr ether. Pour the soln on the column and apply gentle suction. Wash beaker with two small successive portions of 1:4-benz-petr eth mixture, while keeping the top of the column constantly covered with liquid

After the sample has been transferred to the column, develop the chromatograph by pouring 100ml of benz-petr eth mixt on the column and then wash the benz from the column with a final wash of 30ml of petr eth

Remove enough of petr eth from the column by suction so that the column can easily be extruded from the cylinder. For this place the cylinder horizontally on a large piece of glazed paper and push on the porcelain disk with a metallic rod inserted thru the tube attached to the bottom of cylinder. After extruding the column onto glazed paper, remove small portions of the column 1/4 inch apart along the entire length. Test these portions on a spot plate with 3 drops of concd sulfuric and 1 drop of dil nitric (previously prepd by dissolving 6 drops of concd acid in 100ml of distd w). An unbroken series of blue colors indicates the zone of the column contg DPhA. Remove this zone from the rest of column and spread it on the fritted glass bottom of a Büchner funnel. Leach the column with 75 to 100ml of CP ether, using a gentle suction and collect the filtrate in an accurately tared small beaker. Check the completeness of leaching by placing a few of the last drops of ether leach into indentation of glazed porcelain plate and test for blue color as indicated above

Evaporate the ethereal soln in the beaker at RT, desiccate in vacuo and weigh the beaker. This wt minus wt of beaker gives mgs of DPhA, and if the original sample weighed 10mg:

$$\% \text{ DPhA} = \frac{(\text{mgs of DPhA}) \times 100}{\text{Wt of sample}}$$

Note: If it is suspected that the 10mg sample contains impurities, redissolve the evaporated, leached material in ether and add, dropwise with stirring, 1 ml of 1:4-bromine-carbon tetrachloride mixture. Evaporate the eth, carbon tetrachloride and excess of Br with a stream of air and add 25ml of hot 95% alcohol to dissolve the impurities. Then add 25ml of hot w, allow to stand on a steam bath for 10 mins and collect the ppt of tetrabromodiphenylamine in an accurately tared sintered glass crucible. Dry the ppt for 1 hr at 100°, cool in a desiccator and weigh

$$\% \text{ DPhA} = \frac{\text{Wt of TeBrDPhA} \times 34.9}{\text{Wt of sample}}$$

Test 6. Increase in Weight Method. This Method 201.3.3 of MIL-STD-286A (Ref 25) is used for detg the DPhA content of newly manufd proplnts, where the solvent extract contains no other brominatable material. It was not described in Ref 14

Prepare and extract a 5g sample, weighed within 0.2mg as indicated in MIL-STD-286A, Methods 509.3 & 104.1 or in Vol 2 of Ency, p C131-L. When the extraction is complete, evaporate the solvent from the residue and weigh the tared flask together with dry residue, within 0.2mg. Dissolve the residue in ca 50ml of ether and concurrently prep a blank consisting of similar flask with 50ml of ether alone

Place both flasks on the steam bath, maintained at ca 75° on the surface, and located in the hood. Add dropwise in the 1st flask enough bromine (0.6ml for each 1% of DPhA in proplnt) to assure a slight excess over the amount necessary to convert all the DPhA to tetrabromoDPhA. The same amt of bromine must be added to the blank

Swirl the flasks and replace on the steam bath. When the solns start to boil, remove the flasks and evaporate the ether and the excess of bromine under a current of dry air. Place the flasks for 2 hrs in the vacuum desiccator maintained at a pressure (absolute) of ca 255mm Hg and weigh within 0.2mg

$$\% \text{ DPhA} = \frac{53.61(A-B)}{W}$$

where: A = Increase in wt of flask 1, as a result of bromination of the DPhA in the sample and of some material present in ether

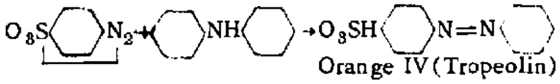
B = Increase in wt of blank flask as a result of bromination of materials in the ether

W = Weight of sample (ca 5g) corrected for total volatiles

Test 7. Available Stabilizer (Diphenylamine or Ethyl Centralite). This is Method 217.2.1 of MIL-STD-286A (Ref 25). It was investigated by Apatoff & Cohen of Frankford Arsenal and found to be "inadequate". They proposed to replace it with a new method which they described in Ref 29. We have described their method in Vol 2 of Encycl, pp C132 & C133

Test 8. Determination of DPhA in Air.

Ponomarenko (Ref 18) described the following colorimetric test for detn of small quantities of DPhA in air. The test is based on the formation of reddish-brown coloration produced by the action of diazosulfanilic acid on DPhA in dilute sulfuric acid:



Orange IV (Tropeolin)

Some primary amines (aniline, toluidine) do not interfere, others (α-naphthylamine) do

The reagent (a soln of diazosulfanilic acid in aq sulfuric acid) was prepd in the following manner:

a) In the 1st operation, the commercial sulfanilic acid was purified by suspending 1 g of acid in 6ml of w and heating the mixt to boiling. Then a 20% aq soln of soda was added dropwise until complete dissolution of sulfanilic acid. After removing the impurities by filtration, the liq was acidified with 20 drops of concd sulfuric acid and the resulting purified sulfanilic acid was pptd in the form of small white grains

b) After cooling the above mixture, the acid was diazotized by adding 3ml of 20% aq soln of Na nitrite and allowing to stand for 1 hr with periodic agitation

c) The small-grain ppt of diazosulfanilic acid was filtered off, washed with 2 portions of w and removed (by means of a stream of w) to a beaker

d) Concd sulfuric acid was added dropwise until complete dissolution of ppt. The resulting colorless liq was the desired reagent

Preparation of Standard. Forty mg of DPhA was dissolved in aq alc and transferred into a 100ml volumetric flask. After filling the flask to the mark and mixing, measured quantities of this "standard" were placed in several 50ml flasks. After adding to each flask 5ml of w, 2ml of concd sulfuric acid and 10 drops of the above reagent, followed by mixing, the flasks were allowed to stand for 2 hrs. Then w was added to the 50ml mark and, after mixing, the colorimetric readings taken. Quantities as low as 0.02mg of DPhA in 50ml may be detected by this method

Note: Low readings might result if the time of standing at ord temp was less than 2 hrs. If, however, the soln in 50ml flask was heated to 50° for 30 mins, the total time of standing could be cut to 1 hr

Test Procedure. For detn of DPhA in atmosphere, the measured vol of air was bubbled thru absorbers contg sulfuric acid together with porous glass plates syst Schott. When 30 liters of air were sucked during 2 hrs, it was recommended to use the 1st absorber contg 90% sulfuric and two other absorbers with 50% sulfuric acid. Petry absorbers were not recommended

If the tests for DPhA in contents of 2nd and 3rd absorbers gave negative results, the contents of the 1st absorber was transferred to a volumetric flask of larger size than the absorber, plus the volume of w required to dilute the acid in absorber to approx the same concn as acid used in the above standards. Then the contents in the vol flask was brought to the mark and colorimetric reading taken in comparison with standards

Note: It was claimed that the best results are obtained for solns contg 0.04 to 0.09mg DPhA per 50ml of soln

Most of the refs listed here are found in open literature, but Refs 13, 14, 21, 22, 24, 26, 27 & 28 come from Picatinny Arsenal; Ref 25 is Department of Defense publication and Ref 29 is Frankford Arsenal publication. Ref 30 is US Military Specification for technical DPhA

US Armed Forces requirements for technical grade DPhA intended for use as a stabilizer in the production of smokeless propellants are covered by the Specification MIL-D-98A (Ref 30)

Workmanship. The standard of workmanship shall be such as to insure the production of material meeting the requirements of this specification. The DPhA shall be free of extraneous material (such as iron rust, wood particles, dirt, colored salts and other visible impurities). The color of DPhA shall be no darker than a light brown

Following are the tests deecribed in this specification:

1) Setting Point – 51.7 to 53.0°
2) Insoluble Material (Residue) – max 0.02%
3) Moisture – max 0.2%
4) Acidity (as HCl) – max 0.005%
5) Alkalinity (as NaOH) – max 0.005%
6) Oxidizable Material (as Aniline) – max 0.1%

They are determined in the following manner:

1) *Setting Point.* Arrange the apparatus as shown in Figure on p A613-L of Vol 1 of Encycl. The thermometer shall be number 92C, "American Society for Testing and Materials", with a range of 40 to 70°C and partial immersion 76mm. Use the same procedure as described for Na azide on pp A612-R & A613-L as "Solidification Point"

2) *Insoluble Matter.* Shake 25.0g sample with 100ml of benzene (of sp gr 0.878 at 20/20°) or with 100ml ethyl ether (of sp gr 0.723 at 20/20°) until complete dissolution of DPhA. Filter off the insol impurities thru an accurately tared sintered glass filtering crucible and rinse the residue with benz or eth. Dry the crucible for 1 hr at 100°, cool in a desiccator and weigh. Inspect visually the impurities

3) *Moisture.* Weigh a 5g sample in a tared weighing dish provided with a stopper. Remove the stopper and heat the dish for 4 hrs at 40°. Replace the stopper, cool the dish and reweigh it

4) *Acidity* and 5) *Alkalinity.* Place 20g of an "as received" sample in a 250ml Erlenmeyer flask, provided with a glass stopper, and add 50ml of nearly boiling distd w. Stopper (loosely) immediately and shake vigorously for 10 mins. Cool to 25° and filter the supernatant liquid, retaining the DPhA in the flask. Repeat the extraction with 50ml of boiling distd w exactly as above. To the combined filtrates add a few drops of phenolphthalein indicator and observe the color. If the soln remains colorless, titrate it with 0.1N alkali and if it turns red, titrate with 0.1N acid. Make a blank titration on the distd w used and correct the titration readings for any acidity or alkalinity found in w. Calculate the acidity to HCl and alkalinity to NaOH. Save this soln for the next test

6) *Oxidizable Material* (Aniline and Its Salts). Pipet 25ml of 0.10N bromide-bromate soln into the Erlen flask contg the soln of previous test. Cool to 15°, add 5ml of concd HCl and follow, after 1 min, with the addn of 10ml of 10% KI soln. Titrate the contents with 0.10N Na thiosulfate soln using starch as indicator. Make blank determination on 25ml of 0.10N bromide-bromate soln exactly as above and calc the aniline from the formula listed below:

$$\%\ \text{Aniline} = \frac{[(VN-vN)-0.033]\,1.551}{W}$$

where: V = ml Na thiosulfate used in blank
v = ml Na thiosulfate used in titration of sample
N = Normality of Na thiosulfate
0.033 = Correction for DPhA dissolved in w
W = Weight of sample (20g)

Refs for DPhA Analytical Procedures

1) O. Turek, Chemicky'Obzor **1**, 295(1927) & CA **22**, 4819(1928) (Volumetric detn of DPhA in smokeless proplnts) 2) H. Levenson, JIEC (Anal Ed) **2**, 246(1930) (Volumetric bromination determination of DPhA in NG proplnts in presence of Centr) 3) O.C. Ellington & H.B. Beard, JSCI **50**, 1517(1931) (Royal Arsenal Method of volumetric bromination for detn of DPhA in proplnts) 4) S.G. Cook, IEC (Anal Ed) **7**, 250–55(1935) (Detn of DPhA in smokeless proplnts) 5) A. Bresser, SS **30**, 42–3(1935) (Detn of DPhA in proplnts) 5a) G.C. Hale, Army Ordn **5**, 674–75(1935) 6) F. Deschaux, RevGénMatPlastiques **12**, 351(1936) & CA **31**, 2007(1937) (Detn of DPhA in Guncotton) 7) F. Becker & G. Hunold, SS **33**, 213 & 244(1938); CA **32**, 9501(1938) (Detn of DPhA in proplnts) 8) R. Dalbert, MP **28**, 119–26(1938) & CA **33**, 7569–70(1939) (Analytical procedure for detection of DPhA and its nitroso- and nitro- derivs in "poudres B" stored at various temps. The method includes extraction with methylene chloride and conversion of nitroso- and nitro-derivs into amine using stannous chloride and HCl) 9) Clift & Fedoroff **1**(1942), Chap XII, pp 4–5 (Detn of setting point, ether insol, moisture, acidity or alkalinity and aniline content) 10 Ibid **2**(1943), p D11 [Description of the following procedure which was recommended by Buisson in "Le Problème des Poudres", Paris (1913), p126 for identification of different products of decompn of proplnts: "Digest for 24hrs 10g of finely divided proplnt with 100ml alc and filter the extract. To test for nitrosoDPhA, boil 5ml of alc extract with 1 ml of 5% soln of α-naphthylamine. Compare the intensity of red coloration with that of std

soln. To test for DinitroDPhA's add to 5ml of alc extract a few drops of alc KOH. The red color produced indicates the presence of DNDPhA (and possibly of TrinitroDPhA). To test for TNDPhA add to 5ml of alc extract few drops of satd alc soln of KCN. A red color indicates TNDPhA] 11) Davis (1943), 312 (Qualitative tests for DPhA and its products of decompn) (See description in the text)
12) Kast-Metz (1944), pp287–88 (Detn of DPhA in proplnts contg camphor); p289 (Detn of DPhA in proplnts by method of Berger); p291 (Detn of DPhA in NG proplnts in presence of Centr); p294 (Detn of DPhA as chlorhydrate); p294 (Colorimetric detn of DPhA by sulfuric and nitric acids); pp294–95 (Chemisch-technischen Reichsanstalt method for detn of Centralite & DPhA in proplnts)
13) S. Weisberger, PicArsnChemLabRept **No 101323** (1944) (Color test for DPhA by means of 10% soln of selenious acid in HCl) (See Qualitative Test 3 in the text)
14) Anon, "Diphenylamine", Pamphlet of 44 pages compiled and typed at Picatinny Arsenal ca 1944 (Various qualitative and quantitative tests for DPhA) (See in the text)
15) Pérez Ara (1945), 438 (Two qualitative tests for DPhA (See in the text)
16) Fedoroff & Clift **4**(1946), pp 27–8 (More detailed description of the tests listed in Ref 9) (More recent description is given in Ref 30) 17) F. Ellington, Analyst, **71**, 305–08(1946) & CA **40**, 5566(1946) (Improved volumetric bromination method for the detn of DPhA in proplnts) 18) B.V. Ponomarenko, ZavodLab **13**, 937–41(1947) (See Quantitative Method 8 in the text) 19) J.G. Norvall et al, Analyst **73**, 389–93(1948) & CA **42**, 7984(1948) (Colorimetric detn of DPhA in proplnts by means of K dichromate reagent) 20) G. Sifre, MP **35**, 374–79(1953) (Polarographic detn of DPhA in proplnts in presence or absence of Centralites) 21) A.H. Castelli et al, PATR **2094**(1954), "Diphenylamine, Dibutylphthalate and Dinitrotoluene Determination in M1 and M6 Propellants by IR Method" 22) W.D. Tullis & M. Roth, PATR **2432**(1957), "Streamlined Methods for Determining the Available Diphenylamine Content of Propellants" 23) L. Marvillet & J. Tranchant, MP **42**, 261–69(1960) (7 refs) (Detn of DPhA and nitrated derivs in non-aqueous medium)
23a) Anon, "Analytical Methods for Powders and Explosives", ABBofors, Nobelkrut, Bofors, Sweden (1960), p85 (Diphenylamine) DPhA gives blue color with sulfuric and nitric acids. The same color is obtd with ceric sulfate, and this reaction is used for qualitative detection of DPhA in the presence of Centralite and methyl- or ethyl-phenylurethanes. Specification tests used at Bofors plant in 1960 were: 1-Appearance and mechanical impurities 2-Solidification point (53°), 3-Matter insol in ether-alcohol, 4-Ash, 5-Reaction with sulfuric acid, 6-Primary amines, 7-Moisture, 8-Reaction (Alkalinity), 9-Granulation. Following solubility values for DPA are given in 100g of solvent: water 0.03g at 25°; methanol 57.5g at 19.5°; ethanol 56.0g at 19.5°; acetone 298.6g at 28°; chloroform 206.3g at 28° and toluene 227.7g at 28° 24) Encycl of Expls **1**(1960), p A10 (Detn of DPhA in propellants contg Acardites I, II and III) 25) US Military Standard **MIL-STD-286A** (1961), Methods 201.2.3; 201.3.3; 201.1.3 and 217.2.1 (See in the text)
26) Encycl of Expls **2**(1962), pp C45–C46-L (Detn of DPhA in proplnts contg Carbazole)
27) Ibid **2**(1962), p C131 (Prepn of proplnt sample for testing and extraction with anhydrous methylene chloride) 28) Ibid **2** (1962), pC132-L (Detn of available DPhA in proplnts contg Centralite 1, using Method 217.2.1 of Ref 25) 29) J.B. Apatoff & Joseph Cohen, Test Report **T62-15-1**, June 1962, Frankford Arsenal, Philadelphia, Pa, "Determination of Admixture of Diphenylamine and Ethyl Centralite in Propellants". This method is described in Vol 2 of Encycl, pp C132 & C133 30) US Military Specification **MIL-D-98A**, 14 May 1962, "Diphenylamine, Technical" 31) StdMethodsChemAnalysis **2B** (1963), p 1385 (DPhA by titrimetric bromination method) 32) L. Marvillet & J. Tranchant, MP **45**, 131–44(1963) (Detn of DPhA and Centralite in presence of both of them) 33) S.K. Yasuda, JChromatog **14**(1), 65–70(1964) & CA **60**, 14325(1964) (Identification of N-nitroso- and nitro- diphenylamine by two-dimensional thin-layer chromatography on silica gel contg Zn dust) 34) J. Curé, MP **46–47**, 59–76(1964–1965) (Detn of DPhA and Centralite in "poudre B" by chromatographic method) 35) E.F. Reese, formerly of PicArsn, private communication (1965)

DIPHENYLAMINE DERIVATIVES

Diphenylamine-4-azide; 4-Azidodiphenylamine or **N-Phenyl-4-azidoaniline,** $N_3.C_6H_4.NH.C_6H_5$; mw 210.24, N 21.65%; yel crysts (from alc) and reddish-yel (from petr eth); mp 71° (decomp above mp); bp – explodes on rapid heating; very sol in alc; sol in petr eth; explodes when treated with concd nitric acid; can be prepd by interaction of hydroxylamine-hydrochloride, $NH_2OH.HCl$, with 4-nitrosodiphenylamine, $ON.C_6H_4.NH.C_6H_5$, in Na carbonate soln (Refs 1 & 2)

Forms various explosive salts and nitroso compd:

4-Azidodiphenylnitrosamine or **N-Nitroso-4-azido-diphenylamine,** $N_3.C_6H_4.N(NO).C_6H_5$, mw 239.24, N 29.28%; yel leaflets (from alc); mp 55°(decomp slightly above mp); bp – explodes on heating on a Pt foil; can be prepd by treating the acetic acid soln of 4-azidoDPhA with Na nitrite at 0°(Refs 1 & 2)

Refs: 1) Beil 12, [429] 2) A. Angeli & A. Pieroni, AttiAccadLinceiMem [5], 32 I, 152 & 453–54(1923); JCS 124 I, 612 & 1026(1923); CA 18, 1119–20(1924)

Diphenylamine, Diazo Compounds and Their Nitrated Derivatives. See under Diazodiphenylamine and Derivatives in this Vol and also: F. Steppes, GerP 291156(1915) & CA 11, 899 (1917) (Several expl compns contg nitrated diazocompounds of DPhA were proposed)

Diphenylaminediazonium Hydroxide and Derivatives. See Anilinobenzenediazonium Hydroxide and Derivatives in Vol 1 of Encycl, p A421

Diphenylaminonitroso Derivatives:

Diphenylnitrosamine or *N-Nitroso-diphenylamine,* $C_6H_5.N(NO).C_6H_5$; mw 198.22, N 14.13% yel or orn crysts; sp gr 1.23; mp 66–67°; insol in w; sol in benz, alc, acet and ethylene dichloride; can be prepd by treating DPA with N dioxide in abs ether and cooling the reaction mixt (Refs 1 & 2). Used as retarder of vulcanization of rubber and in pesticides

Refs: 1) Beil 12, 580, (294)& [310] 2) T.L. Davis & A.A. Ashdown, JACS 46, 1052(1924) (Prepn) 3) CondChemDict (1961), 807-R

p-Nitroso-diphenylamine, $ON.C_6H_4.NH.C_6H_5$; mw 198.22, N 14.13%; grn pltlts (from benz); mp 144.6–145.9°; sl sol in w & ligroin; very sol in alc, eth & chlf; sol in benz; can be prepd by transposition of Diphenylnitrosamine following the method of Goldschmidt and Wurzschmidt (Refs 1 & 2), using hydrogen chloride in alc + ether

p-NitrosoDPhA forms during aging of NC proplnts, stabilized with DPhA. In fact, it is the first transformation product of DPhA and it is a stabilizer by itself. Its presence in small quantity in a proplnt does not indicate instability. Nitroso does not give any color with Amm persulfate and no color with a cold 1% alc soln of α-naphthylamine, but an orange color is produced if soln is heated. One-tenth of a mg of nitroso imparts an intense blue coloration to a few mls of concd sulfuric acid (Ref 3 and Ref 4, p312). Médard & Thomas (Ref 5) detd its heat of formation at C_v as 49.2kcal/mol and heat of combustion as 1520.8kcal/mol

Refs: 1) Beil 12, 207 & [122] 2) S. Goldschmidt & B. Wurzschmidt, Ber 55, 3220(1922) 3) T.L. Davis & A.A. Ashdown, JACS 46, 1052(1924) 4) Davis (1943), 312 5) L. Médard & M. Thomas, MP 38, 50(1956) & CA 50, 3763(1956)

Mononitrodiphenylamines,

$O_2N.C_6H_4.NH.C_6H_5$, mw 214.22, N 13.08%. The following isomers exist:

2-(or ortho)-Nitrodiphenylamine, orn lfts, mp 75–75.5°(from alc + w). It can be prepd by refluxing 2-nitroacetanilide with bromobenzene in the presence of anhyd K carbonate & a trace of cuprous iodide (Ref 1) and by other methods

Desseigne & Rabussier (Ref 3) prepd it by heating 2-nitrochlorobenzene, aniline & Na carbonate for 9 hrs at 193–200°, cooling, and pouring the mixt into w at 40–50°

Marvillet & Tranchant (Ref 4) detd its presence in expls by extracting with methylene chloride, dissolving the residue in glac acet ac, brominating in the presence of CCl_4 and titrating the excess Br with thiosulfate

Tavernier & Lamouroux (Ref 5) detd its Q_c^v = 1488.33kcal/mol and Q_f^v = minus 16.71 kcal/mol

2-Nitrodiphenylamine is one of the products isolated from aged NC proplnts by Becker & Hunold (Ref 2)

It has been used by the US Armed Forces as a stabilizer in cast and solventless double-base proplnt. Methods of testing are described in MIL-STD-286A (Ref 6) and specification requirements in MIL-N-3399A (Ref 7)

Method 218.12. Volumetric Bromination. This method is used for detg the 2-NDPhA content of proplnts contg nitrate esters or nitrocompds. Other brominatable compds must be absent. Prepare the proplnt and extract a 5g portion as described in Vol 2 of Encycl, p C131. Dissolve the dried residue in the extraction flask contg 10ml glac AcOH and transfer the soln quantitatively to a 250ml iodine titration flask. Rinse the extraction flask with several 10ml portions of AcOH, transferring the rinsings to the iodine flask until total volume will be 50ml

Add 25ml of CCl_4 and an accurately measured 10ml portion of 0.5N soln of K bromate-bromide. Moisten the stopper of the flask with a drop of 10% KI soln and add 5ml of 38% HCl, noting the time. Stopper the flask immediately, swirl for a few seconds and at the end of 1 min, after introducing HCl, add 25ml of 10% KI soln. Again swirl the flask, wash down the gutter and the walls with distd w and titrate the resulting soln *immediately* with 0.10N Na thiosulfate soln until near the end point, as indicated by the disappearance of a strong iodine color. Add 5ml of starch indicator soln and titrate carefully until the disappearance of blue color

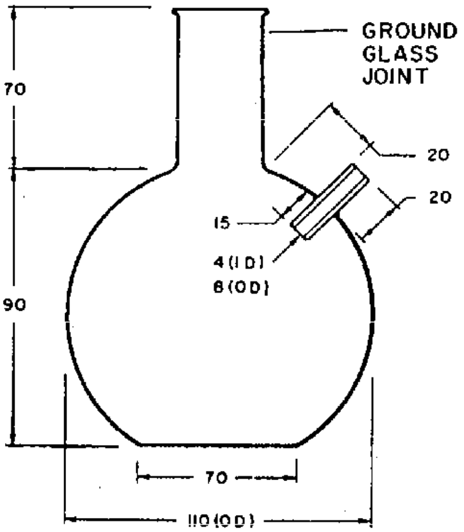
Make a blank detn, using exactly the same volumes of bromate-bromide soln, glac AcOH and carbon tetrachloride

$$\% \text{ of 2-NDPhA} = \frac{3.570(A-B)N}{W}$$

where: A = Na thiosulfate required for blank, ml
B = Na thiosulfate required to titrate the liberated iodine in the soln contg the sample
N = Normality of Na thiosulfate soln (0.10N)
W = Weight of the sample corrected for total volatiles

Method 218.2.1. Titanous Chloride-Buffer Method. This method is used for detg 2-NDPhA content in freshly prepd single-base proplnts contg no other nitrocompds

Prepare and extract a 5g sample of proplnt as indicated in Vol 2 of Encycl, p C131, but use for extraction the titration flask, shown in Fig, after plugging its inlet tube by the rubber stopper. After removing the solvent from extract by a stream of dry air, dissolve the residue in 10ml of ethanol and add 10ml of 0.5N alcoholic KOH soln. Heat the flask on a steam bath for 2 hrs, remove alcohol by a slow stream of dry air, and dissolve the residue in 40ml of glac AcOH



PYREX GLASS FLASK
BULB CAPACITY – 500 ml.
ALL DIMENSIONS IN MILLIMETERS

Figure *Titration flask*

Add 20ml of 20% Na acetate buffer soln and observe the appearance of resulting mixture. If the phthalate ester has not been completely saponified by the KOH, it will appear as an oily, immiscible liquid. Discard such liquid and use a new sample

If complete saponification is achieved, remove the rubber stopper from the inlet tube of the flask and attach a source of inert gas (nitrogen or carbon dioxide) to the tube. After allowing the gas to flow for 5 mins, add (while continuing the flow of gas until the end of test) in 2-ml increments 40ml of the 0.2N titanous chloride soln, swirling the flask after each addn. Then swirl the flask for another 15 secs and add 25ml of 15% HCl soln, followed by 5ml of 20% ammonium thiocyanate soln

Titrate with the std 0.15N ferric ammonium sulfate soln to the first permanent red coloration

Conduct a blank detn using the same reagents and in the same quantities as above, but no sample

$$\% \text{ of 2-NDPhA} = \frac{3.570(A-B)N}{W}$$

where: A = Ferric ammonium sulfate used for blank, ml
B = Ferric ammonium sulfate used for sample, ml
N = Normality of ferric ammonium sulfate soln
W = Weight of sample, corrected for total volatiles

Method 218.3.1. Polarographic Determination. This method is used for detg the 2-NDPhA content of newly manufd proplnts contg no nitrate esters other than cellulose nitrate. It is not described here because instrumentation required for this test is too expensive and needs special training not acquired by an ordinary chemist

Method 218.4.1. Spectrophotometric Determination. This method is used for detg the 2-NDPhA content in newly manufd proplnts.

Prepare a standard curve as follows: Dissolve 0.5000g of pure 2-NDPhA in one liter of ethanol and, using a buret, transfer a series of increments, 1.0, 1.5, 2.0, 2.5 and 3.0ml to 100ml volumetric flasks. Make up to volume with ethanol in each flask

Fill absorption cells of spectrophotometer or colorimeter with above solns, measure the absorption of each of the solns at 430 millimicrons and compare with absorption of ethanol placed in a similar cell

Plot the results on graph paper with the absorbency as the ordinate and the weight of 2-NDPhA as the abscissa and connect the points by a line as close to a straight line as possible

Prepare the specimen as described in Vol 2 of Encycl, p C131-L and place 0.5000g in a 100ml round-bottom flask. Add 50ml of ethanol, attach a reflux condenser to the flask and boil the soln gently on a hot plate for 30 mins. Remove the flask from the hot plate, cool to RT, and disconnect the condenser

Pour the soln thru Whatman No 1 filter paper, into a 100ml volumetric flask and rinse the round-bottom flask and condenser with several portions of ethanol, pouring the washings thru the above filter into the volumetric flask. Dilute the soln with ethanol to 100ml mark

Fill a cell with this soln and a similar cell with ethanol. Set the instrument at 430 millimicrons and det the absorbency of cell contg the proplnt soln in relation to reference and calc the percentage of 2-NDPhA in the proplnt

US Military requirements and tests for 2-Nitrodiphenylamine intended for use in the manuf of proplnts are described in the Specification MIL-N-3399A (1958). They are as follows:

1) *Color and Form* – shall be an orange flake (Class 1) or powder (Class 2) when visually examined

2) *Foreign Material* – shall be free from grit and extraneous foreign material such as sticks, straw, sand, etc, when visually examined

3) *Solidification Point* – shall not be less than 73.0° when detd in the same type of apparatus as shown in this Vol under Diphenylphthalate. Melt a 50g sample in a covered 250ml beaker by immersing its lower half in a water bath at 90 to 95° for 15 mins, and transfer the liquid to the tube C (See Fig on p D**1473**) filling it to within ca 1.5 inches of the top. Close the tube with stopper (a), provided with standard thermometer T, correction thermometer T_1 and stirring rod R. Immerse the stoppered tube in a water bath at 85–90° for 5 mins, wipe it off and place inside tube B, which is, in turn, placed inside 1 liter jar A. Proceed now exactly as described under Diphenylphthalate

4) *Purity* – not less than 97.5% when using one of the following two methods

4.1) *Titanous Chloride Buffer Method.* Transfer quantitatively a 0.20±0.01g sample to a dry 500ml titration flask, add 40ml of glac AcOH and connect with a CO_2 generator, similar to H_2 generator shown on p A416-L of Vol 2. After displacing air in the flask with CO_2 add slowly, with constant swirling, 25ml of of soln contg 1 g of $CH_3COONa.3H_2O$ per ml, followed by exactly 50.00ml of ca 0.2N Titanous chloride soln. Mix horoughly by swirling for at least 15 secs, add 5ml of 20% NH_4SCN soln and titrate with ca 0.15N ferric alum soln until an amber color, which persists at least 30 secs, is produced. Determine concurrently a blank prepd by using the same ingredients as above, except the sample

$$\text{Purity(\% 2-NDPhA)} = \frac{3.570(V-v)N}{W}$$

where: V = ml of ferric alum used for blank
v = ml of ferric alum used for the sample
N = normality of ferric alum
W = weight of sample

4.2) *Bromination Method.* Transfer an accurately weighed 0.9–1.0g sample to a 250ml iodine flask provided with a glass stopper and dissolve it in 25ml of chloroform. Add 60ml of glac AcOH, followed by exactly 10.0ml of

0.5N potassium bromide-bromate soln and 5ml of concd HCl. Close the flask immediately with the stopper previously moistened with a drop of 10% KI soln, mix the contents by swirling and, after allowing to stand for 10±0.5 mins, add 25ml of 10% KI soln. Titrate the liberated iodine with 0.1N Na thiosulfate soln until near discoloration; add 5ml of starch soln and continue titration until the blue color is completely discharged. Run concurrently a blank titration using the same reagents as above

$$\text{Purity(\% 2-NDPhA)} = \frac{3.570(V-v)N}{W}$$

where: V = ml of Na thiosulfate used for blank
v = ml of Na thiosulfate used for the sample
N = normality of Na thiosulfate
W = weight of sample

5) *Volatile Matter* — 0.06% (max)

Transfer a 10g sample to an evaporating dish of ca 60mm diam and accurately weigh. Heat in an oven at 100 to 105° for 3 hrs, cool, weigh and calculate the loss of wt as % volatile matter

6) *Material Insoluble in 95% Ethanol* — 0.2% (max)

Stir thoroughly in a 250ml beaker a 10g sample with 200ml of alcohol until complete dissolution of everything soluble. Filter thru a tared sintered glass crucible and wash the beaker by means of a stream of alc into the crucible. Rinse, with five 10ml portions of alc, the crucible with contents and dry it at 100–105° for 30 mins. Cool in a desiccator, weigh and calc the gain in wt as % insol material. In case of an abnormally high result, run a blank on the alcohol used

7) *Ash* — 0.1% (max)

Transfer a 5.0g sample to a tared porcelain or silica crucible (previously heated and cooled in a desiccator) and add sufficient alc to cover the sample. Place the crucible on a hot plate at low heat and ignite the alc. When the alc burns out and the sample is completely carbonized, heat the crucible over a Bunsen burner until all the carbonaceous matter of sample disappears. Then cool in a desiccator and weigh. Calc the gain in wt as % ash. In case of an abnormally high result, run a blank on the alc used

8) *Acidity* — 0.05mg KOH per 1 g (max)

Transfer a 10g sample to a 125ml Erlen flask and add 50ml of boiling distd water (having a pH of 6.0±0.5 at 25°) to melt the sample. Close the flask with rubber stopper and shake until the sample solidifies. Filter the mixture rapidly thru a dry filter paper (conforming to type II, class 5 of Spec UU-P-236) into a flask, close it and cool to 25°. Determine the pH by means of an app using a glass-calomel electrode pair. The pH value shall be not more than 7.0 and not less than 5.0. Wash the residue in the flask with two 25ml portions of distd w and filter thru the same paper as previously used onto the flask contg the filtrate. Add 5 drops of 1% phpht indicator and titrate to a light pink end point with 0.1N Na (or K) hydroxide soln

$$\text{Acidity (mg KOH per g)} = \frac{56.1VN}{W}$$

where: V = ml of Na (or K) hydroxide soln used in the titration
N = normality of Na (or K) hydroxide soln
W = weight of sample used

Refs for 2-Nitro DPhA: 1) Beil **12**, 690, (341) & [369] 2) F. Becker & G.A. Hunold, SS **33**, 213–15 & 244–47 (1938) & CA **32**, 9501 (1938) 3) G. Desseigne & B. Rabussier, MP **38**, 225–39 (1956) & CA **55**, 21005 (1962) 4) L. Marvillet & J. Tranchant, MP **39**, 329–33 (1957) & CA **52**, 21107 (1958) 5) P. Tavernier & M. Lamouroux, MP **39**, 337–38 & 354 (1957) 6) MIL-STD-286A (1961), Methods 218.1.2, 218.2.1, 218.3.1, 218.4.1 7) US Specification MIL-N-3399A (1958)

Other mononitro derivatives are not of such importance. They are:

3-(or meta)-Nitrodiphenylamine, red lfts, mp 112–14°
Ref: Beil **12**, 702, (346) & [377]

4-(or para)-Nitrodiphenylamine, pale-yel ndls, mp 132–33°; was isolated by Becker & Hunold (Ref 2) as one of the products formed during aging of NC proplnts stabilized with DPhA. It was proposed as a stabilizer in smokeless proplnts
Refs: 1) Beil **12**, 715, (351) & [386] 2) F. Becker & G.A. Hunold, SS **33**, 213–15 & 244–47 (1938) & CA **32**, 3501 (1938) 3) All & En Expls (1946), p41

Nitrodiphenylnitrosamines,
$O_2N.C_6H_4.N(NO).C_6H_5$; mw 243.22, N 17.28%. Two isomers were isolated by Becker & Hunold (Ref 5), three are known:
2-Nitrodiphenylnitrosamine, tablets (from

MeOH), or pltlts (from alc), mp 99–101.1°; sol in glac acet ac, acet, toluene, 95% alc and in 99% MeOH (Ref 1). Methods of prepn & other props are found in Refs

3-Nitrodiphenylnitrosamine, ndls, mp 89–90°; readily sol in chlf & acet; sl sol in alc & glac acet ac; soln in concd sulfuric acid is violet in color (Ref 2)

4-Nitro-diphenylnitrosamine, crysts (from chlf) or prisms (ndls) (from glac acet ac or xylene), mp 130–33°; sol in common solvs (Ref 3). Other props & methods of prepn are found in the Refs

Refs: 1) Beil **12**, 679 & [374] 2) Beil **12**, [383] 3) Beil **12**, 728, (355) & [396] 4) T.L. Davis & A.A. Ashdown, JACS **46**, 1053(1924) 5) F. Becker & G.A. Hunold, SS **33**, 244–47(1938) 6) W.A. Schroeder et al, AnalChem **23**, 1740(1951) & CA **46**, 5434 (1952) (UV & visible absorption spectra in alc of derivs of DPA & others)

Dinitrosodiphenylamines, $C_{12}H_9N_3O_2$, mw 227.22, N 18.49%. The following isomer is described in the literature:

N,4-Dinitrosodiphenylamine,
$ON.C_6H_4.N(NO).C_6H_5$; yel-grn pltlts (from eth), mp ca 98°(dec); sol in alc, eth & benz; insol in w; was prepd by treating a soln of 4-nitrosodiphenylamine in alc & concd HCl with aq $NaNO_2$ soln (Refs 1 & 2)

Refs: 1) Beil **12**, 686 2) P.W. Neber & H. Rauscher, Ann **550**, 182(1942) & CA **37**, 3411(1943) 3) American Cyanamid Co, BritP 824627(1959) & CA **54**, 9345(1960) (Improves dispersion of C black in rubber vulcanizates by incorporating 0.05–2.0% of the dinitroso compd) 4) C.C. Tung, USP 3264254(1966) & CA **65**, 13927(1966) (Vulcanizing butyl rubber with Dinitrosodiphenylamines)

4,4'-Dinitrosodiphenylamine,
$ON.C_6H_4.NH.C_6H_4.NO$ was patented by R.A. Newson, USP 3267132(1966) and listed, without description in CA **65**, 20251(1966). Not found in Beil

NOTE: No other Dinitrosodiphenylamines were found in the literature thru 1966

Dinitrodiphenylamines, $C_{12}H_9N_3O_4$, mw 259.22, N 16.21%. Following isomers are listed in the literature:

2,2'-Dinitrodiphenylamine,
$O_2N.C_6H_4.NH.C_6H_4.NO_2$; golden-yel lfts, mp 166–69°. It can be prep from 2-nitroaniline and 2-bromo-1-nitrobenzene on heating in Na carbonate soln with Cu_2Cl_2 catalyst at 180°

Refs: 1) Beil **12**, 690 & (341) 2) P. Juillard, Bull(Fr) [3] **33**, 1175 & 1179(1905)

2,4-Dinitrodiphenylamine,
$(O_2N)_2C_6H_3.NH.C_6H_5$; red ndls (from benz), mp 157°; sol in acet, chlf & hot alc; can be prepd by interaction of 4-chloro-1,3-dinitrobenzene and aniline or by other methods (Refs 1–4)

Tavernier & Lamouroux (Ref 5) detd its Q_c^v as 1445.1kcal/mol and Q_f^v as minus 7.23 kcal/mol

Refs: 1) Beil **12**, 751, (362) & [407] 2) P. Juillard, Bull(Fr) [3], **33**, 1182(1905) 3) J. Marshall, IEC **12**, 336(1920) 4) T.L. Davis & A.A. Ashdown, JACS **46**, 1051(1924 5) P. Tavernier & M. Lamouroux, MP **39**, 338(1957)

2,4'-Dinitrodiphenylamine,
$O_2N.C_6H_4.NH.C_6H_4.NO_2$; brick-red crysts, mp 220–221.5°; insol in w; sl sol in alc and acet. It was isolated by Davis & Ashdown (Ref 2) and by Becker & Hunold (Ref 3) as one of the products formed during aging of NC proplnts stabilized with DPhA. It gives on nitration 2,4,4'-TrinitroDPhA (Ref 4)

Refs: 1) Beil **12**, 715 & [387] 2) T.L. Davis & A.A. Ashdown, JACS **46**, 1051(1924) 3) F. Becker & G.A. Hunold, SS **33**, 244–47(1938) 4) Davis (1943), 312

2,6-Dinitrodiphenylamine,
$(O_2N)_2C_6H_3.NH.C_6H_5$; red-orn crysts, mp 106–108°; sol in alc & acet; can be prepd by heating 2-chloro-1,3-dinitrobenzene with aniline and Na acetate in alc

Refs: 1) Beil **12**, 758, (365) & [413] 2) K. von Auwers & E. Frese, Ber **59**, 553(1926)

3,3'-Dinitrodiphenylamine,
$O_2N.C_6H_4.NH.C_6H_4.NO_2$; orn plts, mp 186.5°; bp – expl mildly; insol in w and in ligroin; sol in cold pyridine & NBenz; sl sol in chlf, alc, eth & glac AcOH; can be prepd by interaction of 3-bromo-1-nitrobenzene and 3-nitroacetanilide

Refs: 1) Beil **12**, not found 2) A. Albert & W. Linnel, JCS **1936**, p91 3) H. Hodgson & D. Dodgson, JCS **1948**, pp1004–06

3,4'-Dinitrodiphenylamine,
$O_2N.C_6H_4.NH.C_6H_4.NO_2$; pale-yel crysts (from chlf), mp 217°

Refs: 1) Beil **12**, [387] 2) H. Ryan & T. Glover, ProcRoyIrishAcad **34B**, 101(1918) & CA **12**, 2542(1918)

4,4'-Dinitrodiphenylbenzene, $O_2N.C_6H_4.NH.C_6H_4.NO_2$; yel ndls with bluish reflex, mp 216–216.5°; insol in w; sl sol in alc and acet. It is one of the products isolated by Davis & Ashdown (Ref 2) and by Becker & Hunold (Ref 3) from aged NC proplnts stabilized by DPhA. It gives on nitration explosive 2,4,4'-Trinitrodiphenylamine (Ref 4)
Refs: 1) Beil **12**, 716, (351) & [387] 2) T.L. Davis & A.A. Ashdown, JACS **46**, 1051(1924) 3) F. Becker & G.A. Hunold, SS **33**, 213–15 (1938) & CA **32**, 3501(1938) 4) Davis (1943) 312

Dinitro-N-nitroso-diphenylamine, $C_{12}H_8N_4O_5$; mw 288.22, N 19.44%. Four isomers are described in Beil:
2,2'-Dinitro-N-nitroso-diphenylamine [called Bis-(2-nitrophenyl)-nitrosamin in Ger], $O_2N.C_6H_4.N.NO.C_6H_4.NO_2$; not obtd in pure state; dec in alc to 2,2'-Dinitro-DPhA; obtd with the 2,4'-Dinitro-N-nitroso deriv by nitrating 2-nitro-N-nitroso-diphenylamine in glac acet ac with nitric acid below 10°(Ref 1)
2,4'-Dinitro-N-nitroso-diphenylamine, $O_2N.C_6H_4.N.NO.C_6H_4.NO_2$; dk rose colored pltlts, mp 160–62°(dec); sol in acet, v sl sol in glac acet ac, toluene, alc & MeOH; obtd with other products on nitrating Diphenylnitrosamine with 2 moles of nitric acid (d 1.43) (Ref 2)
4,4'-Dinitro-N-nitroso-diphenylamine, $O_2N.C_6H_4.N.NO.C_6H_4.NO_2$; dk orn-yel prisms, mp 150°(dec); v sl sol in alc, toluene & glac acet ac; obtd with a tetranitro deriv on treating 4-nitrodiphenylnitrosamine with 6 moles of nitric acid (d 1.43) (Ref 3)
2,4-Dinitro-N-nitroso-diphenylamine, $(O_2N)_2C_6H_3.N.NO.C_6H_5$; lt yel prisms (from chlf), mp 149–51°; sol in concd sulfuric acid giving an orn-yel color; prepd by treating for several days 2,4-Dinitrodiphenylamine with isoamylnitrite in glac acet acid in the cold (Ref 4)

Other props & methods of prepg Dinitro-N-nitroso-diphenylamine are found in the Refs
Refs: 1) Beil **12**, 697 2) Beil **12**, 728 & [396] 3) Beil **12**, 728, (355) & [396] 4) Beil **12**, [413] 5) P. Juillard, Bull(Fr) [3] **33**, 1175(1905) 6) F. Becker & G.A. Hunold, SS **33**, 213, 244(1938) & CA **32**, 9501(1938) (Prepn & formation of DPhA Dinitro-N-nitroso derivs) 7) W.A. Schroeder et al, IEC **41**, 2818(1949) & CA **44**, 1707(1950) (Nitro-nitroso derivs of DPhA formed in double base proplnts during accelerated aging) 8) W.A. Schroeder, JACS **73**, 1122(1951) & CA **45**, 6004(1951) (H bonding & relative adsorption affinities on silicic acid of nitrated derivs of DPhA) 9) W.A. Schroeder et al, AnalChem **23**, 1740(1951) & CA **46**, 5434 (1952) (UV & visible absorption spectra in alc)

4,4'-Dinitro-diphenylnitramine; Bis (4-nitrophenyl) -nitramine; or N,4,4'-Trinitrodiphenylamine, $O_2N.C_6H_4.N.NO_2.C_6H_4.NO_2$; mw 304.22, N 18.42%; crysts, dec on crystn from ethyl acet forming 4,4 -Dinitro-diphenylamine; was prepd by action of NO on 4,4'-Dinitrodiphenyl nitrogen oxide, $(O_2NC_6H_4)_2NO$, in cold chlf (Refs 1 & 2)
Refs: 1) Beil **12**, [396] 2) H. Wieland & K. Roth, Ber **53B**, 227(1920) & CA **14**, 2638 (1920)

Trinitrodiphenylamines, $C_{12}H_8N_4O_6$; mw 304.22, N 18.42%. Following isomers are described in the literature:
2,4,2'-Trinitrodiphenylamine, $(O_2N)_2C_6H_3.NH.C_6H_4.NO_2$; yel prisms, mp 186–186.2°; sol in concd sulfuric acid with an orn-yel color; was prepd by heating 4-chloro-1,3-dinitrobenzene & 2-nitroaniline in alc in a tube at 175°(Refs 1 & 2)
Refs: 1) Beil **12**, 752 & [408] 2) T.L. Davis & A.A. Ashdown, IEC **17**, 675(1925)
2,4,3'-Trinitrodiphenylamine, $(O_2N)_2C_6H_3.NH.C_6H_4.NO_2$; yel ndls (from glac acet ac) or grn-yel crysts (from acet or pyridine), mp 193–95°; readily sol in acet, glac acet ac & acetic anhydride; mod sol in benz, chlf, alc, eth & CS_2; sl sol in petr eth; was prepd by reaction of 4-chloro or 4-bromo-1,3-dinitrobenzene & 3-nitroaniline, and by other methods (Refs 1 & 2)
Refs: 1) Beil **12**, 752, (362) & [408] 2) H. Ryan & T. Glover, ProcRoyIrishAcad **34B**, 102(1918) & CA **12**, 2542(1918)
2,4,4'-Trinitrodiphenylamine, $(O_2N)_2C_6H_3.NH.C_6H_4.NO_2$; yel ndls, mp 186–186.7°(dec); sol in benz, acet anhyd, alc, chlf & eth. It is one of the products

isolated by Davis & Ashdown (Ref 2) and Becker & Hunold (Ref 3) from aged NC proplnts stabilized by DPhA. It can be prepd from reaction of 4-chloro-1,3-dinitrobenzene & 4-nitroaniline in alc at 150°

Refs: 1) Beil **12**, 752, (362) & [409] 2) T.L. Davis & A.A. Ashdown, JACS **46**, 1051(1924) 3) F. Becker & G.A. Hunold, SS **33**, 213–15 & 244–47(1938)

2,6,2′-Trinitrodiphenylamine, $(O_2N)_2C_6H_3.NH.C_6H_4.NO_2$; crysts (from glac acet ac), mp 220–22°; was prepd when 2,6-dinitroaniline & o-nitroiodobenzene in tech nitrobenz in the presence of Cu powd & K_2CO_3 were heated at 205–10° for 10 mins. Yield 44% (Ref 2)

Refs: 1) Beil, not found 2) B. Hegedüs, Helv **33**, 770(1950) & CA **44**, 8355(1950)

2,4,6-Trinitrodiphenylamine or Picrylaniline, $(O_2N)_3C_6H_2.NH.C_6H_5$; orn-red ndls (from eth acet) or orn colored crysts (from benz, acet or alc), mp 179–80°; was prepd by warming Picrylchloride & aniline in alc, and by other methods (Refs 1 & 2)

Refs: 1) Beil **12**, 765, (368) & [421] 2) T.L. Davis & A.A. Ashdown, JACS **46**, 1051(1924) 3) G. Leandri & A. Tundo, AnnChim(Rome) **44**, 479(1954) & CA **49**, 15783(1955) 4) R. Schaal, JChimPhys **52**, 796(1955) & CA **50**, 5374(1956) (pK acidity function in mixts of w & ethylenediamine)

X,X,X-Trinitrodiphenylamine, $C_{12}H_8N(NO_2)_3$, yel ndls (from acetic acid), mp 135°; readily sol in alc; glac acet ac, eth acet & benz; sol in CS_2; insol in w; was obtd by heating acetyl diphenylamine with nitric acid (d 1.03)

Refs: 1) Beil **12**, 247 2) Norton & Allen, Ber **18**, 1997(1885)

3,X,X-Trinitro-N-nitrosodiphenylamine, $C_{12}H_7N_5O_7$; mw 333.22, N 21.02%; yel ndls (from acet + chlf), mp – softens at 179° & melts at 184–85°(dec); sol in chlf & glac acet ac; sl sol in alc; v sl sol in acet; gives with alc KOH a blood-red soln; was prepd by prolonged treatment of 3-nitro-N-nitrosodiphenylamine with 3 moles of nitric acid (d 1.42) in glac acet acid (Refs 1 & 2). No expl props are reported

Refs: 1) Beil **12**, [383] 2) H. Ryan & T. Glover, ProcRoyIrishAcad **34B**, 104(1918) & CA **12**, 2542(1918)

Tetranitrodiphenylamines (TeNDPhA), $C_{12}H_7N_5O_8$, mw 349.22, N 20.06%

Before description of various isomers we are including here an abstract from the paper of J.B. Tingle & W.A. Lawrance, TransRoySocCanada, **12**, III, 7–11(1918) & **13**, 1462 (1919):

Although nitration of DPhA with nitric acid is difficult, it could be accomplished by placing 1 mol of DPhA in a large beaker surrounded by ice-water and adding slowly, with stirring, 8 mols of concd nitric acid. After thorough cooling, water was added slowly and the ppt was separated and washed with w until free from acid. Then the ppt was treated with nitrobenzene and the portion soluble in NBenz was treated by slowly adding alcohol. The resulting ppt proved to be a Tetranitrodiphenylamine of unknown structure. Its mp was above 250° and it was sl sol in eth, acet chlf and alc; insol in petr eth and in CCl_4

The same investigators reported the prepn of 2,4,6,4′-, 2,4,2′,4′-, 2,4,6,2′- and 2,4,6,3′-isomers and also of a TeNDPhA which melted at 191–92° and was very sol in ethyl acetate. On treating this soln with petr eth a ppt which melted at 191–192° was obtd. The product was insol in petr eth, eth and carbon tetrachloride; sl sol in alc & chlf and sol in acetone; it gave a red coloration with warm NaOH soln

Following TeNDPhA isomers are described in the literature:

2,4,2′,4′-Tetranitrodiphenylamine, $(O_2N)_2C_6H_3.NH.C_6H_3(NO_2)_2$; red-brn lfts (from alc); yel ndls or prisms (from glac AcOH); mp 199–201.5°; diff sol in cold alc, eth acet or toluene; sol in acet (2.26% at 17°); solubilities in other solvents were detd by L. Desvergnes; they are listed in Ref 1, p [409]; can be prepd by nitration of dinitrodiphenylamines (Refs 1 & 2) or by nitration of DPhA by $HNO_3+CCl_3.COOH$ (Ref 3)

Refs: 1) Beil **12**, 752, (362) & [409] 2) P. Juillard, Bull(Fr), [3], **33**, 1186(1905) 3) J.B. Tingle & W.A. Lawrance, TransRoySocCanada, **12**, III, 7–11(1918) & CA **13**, 1462(1919) 4) J. Marshall, IEC **12**, 337(1920) 5) J. Forrest et al, JChemSoc **1946**, 454 & CA **41**, 405(1947) (Prepn) 6) W.E. Bachmann et al, JOrgChem **13**, 390(1948) & CA **43**, 179(1949) (Prepn) 7) W.A. Schroeder, JACS **73**, 1122 (1951) & CA **45**, 6004(1951) (H bonding & relative adsorption affinities) 8) W.A. Schroeder et al, IEC **43**, 939(1951) & CA **45**, 10585 (1951) (Formation in double-base proplnts during accelerated storage) 9) W.A. Schroeder et al, AnalChem **23**, 1740(1951) & CA **46**, 5444 (1952) (UV & visible absorption spectra in ethyl alcohol) 10) J.H. Dijkema, USP 2612523(1952) & CA **47**, 8775(1953) (Prepn)

2,4,6,2'-Tetranitrodiphenylamine or **Picryl-2'-nitroaniline,** $(O_2N)_3C_6H_2.NH.C_6H_4.NO_2$; golden-yel prismatic ndls, mp 233–234°; sl sol in eth acet & toluene; can be prepd by nitration of Picrylaniline with mixed nitric-acetic acid (Refs 1 & 2) or by action of Picryl chloride on o-Nitroaniline (Ref 3)
Refs: 1) Beil **12**, 765 2) P. Juillard, Bull(Fr), [3], **33**, 1187(1905) 3) J.B. Tingle & W.A. Lawrance, TransRoySocCanada **12**, III, 7–11 (1918) & CA **13**, 1462(1919)

2,4,6,3'-Tetranitrodiphenylamine or **Picryl-3'-nitroaniline,** $(O_2N)_3C_6H_2.NH.C_6H_4.NO_2$, orn-yel crysts (from eth acet), mp 210–213°; sol in hot abs alc; diff sol in cold alc; insol in eth; can be prepd by treating Picrylchloride with 3-nitroaniline. It is a mild expl; gives on nitration 2,4,6,2',3',4'-HexanitroDPhA
Refs: 1) Beil **12**, 765, (369) & [422] 2) (?). Austen, Ber **7**, 1248(1874) 3) E. Wedekind, Ber **33**, 431(1900) 4) J.B. Tingle & W.A. Lawrance, TransRoySocCanada **12**, III, 7–11(1918) & CA **13**, 1462(1919)

2,4,6,4'-Tetranitrodiphenylamine or **Picryl-4'-nitroaniline,** $(O_2N)_3C_6H_2.NH.C_6H_5.NO_2$; golden-yel lfts or prisms; mp 222–223°; insol in cold alc; sl sol in eth, toluene, chlf, AcOH & carbon tetrachloride; sol in acet & & eth acet; can be prepd by nitration of Picrylaniline with mixed nitric-acetic acid or nitric-oxalic acid. It gives on nitration 2,4,6,2',4'-PentanitroDPhA
Refs: 1) Beil **12**, 765, (369) & [422] 2) P. Juillard, Bull(Fr), [3], **33**, 1188(1905) 3) J.B. Tingle & W.A. Lawrance, TransRoySocCanada **12**, III, 7–11(1918) & CA **13**, 1462(1918) 4) H. Ryan & T. Glover, ProcRoyalIrishAcad **34B**, 104(1918) 5) M. van Duin & B. van Lennep, Rec **38**, 359(1919)

2,4,3',X-Tetranitrodiphenylamine, $(O_2N)_2C_6H_3.NH.C_6H_3.(NO_2)_2$; yel prisms (from alc + acet), mp 190°; diff sol in alc; sol in chlf; very sol in acet; was obtd after leaving for one week an acetic acid soln of 2,4,3-TrinitroDPhA with iso-amylnitrite. This seems to be the same as the unidentified product of mp 191–192° obtd by Tingle & Lawrance (See above)
Refs: 1) Beil **12**, [409] 2) H. Ryan & T. Glover, ProcRoyIrishAcad **34B**, 104(1918) & CA **12**, 2542(1918)

Pentanitrodiphenylamines, $C_{12}H_6N_6O_{10}$, mp 394.22, N 21.32%. Following isomers are described in the literature:
2,4,6,2',4'-Pentanitrodiphenylamine or **Picryl-2',4'-dinitroaniline,** $(O_2N)_3C_6H_2.NH.C_6H_3(NO_2)_2$; yel prisms (from toluene), mp 196–197°; explodes on heating above its mp; diff sol in cold alc, toluene or AcOH; can be prepd by reaction of Picrylaniline with nitric acid (d 1.49) at RT for 24 hrs

Forms salts, some of them expl
Refs: 1) Beil **12**, 765, (369) & [422] 2) P. Juillard, Bull(Fr), [3], **33**, 1189(1905) 3) H. Ryan & T. Glover, ProcRoyIrishAcad **34B**, 105(1918) 4) M. van Duin & B. van Lennep, Rec **38**, 363(1919) 5) W.A. Schroeder et al, AnalChem **23**, 1740(1951) & CA **46**, 5434 (1952) (UV & visible spectra)

2,4,6,3',4'-Pentanitrodiphenylamine or **Picryl-3',4'-dinitroaniline,** $(O_2N)_3C_6H_2.NH.C_6H_3(NO_2)_2$, brn-yel crysts (from AcOH); mp 232°; explodes on heating above mp; solubilities not given; was prepd by heating at 140° a sealed tube of Picrylchloride with 3,4-dinitroaniline. It gives on nitration with mixed nitric-sulfuric acid the hexanitro compd, probably 2,4,6,2',3',4'-HexanitroDPhA
Refs: 1) Beil **12**, (369) 2) M. van Duin & B. van Lennep, Rec **38**, 363(1919)

Hexanitrodiphenylamines, $C_{12}H_5N_7O_{12}$, mw 439.22, N 22.33%, OB to CO_2 minus 52.8%.

Following isomers are described in the literature

2,4,6,2',4',6'-Hexanitrodiphenylamine (HNDPhA); Dipicrylamine, Hexite, Hexil or **Hexyl** [Hexa, Hexamin, Hexanitrodiphenylamin, Hexyl or SWN/A (Schiesswolle Neuer Art) (New kind of Guncotton in Ger)]; (Hexanitrodiphenylamine in Fr); (Esanitrodifenilammina o Exil in Ital); (Hexanitrodifenilamina o Hexamina in Span); (Gheksonitrodifenilamin in Rus), $(O_2N)_3C_6H_2.NH.C_6H_2(NO_2)_3$; small yel needles existing in two polymorphic forms of which the orthorhombic is stable at RT; cryst density 1.653; cannot be cast-loaded, but when pressed with 1% stearic acid at 5000 psi its sp gr is 1.43; at 10,000 psi 1.59 and at 15,000 psi 1.60g/cc; mp 243–244.5° (decomp). It is insol in chlf and petr eth; nearly insol in water & benz; sparingly sol in eth & cold AcOH; fairly sol in alc; readily sol in cold acet and in warm acetic and nitric acids; sol in alkalies except KOH

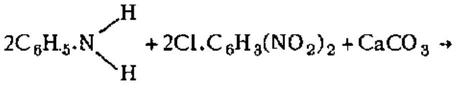
It was prepd in 1874 by Austen (Refs 1 & 2) by nitrating Picryl-p-nitroaniline and in the same year by Gnehm (Refs 1 & 3) by the nitration of methyldiphenylamine. Mertens (Ref 4) was the first to prep it by direct nitration of DPhA. He used fuming nitric acid mixed with concd sulfuric. Juillard (Ref 7) also nitrated DPhA to hexanitro, but found that it was more convenient to nitrate 2,4-DNDPhA

The large scale prepn of HNDPhA by direct nitration of DPhA was conducted, accdg to Davis (Ref 26, p184), in 1910 in Germany

The process from dinitrochlorobenzene and aniline originally described in GerP of 1895 (Ref 6) was reported by Carter (Ref 11) and studied further by Hoffman & Dame (Ref 16) and then by Marshall (Ref 18)

In the two-stage process described by Davis (Ref 26, p185), 70g of aniline and 32g of precipitated Ca carbonate are stirred together with w in such a manner as to form a homogeneous suspension, and the mixt is heated to ca 60°. Dinitrochlorobenzene, 150g, previously melted, is poured in slowly in a fine stream while stirring is continued and the mixture is heated gradually to ca 90°, the rate of heating being regulated by the progress of the reaction

The following reaction takes place:



$$2C_6H_5.N{<}^{H}_{H} + 2Cl.C_6H_3(NO_2)_2 + CaCO_3 \rightarrow$$

$$CaCl_2 + CO_2 + H_2O + 2C_6H_5.NH.C_6H_3(NO_2)_2$$

The resulting 2,4-Dinitrodiphenylamine is washed with HCl to free it from aniline and Ca carbonate, then with w until free from chlorides, and finally dried in the oven at 100°

Fifty grams of finely pulverized 2,4-DNDPhA is added in small portions to 420g of nitric acid (sp gr 1.33) which is stirred vigorously while the temp is maintained at 50–60°. The progress of the nitration is followed by observing the color change from red of the dinitrocompd to yellow of the tetranitrocompd. After all has been added, the temp is raised to 80–90° and kept for 2 hrs longer while stirring is continued. After the mixt has cooled, the product is filtered, washed with w until free from acid, and dried in the air or in the oven at 100°

Fifty g of pulverized 2,4,2',4'-TeNDPhA is added slowly with stirring, during one hr, to a mixt of 250g of nitric acid (sp gr 1.50) and 250g of sulfuric acid (sp gr 1.83). After all has been added the mixt is allowed to stand for 3 hrs at RT and then drowned in ice-water. The 2,4,6,2',4',6'-HNDPhA is filtered off, washed thoroughly with w, dried in the air, recrystallized by dissolving in acet and precipitated with petr eth

In the process patented by James et al (Ref 18a), Tetryl and aniline in warm acetone (or benzene) furnish methylnitramine and Trinitrodiphenylamine (TNDPhA). Then the latter is nitrated to HNDPhA

Stettbacher (Ref 22, p292) described the following one-stage laboratory method of nitration of DPhA to HNDPhA:

Fifty grams of roughly pulverized DPhA was gradually added to 500ml of sulfuric acid (sp gr 1.84) contained in a 1-liter flask. The flask was swirled during the addn to facilitate the dissolution of DPhA. The soln was transferred to a drop-funnel and added drop by drop to 500g (333ml) of fuming nitric acid (sp gr 1.5) contained in a 5-liter flask. The flask was swirled and cooled during the addition, so that its blue-colored contents remained at ca 30°. Then the flask was heated on a water-bath until the evolution of nitrogen oxide fumes ceased and the blue color disappeared, while at the same time precipitation of yellow crysts of HNDPhA took place. The resulting slurry was cooled, poured into ca 5 liters of ice-water and passed thru a Nutsche. The ppt was washed first with cold and then with hot water until acid-free and dried at 100°. Yield of crude product with mp 236° was 88g. Purification was done by dissolving in hot nitric acid of sp gr 1.36–1.40, followed by cooling. The resulting product had mp of 249°

Another lab method of prepn was described by Stettbacher in Ref 42. Manufacturing methods used in Germany during WWII were described in Refs 27, 29a, 30 & 44. Following is the method used at Allendorf Fabrik of WASAG:

To a charge of 1000kg of 99% nitric acid placed in a V2A stainless steel nitrator of 2 cu meters capacity (fitted with an agitator rotating at 60RPM, a cooling jacket and cooking coils) 300kg of pulverized DPhA was gradually added while maintaining the temp at 90°. After completion of reaction, the soln was diluted with weak nitric acid and cooled to 30–40°. Total time required for nitration and cooling was 10 hrs. The pptd HNDPhA was filtered thru a Nutsche and washed with cold w. Then it was removed, slurried with w and fed to a centrifuge for final wringing. From there the crysts were transferred to a "tray-drier" and then to screens. The yield was 420kg per 300kg of DPhA

HNDPhA was used by the Germans during WWII in mixtures with 30–40% of TNT for cast-loading of torpedo warheads, sea mines, depth charges and the 400kg "skip bomb" known as "Kurt Apparatus" (See Ref 44, p Ger 17, item 11)

A similar expl mixture was used in Sweden under the name of **Novit**. HNDPhA was also used by the Germans prior to WWII in the mixtures contg 10–40% of PA, DNT, DNN and vegetable meal 0–4%. It was also used in commercial expls known as **Neurodits**. These expls contd surplus military expls of WWI, mostly TNT. A mixture of HNDPhA with Al powder was known as **Schiesswolle 18** or **TSMVI-101** (Ref 33)

Japanese prepd HNDPhA during WWII by treating dinitrochlorobenzene with aniline in the presence of Na carbonate to obtain 2,4-Dinitrodiphenylamine, followed by one-stage nitration with mixed nitric-sulfuric acid, as described in Ref 31

Japanese used HNDPhA in the following expl mixts: **Seigata** or **Type 97**: HNDPhA 40 & TNT 60% used in bombs and to a certain extent in torpedo warheads and depth charges. **Otsu-B** or **Type A Expl**: HNDPhA 24, TNT 60 & Al (or Zn) powder 16% – used in torpedo warheads, sea-mines and depth charges (Ref 33 & Ref 49, pp8–2 & 8–3)

Various methods of HNDPhA prepn, including the methods already listed, are described in Refs 10, 12, 13, 14, 15, 17, 19, 21, 22, 23, 24, 25, 28, 29, 32, 33, 34, 35, 36 & others

Some of these refs also described props and uses of HNDPhA. History of its prepn and uses is described in Refs 14 & 26

HNDPhA is a pseudo-acid which was investigated by Aleksandrov (Ref 8) and Hantzsch & Opolski (Ref 8a)

Accdg to Davis (Ref 26, p184) and All & En Expls (Ref 33), HNDPhA is more poisonous than NG and attacks the skin, causing severe blisters which resemble burns. Its dust is injurious to the mucous membranes of the mouth, nose and lungs

A detailed study of toxicity was done by Hoffman & Dame (Ref 16) and by Marshall (Ref 18). Sax (Ref 47) reports that its toxicity details are unknown. Urbański (Ref 48) notes that HNDPhA seriously attacks the skin. First it dyes the skin yellow, then a serious eczema appears with blisters often reminiscent of those caused by burns. Air-borne dust of HNDPhA may cause severe irritation of mucous membranes and of the respiratory tract. Workers exposed to large quantities of HNDPhA should be protected from inhaling its dust and from contact with the skin. Good ventilation and protective masks & clothing should be provided. The toxicity of HNDPhA is considered to be much higher than that of TNT

Toxicity was also investigated by Rokstad (Ref 35)

Explosiveness of HNDPhA was first recognized by Hausermann in 1891 (Ref 5), who compared it with TNT. The first patent for a military expl contg HNDPhA, such as its mixture with 20% TNT, was granted in 1909 to von Schroeter (Ref 9)

Explosive properties of HNDPhA were assembled from different sources by Blatt

(Ref 28) and in All & En Expls (Ref 33). They are arranged here in alphabetical order:
Brisance by Sand Test – 0.5g crushed 49.5g sand, vs 48.0g for TNT (103% TNT) (Ref 22a); Hercules gave 105%
Brisance by Compression of Copper Block – 114% TNT (Ref 28)
Brisance by Kast Formula – 111% TNT (Vol 2 of Encycl, p B278)
Compatibility with Metals – does not attack common metals
Detonation Rate – 6898m/sec at density 1.58g/cc; 7150 at 1.67 (Ref 28); 7200 vs 6900 for TNT (Ref 33)
Explosion Temperature – 250° when heated at the rate of 5°/min; instantaneous expln temp is above 360° (Ref 28)
Heat of Combustion – 1314.1kcal/mole (Ref 37a)
Heat of Explosion – 327.1–405.6kcal/mole (Ref 37a)
Heat of Formation – minus 10.3kcal/mole (Ref 37a)
Hygroscopicity at 25° and 100% RH – gains 0.09% (Ref 28)
Impact Sensitivity by 2kg BM Apparatus – max fall for 0/5 shots 35cm vs 27.5cm for Tetryl (Ref 28)
Impact Sensitivity by 2kg PicArsnApp – 9 inches vs 14 for TNT (Ref 33)
Initiation Sensitivity – minimum priming chge of 90/10-MF/$KClO_3$ was 0.18g when 0.4g of Hexil was loaded in the detonator with a reinforced cap loaded at 200psi (Ref 28)
Power (or Strength) by Ballistic Mortar: 111–115% TNT (Ref 28); 113% TNT (Ref 33); 115% TNT (Ref 37)
Power (or Strength) by Trauzl Test: 101 to 106% PA (ca 110% TNT) (Ref 28); 101% PA & 110% TNT (Ref 37)
Rifle Bullet Sensitivity – ca 87% detonations from impact of .30 cal bullet fired from US Army rifle at a distance of 30 yards against 1 lb of HNDPhA placed in cubical 3.5 inch tin box; no detonations were produced with a cardboard box (Refs 28 & 33)
Thermal Stability at 95° – no change in thirty 8-hr days (Ref 28)
Thermal Stability at 135° – no acidity and no expln in 300 hrs (Ref 28)
Toxicity – See above
Vacuum Stability at 120° – 4.4ml gas evolved from 5g sample in 48hrs (Ref 28)
Velocity of Detonation. See Detonation Rate
Uses: HNDPhA was not used in the US as a component of military explosives, but was used in Germany, Japan and Sweden, as was described above

It can serve as a reagent for determination of potassium (Ref 46, p537-L)

The explosive mixture **Hexamite**, contg TNT 60, HNDPhA 24 & Al 16% had the following properties: mp ca 81°, brisance (by Sand Test) 116% TNT, detonation velocity 6900m/sec at density 1.72, power by Ballistic Mortar 130% TNT and by Trauzl Test 116% TNT. Its uses during WWII were listed above

LeRoux (Ref 39) examined HNDPhA from the point of view of its resistance to heat, to ascertain whether it is suitable for use in blasting operations in search of petroleum. Heating for 3 hrs at 140, 160 & 180° did not cause any decompn and all samples detonated after tests without difficulty when intiated by detonators contg 2g of MF, or its equivalent

Refs for 2,4,6,2',4',6'-Hexanitrodiphenylamine:

1) Beil **12**, 766, (369) & [422] 2) P.T. Austen, Ber **7**, 1279(1874) 3) R. Gnehm, Ber **7**, 1399(1874) 4) K.H. Mertens, Ber **11**, 843(1878) 5) (?). Hausermann, ZAngewChem **17**, 508–10(1891) 6) GriesheimChemFabrik, GerP 86295(1895) 7) P. Juillard, Bull(Fr) [3], **33**, 1190(1905) 8) V. Aleksandrov, JRussPhysChemSoc **39**, 1391(1907) 8a) A. Hantzsch & S. Opolski, Ber **41**, 1745(1908) 9) O. Freiherr von Schroeter, USP 934020 (1909) 10) H. Kast, SS **8**, 173(1913) 11) T. Carter, SS **8**, 205–06(1913) & CA **7**, 3228(1913) 12) M. Giua, SS **10**, 111–12 (1915) & CA **9**, 3362(1915) 13) Marshall **1** (1917), 271–72 14) Anon, SS **12**, 233(1917) & CA **12**, 630(1918) 15) H. Ryan & T. Glover, ProcRoyIrishAcad **34B**, 97–105(1918) 16) E.J. Hoffman & P.A. Dame, JACS **41**, 1014(1919) 17) C.F. van Duin & B.C. van Lennep, Rec **39**, 145(1920) 18) J. Marshall, JIEC **12**, 336–40(1920) & CA **14**, 633(1920) 18a) T.C. James, U.I. Jones & R.I. Lewis,

BritP 130357(1920) & CA **14**, 124(1920) 19) H. Kast, "Spreng- und Zündstoffe" Braunschweig (1921), 279 20) C. Henning, USP 1473818(1923) & CA **18**, 472(1924) 21) Marshall **3**(1932), 72–5 22) Stettbacher (1933), 292–94 22a) L.V. Clark, IEC **25**, 663 & 1385(1933) (Expl props of HNDPhA) 23) Pepin Lehalleur (1935), 264–65 24) Thorpe **4**(1940), 489 25) Clift & Fedoroff **2**(1943), pp H3 & H4 26) Davis (1943), 184–87 27) A. Stettbacher, Protar (Swiss) **9**, 3345(1943) 28) A.H. Blatt, OSRD **2014**(1944) 29) Perez Ara (1945), 582–86 29a) O.W. Stickland et al, PBRept **1820**(1945) 30) C.H. Brooks et al, PBRept **38154**(1945) (Manuf and uses of Hexyl in Germany) 31) G.C. Tibbitts et al, PBRept **50394**(1946) (Manuf & uses of Hexyl in Japan) 32) Vivas, Feigenspan & Ladreda, Vol **2**(1946), 275–80 (Hexamina) 33) All & En Expls (1946), 104 & 157 34) J. Forrest et al, JChemSoc **1946**, 454 & CA **41**, 405(1947) 35) I. Rokstad, ActaDermato-Venereol **26**, Suppl 15(1946); BritJDermatol **59**, 115–16(1947) & CA **41**, 4228(1947) (Toxicity) 36) J. Taylor, Research **2**, 67–76(1947) (Advances in British commercial expls) 37) ADLPureExplCompds, Pt **1**(1947), p98 37a) Ditto, Pt **2** (1948), p245 38) Stettbacher (1948), 78–9 38a) L. Médard & M. Thomas, MP **31**, 173(1949) & CA **46**, 11684(1952) (Heat of combstn & method of purifying HNDPhA) 39) A. LeRoux MP **32**, 200–201(1950) 39a) W.A. Schroeder et al, AnalChem **23**, 1740(1951) & CA **46**, 5434(1952) (UV & visible absorption spectra in alcohol) 39b) ÉtatFrançais, FrP 971644(1951) & CA **46**, 9311(1952) (Use of HNDPhA as a suitable combustible for expl powders) 39c) W.C. McCrone, AnalChem **24**, 592(1952) (Crystallographic data of HNDPhA) 40) Stettbacher, Pólvoras (1952), 104–05 (Hexilo or Hexamina) 41) Belgrano (1952), 140 (Exil or Esanitrodifenilammina) 42) A. Stettbacher, Explosivstoffe **No 5/6**, 63–4(1953) (Lab prepn of HNDPhA) 42a) A. Langhans, Explosivst **1/2**, 7(1954) (Thermal decompn of HNDPhA) 43) L. Médard, MP **37**, 26(1955) (Expl props) 43a) K. Pan & Sui-Fong Lin, JChineseChemSoc, Ser II, **2**, 1–14(1955) (Engl) & CA **49**, 14534(1955) (Polarographic study of HNDPhA) 44) Fedoroff et al, PATR **2510**(1958), p88 (Hexa) 45) Giua, Tratto **6**(I)(1959), 325–26 (Esanitrodifenilammina) 46) CondChemDict (1961), 573-L 47) Sax (1963), p875 48) Urbański **1** (1964), p562–66 49) Anon, "Military Explosives", TM 9-1300-214(1967), p8–4 50) F. Taylor, Jr, USP 3418372 (1968) & CA **70**, 69753k (1969) (Prepn of K HNDPh, useful as a booster expl in high temp applications, such as in missiles)

2,4,6,2′,4′,6′-Hexanitrodiphenylamine, Analytical Procedures

There are no US specifications covering the analysis of HNDPhA or its detn in expl mixts. Thomas (Ref 1) used the solubility method in detg the qualitative & quantitative analyses of binary mixts of HNDPhA & TNT and of other mixts of nitrated derivs or of nitrates. A correction for the dissolved fraction of the less sol component must be established experimentally. Approx qualitative analysis by "framing" is described. From the amt of solv just insufficient to dissolve a known wt of sample and the addnl amt reqd to effect soln, identity of the sample is detd. Other tests used are mp, colors obtd in acetone soln with NaOH & NH_3 and the crystn form

See also Refs given below

Refs: 1) M. Thomas, MP **36**, 133–62(1952) & CA **49**, 16435–36(1955) 2) S. Kertes & J.M.E. Goldschmidt, JChemSoc **1956**, 401–04 & CA **50**, 6189(1956) (Spectrophotometric study of HNDPhA. Molecular & forms in dioxane) 3) W. Selig, US AtomEnerg Comm **UCLR-6903** (1962), 13pp & CA **57**, 11861(1962) (Spectrophotometric detn in N,N-dimethylformamide) 4) J. Hansson & A. Alm, JChromatog **9**, 385–87(1962) (French) & CA **58**, 10034–35 (1963) (Thin-layer chromatography applied to analysis of expls) 5) F. Barbulescu et al, Analele Univ "G.I. Parkon" Bucuresti, Ser-StiintNat, Chim **11**(35), 9–17(1962) & CA **61**, 308–09(1964) (Electrochemical props) 6) R.D. Tiwari & J.P. Sharma, Talanta **10**, 933–34(1963) (English) & CA **59**, 12185(1963)

(Spectrophotometric detn of HNDPhA in microgram quantities) 7) S.K. Yasuda, Explosivst **1963** (2), 34–38 & CA **60**, 14325(1964) (Identification N-Nitroso & N-Nitrodiphenylamines by two-dimensional thin-layer chromatography) 8) J. Hansson, Explosivst **11**, 73–77(1963) & CA **63**, 11237(1965) (Thin-layer chromatographic method for analysis of expls, useful for qual & quant detns)

Hexanitrodiphenylamine, Salts of. HNDPhA being a pseudo-acid reacts with oxides, hydroxides and carbonates of metals or of ammonia with formation of salts, most of them explosive

Following is the list of the more important of these salts:

Alkaline Salts were prepd and examined by Kast & Langhans (Ref 4) and by Treadwell & Hepenstrick (Ref 12)

Ammonium Salt, known as Aurantia, $NH_4C_{12}H_4N_7O_{12}$, mw 456.25; N 24.56%; red-brn crysts; mp explodes on heating; heat of combustion 3128cal/g (Ref 4). It can be prepd by the action of ammonia on 2,4,6,2',4',6 -Hexanitrodiphenylamine. It is a very sensitive expl, which was examined by Will (Ref 2) (Ref 9, pA7) (See also Refs 1, 4, 5, 7, 8, 10, 11, 13 & 14). It is more poisonous than NG. It was used in Germany for filling bombs

Barium Salt, $Ba(C_{12}H_4N_7O_{12})_2$; red crysts, mp – explodes on heating; sol in w & alc; was prepd by action of $Ba(OH)_2$ on hexanitrodiphenylamine

Cobalt Ammonia Salt, $[Co(NH_3)_6][C_{12}H_4N_7O_{12}]_3 + 2.5H_2O$; scarlet-red amorph powd, mp – dec on heating to 95° {Ref 1, p [423]}

Lead Salt, $Pb(C_{12}H_4N_7O_{12})_2+8PbO$, dk-red powd, mp – deflgr at 250°; insol in hot w {Ref 1, p [423]}

Lead Salt, Basic, $PbOH.C_{12}H_4N_7O_{12}$; mw 662.43, N 14.80%; was prepd in mixed or double crysts with LA by action of HNDPhA on a Pb salt in the presence of an Azide

Was proposed by Friederich (Ref 4a) for use in primers and percussion caps (Ref 9, p H4)

Potassium Salt, $KC_{12}H_4N_7O_{12}$, mw 477.30, N 20.54%. A purified product was produced by reacting HNDPhA with KOAc in acetone, stirring the mixt for 30 mins at RT, diluting with w, and chilling to ppt the product. It was collected, washed with water & dried. It was 99.7% pure and did not req recrystn before use (Ref 15) (See Refs 4, 6 & 12)

Silver Salt, $AgC_{12}H_4N_7O_{12}$; mw 546.09, N 17.94%; violet crysts (from acet) or red-yel crysts; mp – darkens ca 190°, decomp below 200° leaving a brown mass; explodes weakly when dropped on a surface of molten metal in a bath preheated to 310°. It was first prepd by Hantzsch & Opolski (Ref 3) from HNDPhA and Ag oxide in acetone soln. Taylor & Buxton (Ref 4b) claimed that a better method is to treat a concd soln of 340g of Ag nitrate with sufficient amt of ammonia to redissolve the pptd Ag hydroxide and to add the resulting soln (with stirring) to a hot soln of 8.78g of HNDPhA in 50ml of concd ammonia dissolved in 1800ml water. The resulting slurry is cooled and filtered to separate the fine red crysts of Ag salt. After rinsing them with w, alc, and ether, they are dried over concd sulfuric acid

Silver salt did not explode on impact of 500g weight dropped from a height of 100cm. When brought in contact with a flame it deflagrated, but when pressed into a detonator cap it was not ignited by a spit of a fuse. A chge of 0.4g pressed into a cap and initiated by 0.2g of MF (confined by a reinforcing cap) crushed ca 0.29g sand, as compared with 30g for 0.4g of TNT

Refs for Salts of 2,4,6,2',4',6'-HNDPhA: 1) Beil **12**, 766, (369) & [422] 2) W. Will, ChemInd **1903**, 130 (Ammonium salt) 3) A. Hantzsch & S. Opolski, Ber **41**, 1747(1908) (Silver salt) 4) H. Kast & A. Langhans, SS **14**, 1–4 & 24–7(1909) (Amm & other salts) 4a) W. Friederich, BritP 180605(1921) & CA **16**, 3399(1922) 4b) C.A. Taylor & E.P. Buxton, ArmyOrdn **7**, 69(1926) (Basic lead salt) 5) Pepin Lehalleur (1935), 264 (Jaune Aurantia) 6) J. Kielland, Ber **71B**, 220–26 (1938) & CA **32**, 2865(1938) (Formation of K salt serves in rapid colorimetric analysis) 7) M.V. Favorskii, CR AcadSciUSSR **25**, 71 (1939) & CA **34**, 3285(1940) (Amm salt) 8) O.G. Sheĭntsis, Zavodskaya Lab **8**, 1198 (1939) & CA **37**, 3007(1943) (Amm salt)

9) Clift & Fedoroff **2**(1943), pp A7 & H4 10) Davis (1943), 184 (Aurantia) 11) Pérez Ara (1945), 586 (Aurantia) 12) W.D. Treadwell & H. Hepenstrick, Helv **32**, 1903–10 (1949) & CA **44**, 2464(1950) (Alkaline salts) 13) S. Sato, JChemSocJapan, PureChemSect **72**, 182–87(1951) & CA **46**, 3452(1952) (Systematic analysis of alkali metal salts of HNDPhA by Ca HNDPhA) 14) S. Sato, Ibid **72**, 593–95(1951) & CA **46**, 6042(1952) (Colorimetric detn of K, Rb & Cs salts of HNDPhA) 15) G. Malomy & G. Rehde, Fühner-Wieland's Samml Vergiftungsfällen **14**, 30–35 (1952) & CA **47**, 3476(1953) (Dermatitis caused by Amm & Na HNDPhA salts) 16) S. Furberg & P. Larssen, ActaChemScand **6**, 965–66(1952) (English) & CA **47**, 6732 (1953) (X-ray investigation of HNDPhA and its Na, K(red form), K (violet form), Rb & Cs salts) 17) Giua, Trattato **6**(I)(1959), 326 (Aurantia) 18) M. Kyrs, CollectionCzechChemCommun **27**, 2380–90(1962) (Ger) & CA **58**, 12009(1963) (Solubility of Cs & Rb HNDPhA & the extraction of Cs HNDPhA from the aq phase by Nitrobenz) 19) O. Bagno & P. Bonet-Maury, CR **259**(17), 2841–44(1964) & CA **62**, 4811(1965) (Irradiation of Amm salt of HNDPhA by ^{60}Co γ-rays) 20) F. Taylor Jr, USP 3418372(1968) & CA **70**, 69753k (1969) (K salt of HNDPhA)

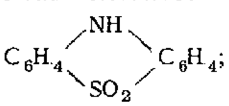
2,4,6,2′,3′, 4′-Hexanitrodiphenylamine, $(O_2N)_3C_6H_2.NH.C_6H_2(NO_2)_3$; mw 439.22, N 22.33%, OB to CO_2 minus 52.8%; yel crysts, mp 273–74°; bp – explodes at higher temps; insol in w, alc or eth; sl sol in hot AcOH. It can be prepd by nitration with mixed nitric-sulfuric acid of either 2,4,6,3′-TeNDPhA (Refs 1 & 2) or 2,4,6,3′,4′-PNDPhA (Ref 3) *Note:* The compd described in Beil **12**, 769 as 2,4,6,x,x,3′-HNDPhA with mp 261° is the same as impure 2,4,6,2′,3′,4′-HNDPhA.

Expl props of 2,4,6,2′,3′,4′-HNDPhA were detd by van Duin & van Lennep (Ref 3) and the following props are listed by Blatt (Ref 4): *Brisance* – not given *Explosion Temperature:* 287° when heated at the rate of 20°/minute and 282° at 5°/min *Impact Sensitivity by Kast Apparatus:* maximum fall with 2kg wt for 0/6 shots was 44–47cm vs 50–53cm for Tetryl *Power* – not given *Thermal Stability at 95°* – evolved nitrogen oxide fumes after 1 hour

It is less suitable than 2,4,6,2′,4′,6′-HNDPhA for use as an expl *Refs:* 1) Beil **12**, 765, (369) & [422] 2) P.T. Austen, Ber **7**, 1249(1874) 3) C.F. van Duin & B.C. van Lennep, Rec **38**, 361 & 368(1919) and **39**, 145 & 170(1920); JSCI **39**, 151A(1920) 4) A.H. Blatt, OSRD **2014**(1944)

Diphenylamine-sulfone and Derivatives

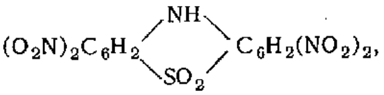
Diphenylamine-sulfone,



$C_6H_4\begin{smallmatrix}NH\\SO_2\end{smallmatrix}C_6H_4$;

mw 231.27, N 6.06%; ndls (from xylol), mp 257–59°; sol in alc; sl sol in hydrocarbon solvs; was prepd by heating 10-methyl-phenothiazine-9,9-dioxide with hydriodic acid (Ref 1)

2,4,6,8-Tetranitrodiphenylamine-sulfone or **2,4,6,8-Tetranitrophenothiazine-5,5-dioxide,**



$(O_2N)_2C_6H_2\begin{smallmatrix}NH\\SO_2\end{smallmatrix}C_6H_2(NO_2)_2$,

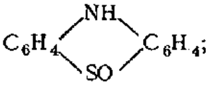
mw 411.27, N 17.03%; crysts (from Nitrobenz), mp 344–47°; was prepd by nitrating the parent compd with HNO_3-oleum mixt (See Refs below). The 2,4,6,8- & 1,3,7,9-Tetranitro derivs appear to be the same compd

It was used by the Germans, under the name of **Gelbmehl S** (Yellow Flour S), as one of the substitutes for Black Powder (Ref 3) *Refs:* 1) Beil **27**, 65 2) Beil, not found (Tetranitro) 2a) K. Pfaff, Reichsamt Wirtschaftsausbau Chem Ber **PB52021**, 1183(1942) & CA **42**, 9045(1948) (Prepn of 2,4,6,8-Tetranitro deriv) 3) CIOS Rept **25–18**(1945), pp 27–8 4) K. Toei, NipponKagakuZasshi **77**, 1270–72(1956) & CA **52**, 961(1958) (Prepn of Tetranitro deriv) 5) Ibid, **78**, 1379(1957) & CA **52**, 14286(1958) (Solubilities of K, Rb & Cs salts of the Tetranitro deriv) 6) C. Bodea & M. Raileanu, AcadRepPopulareRomîne, FilialaCluj. StudiiCercetăriChim **8**, 303(1957) & CA **54**, 22658(1960) (Prepn of 1,3,7,9-Tetranitrophenothiazine-5,5-dioxide, mp

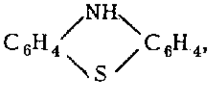
346–47°, by oxidn of the tetranitro-5-oxide deriv *with* H_2O_2, *or* by *nitrating* phenothiazine-5,5-dioxide or 3,7-dinitrophenothiazine-5,5-dioxide) 7) C. Bodea et al, ZhObshchKhim **34**(7), 2369(1964) & CA **61**, 9493(1964) (Prepn of 1,3,7,9-Tetranitro deriv, mp 344–45°)

Diphenylamine-sulfoxide and Derivatives

Diphenylamine-sulfoxide (called Phenthiazin-9-oxyd or "Diphenylaminsulfoxyd" in Ger),



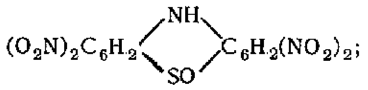
$C_6H_4\begin{smallmatrix} NH \\ SO \end{smallmatrix}C_6H_4$; mw 215.27, N 6.51%; ndls (from acet), mp 250°(dec); was prepd by warming phenthiazine,



$C_6H_4\begin{smallmatrix} NH \\ S \end{smallmatrix}C_6H_4$,

with aq alcoholic KOH & 30% hydrogen peroxide on a w bath (Ref 1)

2,4,5,7-Tetranitrodiphenylamine-sulfoxide [called 2.4.5.7-Tetranitro-phenthiazin-9-oxyd in Ger, and Tétranitro sulfoxy diphénylamine (TNSD) in French],



$(O_2N)_2C_6H_2\begin{smallmatrix} NH \\ SO \end{smallmatrix}C_6H_2(NO_2)_2$;

mw 395.27, N 17.72%; yel crysts (from Nitrobenz), mp – above 250°, dec 280–82°; heat of combustion at const vol 3339cal/g; heat of formn at const vol 294.5cal/g (Ref 6); was prepd by nitrating phenthiazine, 2,4- or 2,7-dinitrophenthiazine or 2,4,7-trinitrophenthiazine with fuming nitric acid & concd sulfuric acid (Refs 2, 3, 4, 5, 6 & 7). (See also Ref 9)

It was found in the German explosive *Pressling* (Ref 5)

It was patented in France (Ref 4) as a combustible ingredient of expls suitable for use in weapons operating at high temp, or for oil-sounding at great depth

Refs: 1) Beil **27**, 65 & (226) 2) Beil **27**, 68 & (230) 3) K. Pfaff, Reichsamt Wirtschaftsausbau Chem Ber **PB52021**, 1183(1942) & CA **42**, 9045(1948) (Prepn of Tetranitro deriv) 4) État Français, FrP 971644(1951) & CA **46**, 9311(1952) 5) C. Monard et al, MP **34**, 179 (1952) & CA **48**, 9063(1954) (Semi-industrial prepn) 6) K. Toei, NipponKagakuZasshi **76**, 1083–85(1955) & CA **51**, 11913(1957) (Prepn of Tetranitro deriv from thiodiphenylamine) 7) P. Tavernier & M. Lamouroux, MP **39**, 341–43, 354(1957) & CA **54**, 21974(1960) (Prepn, props & thermochemical data) 8) K. Toei, NipponKagakuZasshi **78**, 1379–85(1957) & CA **52**, 14286(1958) (Solubilities of K, Rb & Cs salts of Tetranitro deriv) 9) C. Bodea & M. Raileanu, AcadRepPopulareRomîne, Filiala Cluj. Studii Cercetări Chim **8**, 303 (1957) & CA **54**, 22657(1960) [Prepn of 1,3,7,9-Tetranitrophenothiazine-5-oxide, yel-orn ndls (from Nitrobenz), mp 354–55°, by nitrating phenothiazine with a mixt of nitric acid (d 1.50) & 20% oleum and heating on a w-bath for 20 mins]

Diphenylaminocarboxylic Acid or **N-Phenylanthranilic Acid.** See Anilinobenzoic Acid in Vol 1, pA421-R

Diphenylaminochloroarsine. One of the Chemical Warfare Agents, described as **PM** or *Adamsite* in Vol 2, p C167-R. It is described in CondChemDict (1961), p873-R & p874-L as *Phenarsazine Chloride* (See also Specification MIL-D-11772A)

Di(phenylamino)-ethane. See Bis(anilino)-ethane in Vol 2, p B131-L

Diphenylaminoethanol and Derivatives

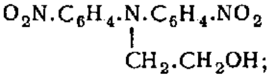
Di(or Bis)(phenyl)amino-N-ethanol,

$C_6H_5.\underset{\textstyle CH_2.CH_2.OH}{\underset{|}{N}}.C_6H_5$, or *Diphenylamino-N-ethylol*

mw 213.27, N 6.57%; liq, bp 183–86° at 5mm press, d 1.121 at 20°, n_D 1.6210 at 20°; was prrpd by heating DPhA with ethylene oxide in an autoclave to 220–40°, with a yield of 60–65% (Refs 2 & 3)

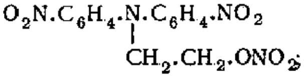
Refs: 1) Beil, not found 2) M.F. Shostakovskii & I.A. Chekulaeva, IzvestAkadNauk SSSR, OtdelKhimNauk **1955**, 913 & CA **50**, 9325(1956) 3) Ibid, BullAcadSci USSR, DivChemSci **1955**, 829–33 (Engl translation) & CA **50**, 11273(1956)

Dinitrodiphenylamino-N-ethanol,



$O_2N.C_6H_4.N(CH_2.CH_2OH).C_6H_4.NO_2$; mw 303.27, N 13.86%; not found in Beil. or in CA thru 1966

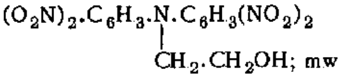
Dinitrodiphenylamino-N-ethanolnitrate,



$O_2N.C_6H_4.N(CH_2.CH_2.ONO_2).C_6H_4.NO_2$; mw 348.27, N 16.09%; not found in the literature thru 1966.

Trinitrodiphenylamino-N-ethanol, $C_{14}H_{12}N_4O_7$, not found in Beil. or in CA thru 1966

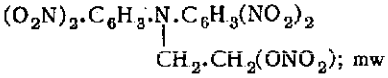
Tetranitrodiphenylamino-N-ethanol or **Bis(Dinitrophenyl)aminoethanol;**



$(O_2N)_2.C_6H_3.N(CH_2.CH_2OH).C_6H_3(NO_2)_2$; mw 393.27, N 17.81%; lemon-yel powd, mp 222°; obtd as a by-product of the action of 2,4-dinitrochlorobenzene & ethanolamine; used to prep 2,4-dinitrophenylaminoethanol which was nitrated to Pentryl (2,4,6-Trinitrophenylnitraminoethyl Nitrate) (Ref 2)

Refs: 1) Beil, not found 2) L.V. Clark, IEC **25**, 1385(1933) 3) Not found in later refs thru 1966

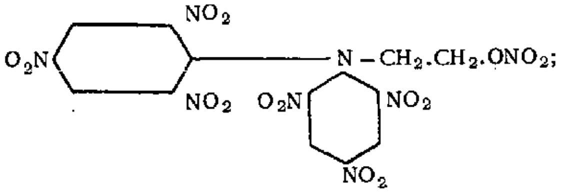
Tetranitrodiphenylamino-N-ethanolnitrate,



$(O_2N)_2.C_6H_3.N(CH_2.CH_2(ONO_2)).C_6H_3(NO_2)_2$; mw 438.27, N 19.18%; not found in the literature thru 1966.

Hexanitrodiphenylamino-N-ethanol, Dipicrylaminoethylol or *2-[Di(or Bis)-2,4,6-trinitrophenylamino]-ethanol,* $C_{14}H_9N_7O_{13}$, not found in Beil. or in CA thru 1966

β(Hexanitrodiphenylamino) Ethyl Nitrate; 2-[Bis(2,4,6-trinitrophenyl) amino-ethanol] Nitrate or **Dipicrylamino-ethylol Nitrate,**



mw 528.26, N 21.21%; crysts, mp 184°, O bal to CO_2 minus 51.5%, d 1.69, d at 3400lb/sq in 1.27. It is prepd from Tetranitrodiphenylaminoethanol, which is a by-product of the action between dinitrochlorobenzene & ethanolamine, by nitrating with mixed acid below 30°

Its expl props are found in Blatt (Ref 3), as detd by Clark (Ref 2)

Brisance by Sand Test – 0.5g crushes 51.1g sand (120% TNT)

Impact Sensitivity by BM App – max fall for 0/5 shots is 35cm with a 2kg wt

Ignition Temperature – ignites at 390–400°

Initiation Sensitivity – complete deton is brought about by 0.5g DDNP (Diazodinitrophenol), 0.16g MF or 0.04g LA

Power by Lead Block – 326cc or 134% TNT

Thermal Stability – International Test at 75° – no discoloration or loss in wt in 48 hrs. Dry storage in the lab at RT for 6 months caused no deterioration

It was recommended as a base chge in compound detonators

Refs: 1) Beil, not found 2) L.V. Clark, IEC **26**, 554(1934) & CA **28**, 3903(1934)
3) Blatt, OSRD **2014**(1944)

Diphenylamino-ethylol. See Diphenylaminoethanol

Diphenyl-(amino-4-guanidovaleric Acid and Derivatives

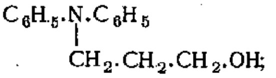
Di(or Bis)phenyl-(amino-4-guanidovaleric Acid or *Diphenylarginine,*
$(C_6H_5)_2{:}N.C({:}NH).NH(CH_2)_3.CH.(NH_2).COOH$, mw 326.39, N 17.17%; may be considered as the parent compd of its hexanitro deriv although not used to prep it:

Di(or Bis)-(2,4,6-Trinitrophenyl)-(amino-4-guanidovaleric Acid) or **Dipicrylarginine,**
$[(O_2N)_3C_6H_2.NH]_2C_6H_{10}N_2O_2$; mw 596.39, N 23.49%, crystalline mass; diffc sol in alc, eth & water. It was prepd in impure state by Hirayama by shaking picryl chloride, $Cl.C_6H_2(NO_2)_3$ (dissolved in toluene) with an equivalent proportion of arginine, $H_2N.C({:}NH).NH(CH_2)_3.CH(NH_2).COOH$

Its expl props were not reported (Refs 3 & 4)
Refs: 1) Beil **4**, 420, (510) & [845](Arginine) 2) Beil, not found [Diphenyl-(amino-4-guanidovaleric acid] 3) Beil **12**, 770 (Dipicryl-arginine) 4) K. Hirayama, ZPhisiolChem **59**, 292(1909); JCS **96**, I, 341(1909) & CA **4**, 222(1910) 5) Not found in later refs thru 1966

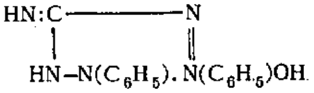
Di(phenylamino)-propane. See Bis(anilino)-propane in Vol 2 of Encycl, p B132-R

Diphenylaminopropanol and Derivatives

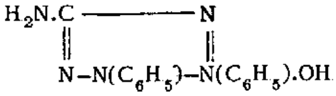
Diphenylaminopropanol,

$C_6H_5.N(CH_2.CH_2.CH_2.OH).C_6H_5$;
mw 227.29, N 6.16%; was prepd in 1887 by Fauconnier (Ref 2) by interaction of aniline, $C_6H_5.NH_2$, and glycerin monochlorohydrin, $Cl.C_6H_2.CHOH.CH_2OH$. We could not find any later info on this compd nor on its nitrated derivs in the literature thru 1966
Refs: 1) Beil – not found 2) Fauconnier, CR **106**, 605(1887)

Diphenylaminotetrazolium Hydroxide and Derivatives

2,3-Diphenyl-5-aminotetrazolium Hydroxide,



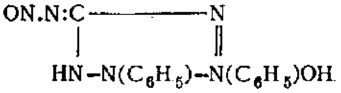
or



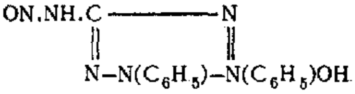
mw 255.27, N 27.44%; the chloride salt is obtd by action of isoamylnitrite & HCl on alcoholic N,N′-diphenyl-C-aminoformazan, $C_6H_5.N:N.C(:NH).NH.NH.C_6H_5$ or $C_6H_5.N:N.C(NH_2):N.NH.C_6H_5$. From the chloride & AgO in aq soln the free base is obtd, yel, unstable & volatilizes (Refs 1 & 2)

The *Chloride salt,* $C_{13}H_{12}N_5.Cl$, ndls (from alc); mp – dec 269°; sol in w & alc; insol in eth, benz, chlf & petr eth
Dichromate salt, $(C_{13}H_{12}N_5)_2Cr_2O_7$, orn-yel ndls (from w); mp – detonates at 189°
Dichromate–HCl salt, $(C_{13}H_{12}N_5)_2Cr_2O_7$+2HCl, orn-yel ndls (from dil HCl), mp – dec at 175°
Chloroplatinate salt, $(C_{13}H_{12}N_5)_2PtCl_6$, pale-yel ndls with brown luster (from dil HCl), mp – dec 249° and
Permanganate salt, $(C_{13}H_{12}N_5)MnO_4$, violet-red ppt, mp – explodes on heating to 106–10°, by friction or by contact with fuming nitric acid; sl sol in hot acet & water (Refs 1 & 2)
Refs: 1) Beil **26**, [247] 2) E. Bamberger et al, Ann **446**, 300(1926) & CA **20**, 1224(1926)

2,3-Diphenyl-5-nitrosamino-tetrazolium Hydroxide,



or



mw 284.27, N 29.57%; orn-yel cubes (from acet or pyridine), mp 177°; v sl sol in acet, eth, benz, chlf & pyridine; was prepd by reaction of Na nitrite on the above chloride salt in dil HCl at 20°. Heating the compd with 70% alc for 3–4 hrs gives an isomeric deriv, $C_{13}H_{10}N_6O$, orn-yel ndls (from alc), mp 154–55°

Its *Chloroaurate salt,* $2(C_{13}H_{10}N_6O)$+$AuCl_3$, orn-yel crysts, mp – sinters at 103° & melts at 119–22°; v sl sol in water; insol in chlf, eth & benz
Hydrochloride salt, $C_{13}H_{10}N_5$+HCl, flakes, mp 168°; sol in alc & dil HCl; insol in benz, petr eth & acet and
Silver Nitrate salt, $2(C_{13}H_{10}N_6O)$+$AgNO_3$, yel cubes, (from w), mp – dec 157–61° deflagrating mildly; insol in chlf & eth; sl sol in hot alc; sol in dil nitric acid (Refs 1 & 2)
Refs: 1) Beil **26**, [248] 2) E. Bamberger et al, Ann **446**, 302(1926) & CA **20**, 1224(1926)

Diphenylarginine. See Diphenyl-(amino-4-guanidovaleric Acid) in this Vol

Di(phenylazo)-amine [called 1.5-Diphenyl-pentazdien-(1.4) or Bisdiazobenzolamid in Ger]
$C_6H_5.N:N.NH.N:N.C_6H_5$; mw 225.24, N 31.09%;

yel prisms, mp – explodes when the dry compd is heated, by impact or by friction; insol in water; sol in alc & eth with decompn; sol in dil KOH; insol in cold dil acids; dec by heating in acids into aniline, phenol & nitrogen; was obtd with other products by pouring a cold saturated aq soln of benzenediazonium nitrate in ammonia under cooling (Refs 1, 2 & 3)
Refs: 1) Beil **16**, 753 2) P. Griess, Ann **137**, 81(1866) 3) Pechmann & Frobenius, Ber **27**, 898–99(1894) & **28**, 171(1895) 4) Not found in later refs thru 1966

Di(phenylazo)-cyclo-pentamethylene-diamine. See N,N'-Bis(phenylazo) -cyclo-2,3,5,6,7-pentamethylene-1,4-diamine in Vol 2, p B152-R
Note: This compd was called erroneously N,N'-Bis(phenylazo)-cyclo-2,3,5,7-pentamethylene-1,4-diamine

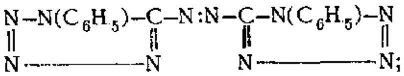
Di(phenylazo)-dihydroxy-naphthalene. See Bis(phenylazo)-dihydroxy-naphthalene in Vol 2, p B152-R

Di(phenylazo)-glycine or **Di(benzenediazo)-glycocoll.** See Bis(phenylazo)-glycine in Vol 2, p B153-L

Di(phenylazo)-naphthol. See Bis(phenylazo)-naphthol in Vol 2, p B153-L

Diphenylazotetrazoles

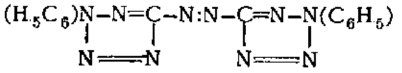
1,1'-Diphenyl-5,5'-azotetrazole,



$$\begin{array}{l} N{-}N(C_6H_5){-}C{-}N{:}N{-}C{-}N(C_6H_5){-}N \\ \| \qquad\qquad\quad \| \qquad\quad \| \qquad\qquad\quad \| \\ N{-}{-}{-}{-}{-}N \qquad N{-}{-}{-}{-}{-}N; \end{array}$$

mw 318.30, N 44.01%, OB to CO_2 minus 165.9%; yel crysts, mp 228°; gives a blood-red soln in concd sulfuric acid; reduction gives a hydrazo deriv, crysts + $2H_2O$, mp 190°(dec); was prepd by passing Cl thru 1-phenyl-5-aminotetrazole in boiling water (Ref 2)
Refs: 1) Beil, not found 2) R. Stollé et al, JPraktChem **134**, 282(1932) & CA **26**, 5565(1932)

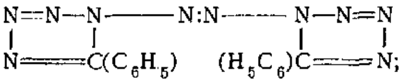
2,2'-Diphenyl-5,5'-azotetrazole or **5,5'-Azo-2,2'-diphenyltetrazole,**



$$\begin{array}{l} (H_5C_6)N{-}N{=}C{-}N{:}N{-}C{=}N{-}N(C_6H_5) \\ \quad | \qquad\quad | \qquad\qquad | \qquad\quad | \\ \quad N{=}{=}N \qquad\quad N{=}{=}N \end{array}$$

mw 318.30, N 44.01%, OB to CO_2 minus 165.9%; yel ndls (from chlf), mp 148°(with brn color), deflagrates on rapid heating in a flame; insol in water; sl sol in eth, alc, benz & glac ac acid; mod sol in hot chlf; evolves gas & becomes red in concd sulfuric acid; was prepd by warming a suspension of 2-phenyl-5-aminotetrazole in w with $Ca(OCl)_2$ on a water bath (Refs 1 & 2)
Refs: 1) Beil **26**, [349] 2) R. Stollé & O. Orth, Ber **58B**, 2104(1925) & CA **20**, 764(1926)

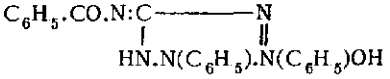
5,5'-Diphenyl-1,1'-azotetrazole (Called 1.1'-Azo-5,5'-diphenyl-tetrazol by Stollé),



$$\begin{array}{l} N{=}N{-}N{-}{-}{-}N{:}N{-}{-}{-}N{-}N{=}N \\ | \qquad | \qquad\qquad\qquad\quad | \qquad | \\ N{=}{=}{=}C(C_6H_5) \quad (H_5C_6)C{=}{=}{=}N; \end{array}$$

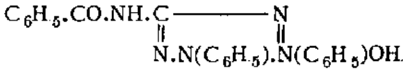
mw 318.30, N 44.01%, OB to CO_2 minus 165.9%; yel crysts; mp – deflagrates ca 141° when heated in the open and detonates on rapid heating in a sealed tube; it also detonates on impact. It can be prepd by treating a soln of 1,1-dichloroamino-5-phenyl-α-tetrazole with a 10% aqueous KI soln, followed by addn of an aqueous soln of a thiosulfate (Ref 2)
Refs: 1) Beil, not found 2) R. Stollé et al, JPraktChem **138**, 5–6(1933) & CA **27**, 4798(1933)

2,3-Diphenyl-5-benzaminotetrazolium Hydroxide,



$$\begin{array}{l} C_6H_5.CO.N{:}C{-}{-}{-}{-}{-}N \\ \qquad\qquad\quad | \qquad\qquad \| \\ \qquad\qquad HN.N(C_6H_5).N(C_6H_5)OH \end{array}$$

or



$$\begin{array}{l} C_6H_5.CO.NH.C{-}{-}{-}{-}{-}N \\ \qquad\qquad\qquad \| \qquad\qquad \| \\ \qquad\qquad\qquad N.N(C_6H_5).N(C_6H_5)OH \end{array}$$

mw 359.38, N 19.49%; golden-yel crysts (from alc), mp – deflgr at 200°; heating with alc KOH or NaOH for several mins is w/o effect; was prepd by treating 2,3-diphenyl-5-aminotetrazolium chloride with benzoyl chloride in dil NaOH at 20°(Refs 1 & 2)

The *Chloride salt,* $C_{20}H_{15}N_5O{+}HCl$ or $C_{20}H_{16}N_5O.Cl$, ndls mp 280.5°(dec), turns yel in sunlight; v sl sol in hot w
Chloroplatinate salt, $2(C_{20}H_{15}N_5O){+}H_2PtCl_6$, flakes, mp 198°(dec); v sl sol in w & alc

Dichromate salt, $2(C_{20}H_{15}N_5O)+H_2Cr_2O_7$ or $(C_{20}H_{16}N_5O)_2Cr_2O_7$, citron-yel powd, mp 170.5°(dec), turns green in sunlight; decomp in hot water

Permanganate salt, $C_{20}H_{15}N_5O+HMnO_4$ or $C_{20}H_{16}N_5O.MnO_4$, violet crysts, mp – explodes on heating to 144° or by friction; sl decomposes in hot water

Refs: 1) Beil **26**, [248] 2) E. Bamberger et al, Ann **446**, 305(1926) & CA **20**, 1224(1926)

2,3-Diphenylbenzopyrylium Perchlorate. See under Benzopyrylium Compounds in Vol 2, p B79-R

Di(p-phenylbenzoyl)-furoxan,

mw 464.44, N 6.28%; wh crysts (from ether, then from benz), mp 183° with decompn to a brn liq; prepd in 60% yield when nitric acid in water was added to a soln of p-phenylacetophenone in glac acet acid and the mixt warmed to 60°, Na nitrite was added and the soln allowed to stand for 30 mins at 60° while N oxides developed. The yellow crysts which formed were filtered & crystallized (Refs 2 & 3)

No expl props or expl derivs were reported

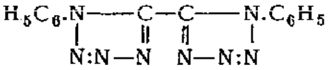
Refs: 1) Beil, not found 2) ADL, Synthesis HE's, 3rd Rept (1953), p384 3) ADL, Synthesis HE's, 4th Rept (1956), p58

p,p'-Di(or Bis)phenylbenzoyl Peroxide, $C_6H_5.C_6H_4.CO.O_2.CO.C_6H_4.C_6H_5$; mw 394.40, O 16.23%; crysts (from toluene), mp 171°(with very violent decompn); canbe prepd by adding (while shaking) to an acetonic soln of p-phenylbenzoyl chloride a slight excess of alkaline 10% hydrogen peroxide

Refs: 1) Beil, not found 2) H. Wieland & G. Rasuwajew, Ann **480**, 164–65(1930) & CA **24**, 4013(1930)

Diphenyl-bis(4,4'-diazoniumhydroxide). See Biphenyl-bis(4,4'-diazoniumhydroxide) in Vol 2, p B125-L

1,1'-Diphenyl-5,5-bistetrazole,



$$\begin{array}{l} H_5C_6.N—C—C—N.C_6H_5 \\ \quad\quad | \quad\;\; \| \quad \| \quad\;\; | \\ \quad\; N{:}N—N \quad N—N{:}N \end{array}$$

mw 290.28, N 38.60%; ndls (from glac acet ac), mp 212°; sol in benz & glac acet ac; sl sol in hot alc; prepd by action of NaN_3 in boiling alc on oxalic acid-bis(phenylimidechloride) (Refs 1 & 2)

Refs: 1) Beil. **26**, [363] 2) R. Stollé, Ber **55**, 1296(1922)

Diphenylbromoarsine, $(C_6H_5)_2AsBr$; wh crysts, mp 54–56°; a very irritant substance (Refs 1 & 2), similar in its properties to the Chemical Warfare Agent Diphenylchloroarsine described as **DA** in Vol 2 of Encycl, p C166

Refs: 1) Beil **16**, 845 & [431] 2) Cond-ChemDict (1961), 406-L

Diphenylcarbamide (or Diphenylcarbamyl) Complexes, proposed by Lévy (See Ref) as suitable stabilizers and gelatinizers in smokeless propellants, were prepd by interaction of diphenylcarbamylchloride (DPhCCl), $(C_6H_5)_2N.CO.Cl$, (described in Beil **12**, 428) with organic bases such as allylamine, $C_3H_5NH_2$. Preparation was conducted by agitating the reactants in alcoholic medium under reflux, in the presence of dry, pulverized Na carbonate (which served to neutralize HCl formed during reactions)

Following complexes were prepd and tested by Lévy:

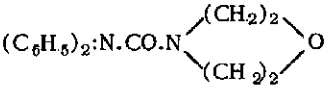
Bis-(diphenylcarbamyl)-ethylenediamine, $(C_6H_5)_2{:}N.CO.NH.CH_2.CH_2.NH.CO.N{:}(C_6H_5)_2$; mw 450.52, N 12.44%; wh crysts, mp 184°; sol in alc; was prepd by refluxing for 6 hrs, with agitation, DPhCCl with ethylenediamine in alc contg Na carbonate

Diphenylcarbamylallylamine, $(C_6H_5)_2{:}N.CO.NH(CH_2.CH{:}CH_2)$; mw 252.30, N 11.10%; wh crysts, mp 82°, sol in ethanol, methanol & acet; was prepd by refluxing for 6 hrs, with agitation, DPhCCl with allylamine in alc contg Na carbonate

Diphenylcarbamyldiethanolamine, $(C_6H_5)_2$:N.CO.N:$(CH_2.CH_2.OH)_2$; mw 300.35, N 9.33%; wh crysts; mp 124.5–125°; sol in alc; was prepd by refluxing for 6 hrs, with agitation, DPhCCl with diethanolamine in alc contg Na carbonate

Diphenylcarbamylmonoethanolamine, $(C_6H_5)_2$:N.CO.NH.$(CH_2.CH_2.OH)$; mw 256.29, N 10.93%; wh crysts, mp 117.5°; sol in ethanol, methanol & acet; was prepd by refluxing for 6 hrs, with agitation, DPhCCl with monoethanolamine in alc contg Na carbonate

Diphenylcarbamylmorpholine,



$$(C_6H_5)_2\text{:N.CO.N}\left\langle\begin{matrix}(CH_2)_2\\(CH_2)_2\end{matrix}\right\rangle O$$

mw 282.33, N 9.92%; wh crysts, mp 108.5°; sol in ethanol, methanol & acet; was prepd by refluxing for 3 hrs, with agitation, DPhCCl with morpholine in alc contg Na carbonate

Diphenylcarbamyl-α-naphthylamine, $(C_6H_5)_2$:N.CO.NH$(C_{10}H_7)$; mw 338.39, N 8.28%; wh crysts with sl rose tinge; mp 185–185.5°; sl sol in ethanol & methanol; was prepd by refluxing for 6 hrs, with agitation, DPhCCl & α-naphthylamine in alc contg Na carbonate

Diphenylcarbamyl-β-naphthylamine, $(C_6H_5)_2$:N.CO.NH$(C_{10}H_7)$; mw 338.39, N 8.28%; wh crysts, mp 182°; sl sol in cold alc; was prepd by refluxing for 6 hrs, with agitation, DPhCCl with β-naphthylamine in alc contg Na carbonate

Diphenylcarbamylpiperidine, $(C_6H_5)_2\text{:}\underline{\text{N.CO.N.CH}_2\text{.CH}_2\text{.CH}_2\text{.CH}_2\text{.CH}_2}$; mw 280.36, N 9.99%; wh crysts, mp 123.5°; sol in ethanol, methanol & acet; was prepd by refluxing for 3 hrs, with agitation, DPhCCl with piperidine (cyclopentamethyleneamine) in alc, contg Na carbonate

Ref: R. Lévy, MP **32**, 309–12(1950)

Diphenylcarbamoyl Azide. Same as Diphenylcarbamyl Azide

Diphenylcarbamyl Azide (Diphenylcarbamidsäureazid in Ger), $(C_6H_5)_2N.CO.N_3$; mw 238.24, N 23.52%; ndls (from alc); mp 85° (decomp); v sl sol in w; v sol in alc, eth, chlf & benz. It can be prepd by interaction of diphenylcarbamylchloride, $(C_6H_5)_2N.CO.Cl$ with Na azide in boiling ethanol

Explodes mildly when heated in a flame

Refs: 1) Beil **12**, (258), [242] 2) R. Stollé, Ber **57**, 1064(1924) 3) F.L. Scott, Chem & Ind **1954**, 959 & CA **49**, 15(1955) (Carbamoyl Azide) 4) F.L. Scott & M.T. Scott, JACS **79**, 6077(1957) & CA **52**, 6228(1958)

Diphenylcarbazide or 1,5-Diphenylcarbohydrazide, $(C_6H_5.NH.NH)_2CO$; mw 242.27, N 23.13%; wh crysts, decomposing in light; mp 173–75°; insol in w & eth; sol in hot alc; can be prepd by interaction of phenylhydrazine, $C_6H_5.NH.C_6H_5$ and urea, $H_2N.CO.NH_2$

Used in analytical chemistry for determination of Cu and other metals and is probably suitable for use in proplnts

Refs: 1) Beil. **15**, 292, (72) & [107] 2) CondChemDict (1961), 406-L

Diphenylcarbazone and Derivatives

s-Diphenylcarbazone, $C_6H_5.N{:}N.CO.NH.NH.C_6H_5$; mw 240.26, N 23.32%; orn-red ndls, mp ca 127° (decomp); insol in w; sol in alc, eth, benz & chlf; can be prepd by action of phosgene on an ethereal soln of 1,5-diphenylhydrazine (Refs 1 & 2)

It has been used by the US Armed Forces as an analytical reagent and the requirements and tests were covered by the Spec MIL-D-11364 (Cml C), 22 Aug 1951

Now, accdg to Notice 1 to this spec dated 14 Nov 1967, the above spec is cancelled and future procurement of sym-Diphenylcarbazone, Reagent should be made under Federal Specification O-C-265A, "Chemicals, Analytical; General Specification For"

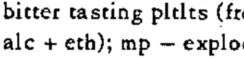
Assuming that requirements and tests of O-C-265A are the same as used before 1967, we are using here the Spec MIL-D-11364 (because O-C-265A is not at our disposal)

1) *Form* – crystalline, when examined visually
2) *Color* – orange, when examined visually
3) *Sensitivity.* The material shall pass the following test:

Dissolve 0.270g of mercuric chloride in distd water contained in a 1000ml volumetric flask and dissolve 0.100g of sample in 5ml of 95% ethanol contained in a small flask. To 20ml of mercuric chloride soln in a small beaker add 0.1ml of alcoholic sample soln and observe if any violet color is produced. If no color is produced the sample must be rejected

Refs: 1) Beil. **16**, 24, (222) & [9] 2) P. Krumholtz & E. Krumholtz, Monatsh **70**, 431 (1937) & CA **32**, 126(1938) 3) USMilitary-Specification MIL-D-11364 (Cml C), 22 Aug 1951 and Notice 1, 14 Nov 1967 4) Federal Specification O-C-265A (21 May 1969) (Not at our disposal)

2,3-Diphenylcarbodiazone {called C-Hydroxydiphenyltetrazolium Betaine or 1,2-Diphenyl-5-oxa-1,2,3,6-tetrazabicyclo [2.1.1]-1(6),3-hexadiene in CA Coll Formula Index **14–40** (1920–46), p718}(called Diphenylcarbodiazon or Anhydrid of 2.3-Diphenyl-5-oxytetrazoliumhydroxid in Ger)



$$\begin{array}{lll}\bar{O}.C & \text{————} & N \\ \quad \| & & \| \\ \quad N.N(C_6H_5) & . & \overset{+}{N}.C_6H_5\end{array}$$

;mw 238.24, N 23.52%; bitter tasting pltlts (from alc) or ndls (from alc + eth); mp – explodes at 178° or by impact; sol in MeOH, alc, hot w & concd sulfuric acid; insol in eth & benz. Can be prepd by oxidizing 1,5-diphenylcarbohydrazide with isoamylnitrite in glac acet ac + acet on a water bath, or with $KMnO_4$ in acet (Refs 1 & 2)

It forms salts, some of which are expl:

Chloroplatinate, $2C_{13}H_{10}N_4O+H_2PtCl_6$, flesh-colored powd, mp 196°(dec)

Dibromide, $C_{13}H_{10}N_4OBr_2$, yel crysts (from alc + eth), mp – darkens at 60° & decomp; sol in alc; insol in eth & w

Perchlorate, $C_{13}H_{10}N_4O+HClO_4$, col crysts, mp – explodes at 194°

Picrate, $2C_{13}H_{10}N_4O+C_6H_3N_3O_7$, golden-yel pltlts (from alc), mp 169°(dec); sol in hot w (Refs 1 & 2)

Refs: 1) Beil **26**, 408, (124) & [247] 2) E. Bamberger et al, Ann **446**, 281, 283, 287(1926) & CA **20**, 1223(1926)

Diphenylcarbohydrazide. See Diphenylcarbazid, p D1446-R in this Vol

Diphenylcarbonate or **Diphenylester of Carbonic Acid,** $(C_6H_5O)_2CO$; mw 214.21; wh ndls (from alc); sp gr 1.272 at 14°; mp 78–80°; bp 302–06°; insol in w; sol in acet, benz, eth, glac AcOH, hot alc and other org solvs; can be prepd by reacting phenol & carbonyldifluoride in benz & pyridine, or from phenol & phosgene in the presence of $AlCl_3$ at 170–90°(Refs 1 & 2)

Used as a plasticizer and solvent for NC (Ref 3)

Refs: 1) Beil **6**, 158, (88), [156] & {607} 2) H.J. Emeléus & J.F. Wood, JChemSoc **1948**, 2183, 2186 3) CondChemDict (1961), 406-L

Di(N-phenylcarboxamide)-acetylene and Derivatives

Di(N-phenylcarboxamide)-acetylene, listed as *Bis(carboxanilide)-acetylene* in Vol 2, p B138-L. Its formula and formula of its hexanitro- derivative were given but prepn and props were not described because the ADL Report serving as a source of information was classified at the time of publication of Vol 2 of Encycl

As the report is now declassified, we are describing these compds here:

Di(N-phenylcarboxamide)-acetylene, $C_6H_5.NH.CO.C \vdots C.CO.NH.C_6H_5$; mw 264.27, N 10.60%; pink-wh crysts (from $MeOH+H_2O$), mp 194–96°; was obtd when bromofumaranilide was added to a soln of KOH in MeOH and the mixt refluxed for 2 hrs & hot water added to ppt the solid on cooling (Ref 2)

Dipicrylcarboxamide-acetylene or **2,4,6,2',4',6'-Hexanitro-di(N-phenylcarboxamide)-acetylene,** $(O_2N)_3C_6H_2.NH.CO.C \vdots C.CO.NH.C_6H_2(NO_2)_3$, mw 534.27, N 20.97%; wh crysts, mp 272–80°; was prepd by nitrating the parent compd with concd nitric acid & concd sulfuric acid in the cold, and pouring the reaction mixt into ice water and collecting the solid on a funnel. The product was boiled with methyl ethyl ketone and filtered while hot to remove incompletely nitrated material (Ref 2)

No expl props of the compd were reported

Refs: 1) Beil, not found (Parent) 2) Beil, not found (Hexanitro) 3) ADL, Synthesis HE's, 2nd Rept (1951), 153–54 4) Not found in later refs thru 1966

Diphenylchloroarsine. One of the Chemical Warfare Agents, designated as **DA** or **PD.** See Vol 2, p C167-L and in CondChemDict (1961), p406-L

Diphenylcyanoarsine. One of the Chemical Warfare Agents, designated as **DC** and described in Vol 2 of Encycl, p C167-R and in CondChemDict (1961), p406-R

Diphenylcyclopentane and Derivatives

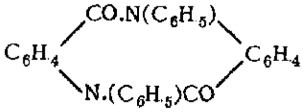
1,2-Diphenylcyclopentane, $(C_6H_5)\underbrace{CH.CH_2.CH_2.CH_2.CH}.(C_6H_5)$; mw 222.31. It exists as cis-, crysts (from petr eth), mp 47° and trans-, pltlts (from MeOH) or ndls (from acetic acid; can be prepd from (±)cis- or (±)trans 1,2-diphenylcyclopentanone-(3) or -(4) by heating with Zn amalgam & aq HCl in toluene (Refs 1 & 4)

x,x,x,x-Tetranitro-1,2-diphenylcyclopentane, $C_{17}H_{14}(NO_2)_4$; mw 402.31, N 13.93%; pltlts (from glac acet ac) or ndls (nitric acid), mp 217° (dec); was prepd by treating 1,2-dicyclopentane of mp 46° with ice-cold fuming nitric acid (Refs 2 & 3)

Refs: 1) Beil **5**, 650, (316) & {2025} 2) Beil **5**, (316) (Tetranitro) 3) H. von Liebig, Ann **405**, 208 (1914) 4) H.A. Weidlich, Ber **71**, 1601, 1603 (1938) & CA **32**, 8375 (1938) 5) Not found in later refs thru 1966

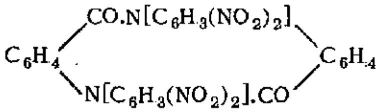
Diphenyldianthranilide and Derivatives

Diphenyldianthranilide,



mw 390.42, N 7.17%; may be considered as the parent compd of its nitrated derivs, although not used to prep them:

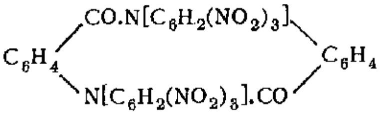
2,4,2',4'-Tetranitrodiphenyldianthranilide or **N,N'-Di(2,4-dinitrophenyl)-dianthranilide,**



mw 570.42, N 14.73%; ndls (from acet+alc), mp – dec; readily sol in acet, chlf, benz, acet ac, & phenol; sl sol in alc, eth, CS_2 & CCl_4; was prepd from reaction of 2',4'-dinitrodiphenylamine-2-carbonyl chloride & pyridine (Refs 2 & 3)

No expl props were reported

2,4,6,2',4',6'-Hexanitrodiphenyldianthranilide or **N,N'-Dipicryldianthranilide,**



mw 660.42, N 16.97%; citron-yel ndls (from phenol+acet ac), mp >200° (dec); sol in hot phenol & boiling ethylene bromide; v sl sol in most other solvs; was prepd by reaction of pyridine & N-picryl-anthranilic chloride at 100° (Refs 2 & 3)

No expl props were reported

Refs: 1) Beil **24**, not found (Parent) 2) Beil **24**, 409 (Hexanitro) 3) G. Schroeter & O. Eisleb, Ann **367**, 116, 120 (1909) & CA **3**, 2976 (1909)

4,4'-Diphenyl-dizaoaminobenzene or **4-Diazo-aminobiphenyl.** See I, III-Bis(p-biphenyl)-triazine in Vol 2, p B136-L

Diphenyldiazomethane. See Biphenyldiazomethane in Vol 2, p B125-R

Diphenyldiazonium Perchlorates
Davis & Huntress (Ref) patented the following method of prepn of diazonium perchlorates of general formula $R.N_2.ClO_4$, in which R is an organic radical, such as diphenyl-, nitrodiphenyl-, etc
Example: Diazotize 2,2′-dinitrobenzidine, $H_2N.C_6H_3(NO_2)-C_6H_3(NO_2).NH_2$, also known as 2,2′-dinitrodiaminodiphenyl, by adding Na nitrite to its soln in cold HCl. Filter, if necessary, and add to the filtrate an equivalent amount of Amm perchlorate dissolved in w. Filter off the precipitated orange-colored substance, wash it with w, followed by alc & ether and then dry in the air. The dried material is much more sensitive than when wet. It explodes very violently at the slightest shock, or when heated
Ref: T.L. Davis & E.H. Huntress, USP 1828960(1931)

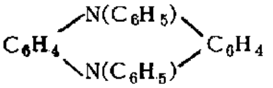
Diphenyldibutylurea. See Centralite, Butyl, under CENTRALITES in Vol 2, p B140-L

Diphenyldicarboxylic Acid or **Diphenic Acid.** See Biphenyldicarboxylic Acid in Vol 2, p B125-R

Diphenyldiethylurea. See Centralite 1 in Vol 2, p B127-L. It has been used as a stabilizer in some US proplnts such as Units No 548 & 549 described in conf Proplnt Manual **SPIA M/2** (1962)

9,10-Diphenyl-9,10-dihydrophenazine and Derivatives

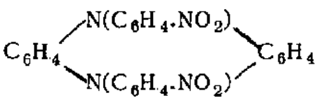
9,10-Diphenyl-9,10-dihydrophenazine



$$C_6H_4\begin{matrix}N(C_6H_5)\\N(C_6H_5)\end{matrix}C_6H_4$$

mw 334.40, N 8.38%; ndls (from benz + alc), mp 172–75°; sol in chlf; sl sol in cold benz; v sl sol in eth, alc & acet ac; was obtd & separated from other products when tetraphenylhydrazine was boiled with toluene for 1/2 hr in an atm of CO_2 (Refs 1 & 2)
Refs: 1) Beil **23**, (54) 2) H. Wieland, Ann **381**, 206, 212(1911) & CA **5**, 3286(1911)

9,10-Di(4-nitrophenyl)-9,10-dihydrophenazine,



$$C_6H_4\begin{matrix}N(C_6H_4.NO_2)\\N(C_6H_4.NO_2)\end{matrix}C_6H_4$$

mw 424.40, N 13.20%; reddish-brn crysts, mp – sinters at 154° & melts ca 183°; sol in chlf & in sulfuric acid giving a faint grn color changing to violet on addn of CrO_3, in alc KOH with faint red color deepening on heating; sl sol in benz & acet ac; v sl sol in alc & eth; was obtd with 4-nitro DPhA on heating of N-nitroso-4-nitrodiphenylamine with xylene (Refs 1 & 2)
Refs: 1) Beil **23**, (54) 2) H. Wieland & H. Lecher, Ann **392**, 165(1912) & CA **7**, 597(1913)

Tetranitro, $C_{24}H_{14}N_6O_8$, and *Hexanitro*, $C_{24}H_{12}N_8O_{12}$, derivs of 9,10-Diphenyl-9,10-dihydrophenazine were not found in the literature thru 1966

Diphenyldiimide. One of the names for Azobenzene described in Vol 1, p A646-R and in CondChemDict (1961), p 120-R

Diphenyldiketone; Diphenylglyoxal or **Dibenzoyl.** See Benzil in Vol 2, p B64

Diphenyldimethylurea. See Centralite 2 in Vol 2, p B137-L and ADL Rept, Pt 1 (1947), p 111, where it is stated that power & brisance of Hexanitrodimethylurea are lower than those values of Picric Acid

Diphenyleneimine. Same as Carbazole described in Vol 2, pp C45 to C50

4,4′-Diphenyl-diphenylyl-amine. See Bis-(p-biphenyl)-amine in Vol 2, p B136-R

Diphenyl-dipicryl-ethylene-dihydrazine. See α,β-Bis(α-phenyl-β-picryl-hydrazine)-ethane in Vol 2, p B154-R

Diphenyl-diquinone-3,4,3′,4′-bis-diazide-(4,4′). One of the names for compd listed as 4,4′-Bis(diazo)-3,3′-dioxy-biphenyl in Vol 2, p B139-L

Diphenydisulfide and Derivatives

Diphenyldisulfide or *Bis(phenyl)-disulfide*, $C_6H_5.S_2.C_6H_5$; mw 218.34; ndls (from alc), mp 61–62°, bp 310°; insol in w; sl sol in eth, benz & CS_2; sol in alc & in hot concd sulfuric acid with a red-violet color; can be prepd by heating thiophenol in ammoniacal aq soln & passing thru of air, and by other methods (Ref)

Ref: Beil **6**, 323, (148), [294] & {1027}

Mononitrodiphenyldisulfide,

$C_6H_5.S_2.C_6H_4.NO_2$; mw 253.34, N 5.53%. Two isomers are known:

2-Nitrodiphenyldisulfide, yel crysts (from alc & petr), mp 55°(Ref 1)

4-Nitrodiphenyldisulfide, yel ndls (from hexane), mp 58–58.5°(Ref 2)

Refs: 1) Beil **6**, [306] & {1061} 2) Beil **6**, {1076}

Dinitrodiphenyldisulfide,

$O_2N.C_6H_4.S_2.C_6H_4.NO_2$; mw 308.34, N 9.09%.

2,2′-Dinitrodiphenyldisulfide, yel ndls (from benz or glac acet ac), mp 197–98°(Ref 1)

3,3′-Dinitrodiphenyldisulfide, yel ndls (from alc), mp 81–84°(Ref 2)

4,4′-Dinitrodiphenyldisulfide, crysts, mp 181–83°(Ref 3)

2,4′-Dinitrodiphenyldisulfide, crysts (from eth + chlf), mp 159.2–159.6°(Ref 4)

2,4-Dinitrodiphenyldisulfide, $(O_2N)_2C_6H_3.S_2.C_6H_5$, yel ndls (from alc), mp 86–87°(Ref 5)

Other props & methods of prepn are found in Refs

Refs: 1) Beil **6**, 338, (157), [307] & {1061} 2) Beil **6**, 339 & {1067} 3) Beil **6**, 340, (160), [312] & {1077} 4) Beil **6**, {1077} 5) Beil **6**, {1101}

Trinitrodiphenyldisulfide,

$(O_2N)_2C_6H_3.S_2.C_6H_4.NO_2$; mw 353.34, N 11.89%

2,4,2′-Trinitrodiphenyldisulfide, crysts, mp 175–76°; was prepd by reaction of 2,4-dinitrothiophenol & 2-nitrobenzenesulfenyl chloride in eth (Refs 1 & 2)

2,4,4′-Trinitrodiphenyldisulfide, crysts, mp 178°; was prepd from 2,4-dinitrothiophenol & 4-nitrobenzene-sulfenyl chloride (Ref 3)

Refs: 1) Beil **6**, {1101} 2) V.O. Lukashevich & M.M. Sergeeva, DoklAkadNauk **67**, 1041 (1949) & CA **44**, 1921 (1950); ZhurObshchKhim **19**, 1493, 1504(1949) & CA **44**, 3452(1950) 3) A.J. Parker & N. Kharasch, JACS **82**, 3075 (1960) & CA **55**, 3497(1961)

Tetranitrodiphenyldisulfide,

$(O_2N)_2C_6H_3.S_2.C_6H_3(NO_2)_2$; mw 398.33, N 14.06%

2,4,2′,4′-Tetranitrodiphenyldisulfide, yel ndls, mp – explodes at 280°; sol in nitrobenz, pyridine & hot aniline; can be prepd by reacting 4-chloro-1,3-dinitrobenzene & Na_2S_2 in alc and by many other methods (Refs 1 & 4)

3,5,3′,5′-Tetranitrodiphenyldisulfide, yel crysts (from benzene + benzine or benz + alc), mp 175–177.5°(dec); sol in chlf & acet ac; sl sol in eth, benzine & alc; was prepd by heating 3,5-dinitrobenzene sulfonyl chloride with PBr_3 at 100–10° and by other methods (Refs 2 & 3)

Refs: 1) Beil **6**, 344, (163), [316] & {1101} 2) Beil **6**, {1102} 3) A.H. Kohlhase, JACS **54**, 2441, 2446(1932) 4) V.O. Lukashevich & M.M. Sergeeva, DoklAkadNauk **67**, 1493, 1503(194

Pentanitrodiphenyldisulfide, $(O_2N)_3C_6H_2.S_2.C_6H_3(NO_2)_2$, not found in the literature thru 1966

Hexanitrodiphenyldisulfide; Di(2,4,6-Trinitrophenyl)-disulfide or **Dipicryldisulfide,**

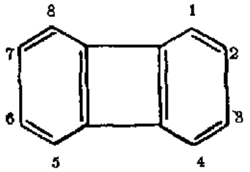
$(O_2N)_3C_6H_2.S_2.C_6H_2(NO_2)_3$; mw 488.33, N 17.21%; dk-red solid, no props are reported but the compd is definitely expl; was prepd, in addn to Picrylsulfide, by reaction of picryl chloride with thiourea, $CS_2(NH_2)_2$, in the presence of Ca carbonate in alc (Refs 1 & 2)
Refs: 1) Beil **6**, [317] 2) M. Giua, AttiCongressoNazChimInd **1924**, 373 & CA **19**, 1561(1925) 3) Not found in later refs thru 1966

1,3-Diphenyl-4-disulfonmethylene-tetrazene-(1) or Benzenediazophenyl-hydrazonemethane-disulfonic acid,
$C_6H_5.N:N.N(C_6H_5).N:C(SO_3H)_2$. It is known in the form of its *Dipotassium Salt,* $K_2C_{13}H_{10}N_4O_6S_2$, mw 460.56, N 12.17%, prepd by von Pechmann in 1896. It consists of orn-yel ndls, deflagrating at 180° when confined; sol in w and in concd sulfuric acid. It can be prepd by pouring a well-cooled soln of benzenediazonium chloride, $C_6H_5.N_2.Cl$, in aqueous KOH into an aq soln of the tripotassium salt of sulfohydrazonemethylenedisulfonic acid, $(HO_3S)_2C:N.NH.SO_3H$
Refs: 1) Beil **16**, 747 2) H. von Pechmann, Ber **29**, 2165(1896)

1,4-Diphenyl-3,5-endoanilino-1,2,4-triazoline. See 1,4-Diphenyl-3,5-phenylimino-1,2,4-triazole in this Vol

Diphenylene and Derivatives

o-Diphenylene or *Biphenylene* (called Biphenylen or Cyclobutadibenzol in Ger),



mw 152.18; lt-yel crysts (from alc), mp 110–12°; bp 105° at 4mm press; was prepd by distilling 2,2′-dibromobiphenyl or dibenzoiodonium iodide with cuprous iodide (Ref 2), and also by the action of cupric chloride on the Grignard reagent prepd from 2,2′-dibromodiphenyl (Ref 3). It forms a *Picrate salt,* $C_{12}H_8.C_6H_3N_3O_7$, red ndls (from alc), mp 122° (Ref 1)
Refs: 1) Beil **5**, (298), [530] & {1935}
2) W.C. Lothrop, JACS **63**, 1187, 1190(1941)
3) W.S. Rapson et al, JChemSoc **1943**, 326

Dinitro-o-diphenylene, $C_{12}H_6(NO_2)_2$; mw 242,18, N 11.57%. Two isomers are reported in the literature:
2,6-Dinitro-o-diphenylene, deep-yel ndls (from alc), mp – sublimes w/o melting >260°; was prepd by nitrating diphenylene, and by heating either 4,5′-dinitrodiphenylene-2,2′-iodonium iodide or 2,2′-di-iodo-4,5′-dinitrodiphenyl with cuprous oxide at 350° (Ref 3)
X,X-Dinitro-o-diphenylene, ndls (from alc), mp 204°; insol in eth, benz & CS_2; sl sol in alc, glac acet ac & acet; was obtd in small quantity by heating o-diphenylene with nitric acid (d 1.2) in a tube at 150° (Refs 1 & 2)
Refs: 1) Beil **5**, (299) 2) J.J. Fox et al, JChemSoc **103**, 40(1913) & CA **7**, 1490(1913)
3) Not found in later refs thru 1966

X,X,X,X-Tetranitro-o-diphenylene,
$C_{12}H_4(NO_2)_4$; mw 332.18, N 16.87%; yel ndls (from alc), mp 223°; sl sol in alc & glac acet ac; insol in other org solvs; was obtd by nitrating o-diphenylene with a freezing mixt of concd sulfuric acid and nitric acid (d 1.5) (Refs 1 & 2)

No expl props were reported
Refs: 1) Beil **5**, (299) 2) J.J. Fox et al, JChemSoc **103**, 39(1913) & CA **7**, 1490(1913)
3) Not found in later refs thru 1966

Hexanitro-o-diphenylene, $C_{12}H_2(NO_2)_6$, not found in the literature thru 1966

Di(phenylene)-azo-trinitromethane. See p,p′-Bi(phenylene-azo-trinitromethane) in Vol 2, p B126-L

Diphenyleneoxide. See Dibenzofuran in this Vol, p D1183-L

Diphenylethanes, $C_{14}H_{14}$, mw 182.25. Two isomeric forms exist:
1,2-(or sym)Diphenylethane. It is listed as *Bibenzyl* in Vol 2, p B110-L, but without giving its properties such as: wh crysts, sp gr 0.978 at 50°/50°, mp 52.0°, bp 284°; insol in w; sol in alc, chlf, eth & CCl_4. It can be prepd by treating benzyl chloride with metallic Na or by the action of benzylchloride on benzylmagnesium chloride. A third method is described in Vol 2, p B110-L (Refs 1 & 7)

Its nitro- derivatives, including explosive tetra-, penta- and hexanitro compds, are described in Vol 2, pp B110-R & B111-L
1,1-(or unsym)Diphenylethane, $(C_6H_5)_2CH.CH_3$; col oil, sp gr 1.004 at 20°, fr p −21.5°, bp 272°; insol in w; sol in alc, eth, chlf & CCl_4. It can be prepd by the action of acetyaldehyde upon benzene in the presence of concd sulfuric acid; used as a solvent for NC (Refs 2 & 7)
1,2-Dinitro-1,1-diphenylethane, $(C_6H_5)_2C(NO_2).CH_2NO_2$; mw 272.25, N 10.29%; ndls (from petr eth), mp 68°; sol in common org solvs; was prepd, with the greatest precaution to exclude moisture, by reaction of NO_2 in petr eth & α,α-diphenylethylene in the cold (Refs 3 & 4)
1,1-Di(p-nitrophenyl)-ethane, $(p\text{-}O_2N.C_6H_4)_2CH.CH_3$; crysts, mp 112–18°; was prepd by adding 1,1-diphenylethane dropwise to nitric acid (d 1.5) at −15°, followed by immediate quenching on ice and rubbing the crude product with acetone (Refs 5, 6 & 7)
1,1-Di(2,4-dinitrophenyl)-ethane, $[C_6H_3(NO_2)_2\text{-}2,4]_2CH.CH_3$; mw 362.25, N 15.47%; crysts (from acet ac), mp 157°; was prepd by addg 1,1-diphenylethane to nitric acid (d 1.35) & concd sulfuric acid at 30–35° (Ref 6). No expl props were reported

Pentanitrodiphenylethane, $C_{14}H_9N_5O_{10}$, not found in Beil or in CA thru 1966

Hexanitrodiphenylethane, $C_{14}H_8N_6O_{12}$, not found in Beil or in CA thru 1966
Refs: 1) Beil **5**, 598, (280), [506] & {1809} (Bibenzyl) 2) Beil **5**, 605, (285), [509] & {1809} (Unsym Compd) 3) Beil **5**, [511] 4) H. Wieland & F. Rahn, Ber **54**, 1773(1921) & CA **16**, 903(1922) 5) G. Benoit & F. Eliopoulo, BullSocChimFranc **1951**, 890 & CA **47**, 2148(1953) 6) I.P. Tsukervanik & M.D. Sokol'nikova, ZhurObshKhim **24**, 1435 (1954) & CA **49**, 10244(1955) 7) H.H. Szmant & J.F. Deffner, JACS **81**, 958(1959) & CA **54**, 7641(1960) 8) CondChemDict (1961), 407-L (Bibenzyl) ; 407-L (Unsym compd)

Diphenyl-ethanol. See Diphenyl-ethylol in this Vol

Diphenyl-ether and Derivatives

Diphenylether, Phenylether, Diphenyloxide or *Bis(phenyl)-ether,* $C_6H_5.O.C_6H_5$; mw 170.20, col crysts, sp gr 1.073 at 20°, mp 20.9°, bp 258.3°; v sl sol in w; sol in alc & eth; can be prepd by heating under pressure bromobenzene with Na phenate; used in org synthesis as a heat transfer medium, and in manuf of soaps
Refs: 1) Beil **6**, 146, (84), [148] & {562} 2) CondChemDict (1961), 408-L (Diphenyloxide)

Following nitrated products are listed in the literature:
Mononitrodiphenylethers, $C_6H_5.O.C_6H_4.NO_2$; mw 215.20, N 6.51%. Three isomers, 2-, 3- & 4-, none of them expl, are described in Beil **6**, 218, 224, 232, (114, 119), [210, 214, 229] & {801, 809, 821}
Dinitrodiphenylethers, $C_{12}H_8N_2O_5$; mw 260.20, N 10.77%. Six isomers, 2,2'- 2,4'-, 2,4-, 2,6-, 3,4- & 4,4'-, none of them expl, are described in Beil **6**, 255, (126), (129), [243] & {802}, {822}, {860}
Trinitrodiphenylethers, $C_{12}H_7N_3O_7$; mw 305.20, N 13.77%. Five isomers: 2,4,2'-, 2,4,3'-, 2,4,4'-, 2,4,5'- & 2,4,6'- are described in Beil **6**, 255, (126), (129), [243] & {862}, {970}. They are, judging by their N content, weak expls
Tetranitrodiphenylethers, $C_{12}H_6N_4O_9$; mw 350.20, N 16.00%, OB to CO_2 minus 82.2%

Following isomers are described in the literature:

2,4,2',4'-Tetranitrodiphenylether, $(O_2N)_2C_6H_3.O.C_6H_3(NO_2)_2$; yel ndls (from alc), mp 194–95° (186°, probably impure); flash point above 360°; diff sol in alc or eth; easily sol in hot AcOH, ethyl acet, benz or chlf. It can be prepd by nitrating diphenylether with cold fuming nitric acid or with mixed nitric-sulfuric acid (Refs 1 & 2). It was prepd and examined by the Hercules Powder Co, Wilmington, Del and found to be of excellent stability. It can be detonated by No 6 (or stronger) caps, but not by impact or friction (Ref 3)
Refs: 1) Beil **6**, 255, (126), [243] & {863} 2) A. Mailhe & M. Murat, CR **154**, 715(1912); BullFr [4], **11**, 444(1912) & JCS **102**, I, 346 (1912) 3) A.H. Blatt, OSRD **2014**(1944)

2,4,6,2'-Tetranitrodiphenylether, $(O_2N)_3C_6H_2.O.C_6H_4(NO_2)$; ndls (from alc), mp 172–73°. It can be prepd by heating alcoholic picrylchloride with K-nitrophenolate
Refs: 1) Beil **6**, 291 & {970} 2) (?) Willgerodt & (?) Hüetlin, Ber **17**, 1766(1884). No later refs found in the literature

2,4,6,3'-Tetranitrodiphenylether, $(O_2N)_3C_6H_2.O.C_6H_4(NO_2)$; pale-yel lflts (from ethyl acetate), mp 169.5–171°. It can be prepd by warming chloroformic soln of picrylchloride with Na 3-nitrophenolate
Refs: 1) Beil **6**, (140) & [282] 2) F. Arnall, JCS **125**, 816(1924)

2,4,6,4'-Tetranitrodiphenylether, $(O_2N)_3.C_6H_2.O.C_6H_4(NO_2)$; lflts (from alc), mp 153°. It can be prepd by warming an alcoholic picrylchloride with K 4-nitrophenolate
Refs: 1) Beil **6**, 291 2) (?) Willgerodt & (?) Hüetlin, Ber **17**, 1766(1884). No later refs found in the literature

Pentanitrodiphenylethers, $C_{12}H_5N_5O_{11}$, mw 395.20, N 17.72%, OB to CO_2 minus 62.8%

Following isomers are described in the literature:

2,4,5,2',4'-Pentanitrodiphenylether, $(O_2N)_3C_6H_2.O.C_6H_3(NO_2)_2$; crysts (from eth acet); can be prepd by warming on a water bath for 1 hr 4,6-dinitro-3-hydroxylaminodiphenylether with concd nitric acid (sp gr 1.52)
Refs: 1) Beil **6**, [253] 2) W. Borsche & E. Feske, Ber **59**, 819(1926)

2,4,6,2',4'-Pentanitrodiphenylether or **2',4'-Dinitrophenyl-picrate,** $(O_2N)_3C_6H_2.O.C_6H_3(NO_2)_2$; crysts (from acet), mp 210–210.5° (Ref 1) & 208° (Ref 3); decompd by w; its solubility in various solvents was detd by Desvergnes (Ref 2). It can be prepd by nitration with mixed nitric-sulfuric acid of 2,4,6-Trinitrodiphenylether or of 2,4,6,2'- or of 2,4,6,4'-Tetranitrodiphenylether (Refs 1 & 3). It is an expl more sensitive to impact than TNT and less powerful (93% of TNT)
Refs: 1) Beil **6**, 291, [282] & {970} 2) L. Desvergnes, Moniteur Scientifique [5], **16**, 204(1924) & ChemZtr **1926**, II, 3043 3) A.H. Blatt, OSRD Rept **2014**(1944)

2,4,6,3',4'-Pentanitrodiphenylether or **3',4'-Dinitrophenylether-picrate,** $(O_2N)_3.C_6H_2.O.C_6H_3(NO_2)_2$; lflts (from eth acet), mp 200–204°; insol in w; sl sol in alc or eth; sol in benz, toluene & Nitrobenzene; can be prepd by nitration of 3,4-dinitrophenylether with mixed nitric-sulfuric acid at 110° (Refs 1, 2 & 3). It is a powerful expl patented in Germany by Westfälisch-Anhaltische AG during WWI for use as an HE (Refs 3 & 4)
Refs: 1) Beil **6**, (141) & [282] 2) C. van Duin & B. van Lennep, Rec **39**, 155(1920) 3) Clift & Fedoroff **2**(1943), p P5 4) A.H. Blatt, OSRD Rep **2014**(1944)

Hexanitrodiphenylethers, $C_{12}H_4N_6O_{13}$, mw 440.20, N 19.09%, OB to CO_2 minus 47.3%

Following isomers are described in the literature:

2,4,6,2',4',6'-Hexanitrodiphenylether or **Dipicrylether** or **Dipicryloxide.** $(O_2N)_3C_6H_2.O.C_6H_2(NO_2)_3$; col ndls or lflts; mp 252° (decomp) (Ref 4); insol in w; sl sol in alc and in eth; very sol in nitrobenzene; can be prepd by two-step nitration of diphenylether using nitric acid-oleum, first to tetra and then to hexa, or by one-step nitration of nitroderivatives unsubstituted in 3 and 3' positions, using mixed nitric acid-oleum at 120° (Refs 1, 2, 3 & 4)

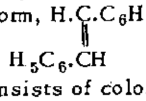
The following props are listed in Blatt (Ref 4)
Flash Point — above 360°
Impact Sensitivity — does not detonate
Initiation — only partial detonation with No 6 cap
Power, by Ballistic Mortar — 63.6% of Blasting Gelatin
Thermal Stability — excellent
Refs: 1) Beil **6**, {970} 2) A. Mailhe & M. Murat, CR **154**, 715(1912) & JCS **102**, I, 346 (1912) 3) Clift & Fedoroff **2**(1943), p H4 4) A.H. Blatt, OSRD Rept **2014**(1944)

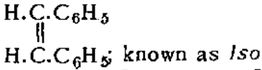
2,4,6,3′,4′,6′-Hexanitrodiphenylether or **3′,4′,6′-Trinitrophenylether-picrate** (called 2.4.5.2′.4′.6′-Hexanitro-diphenyläther in Beil), $(O_2N)_3C_6H_2.O.C_6H_2(NO_2)_3$; crysts, mp 269°; 278°(corr); insol in w; can be prepd by interaction of m-nitrophenol and 2,4-dinitrochlorobenzene, followed by nitration; can also be prepd by nitrating with mixed acid at 100° the hexa-nitroderivatives of diphenylether, such as 2,4,6,3′-Tetranitrodiphenylether (Refs 1, 2, 3 & 4)

The following properties are listed in Blatt (Ref 4):
Flash Point — expl at 318° on heating at 20°/min rise and at 313° at 5°/min rise
Impact Sensitivity with 2kg Kast app — max fall for 0/6 shots is 32–35cm vs 50–53 for Tetryl
Power — more powerful than Picric Acid
Thermal Stability at 95° — no change in 3 hours, but after 8 hrs nitrous fumes start to evolve
Refs: 1) Beil **6**, (141) & [282] 2) C. van Duin & B. van Lennep, Rec **39**, 145(1920) 3) Stettbacher(1933), 294 4) A.H. Blatt, OSRD Rept **2014**(1944)

Diphenylethylene and Derivatives

1,2-Diphenylethylene, Bibenzal or *Stilbene*, $C_6H_5.CH:CH.C_6H_5$, mw 180.24

Its *trans* form,  $\begin{array}{l}H.C.C_6H_5\\ \quad\|\\ H_5C_6.CH\end{array}$, known as *Toluylene*, consists of colorless (or sl yel) crysts, sp gr 0.970 at 125°/13°, mp 124–125°, bp 306–307°; insol in w; sol in benz & eth; sl sol in alc; can be prepd by passing toluene over hot oxide. It is used in manuf of dyes (Refs 1 & 3)

Its *cis* form,  $\begin{array}{l}H.C.C_6H_5\\ \quad\|\\ H.C.C_6H_5\end{array}$; known as *Isostilbene*, is a yel oil, fr p 1° and bp 145° at 13mm, d 1.0143 at 20°; n_D 1.6234 at 13°; can be obtd by exposing the trans- form to UV light, or from tolane (diphenylacetylene) thru electrolytic reduction at the Ni cathode in alcoholic sulfuric acid, and by other methods (Refs 2 & 3)

Diphenylethylene forms many addn comps & nitro derivs, some of which are expl
Refs: 1) Beil **5**, 630, (302), [537] & {1953} 2) Beil **5**, 633, (303), [539] & {1958} 3) CondChemDict (1961), 1076-R & 1077-L (Stilbene)

Mononitrodiphenylethylene or *Nitrostilbene*, $C_6H_5.CH:CH.C_6H_4.NO_2$; mw 225.24, N 6.22%. The following isomers are described in Beil: 2-Nitro, 3-Nitro, 4-Nitro & α-Nitro-stilbene, $C_6H_5.CH:C(NO_2).C_6H_5$ (Ref)
Ref: Beil **5**, 636, (305), [540] & {1966–68}

Dinitrodiphenylethylene or **Dinitrostilbene,** $C_{14}H_{10}N_2O_4$; mw 270.24, N 10.37%.
The following isomers are known:
2,4-Dinitrostilbene, $C_6H_5.CH:CH.C_6H_3(NO_2)_2$, exists in high- & low- melting forms (Ref 1)
2,5-Dinitrostilbene, brn ndls (from alc), mp 149.5°(Ref 2)
2,6-Dinitrostilbene, orn-yel ndls (from aq alc), mp 111°(Ref 3)
2,2′-Dinitrostilbene, trans, $O_2N.C_6H_4.CH:CH.C_6H_4.NO_2$, lt-yel ndls (from chlf), mp 191–96° & 199°, bp — explodes on heating to 420° in a tube; v sl sol in alc, eth & petr eth; sol in hot benz; was obtd, in addn to a low-melting form by treating a soln of o-nitrobenzylchloride in 98% alc with a soln of potash in alc, and separating the isomers by crystg the product from epichlorohydrin in which the higher-melting isomer is v sl sol in the cold solv (Ref 3)
2,3′-Dinitrostilbene, orn-yel ndls (from alc), mp 150°, bp — sublimes at 160° under 0.001mm pressure (Ref 4)

2,4′-Dinitrostilbene, trans, yel pltlts (from alc), mp 143° or yel ndls (from glac acet ac), mp 140°; bp – sublimes at 130–40° under 0.001mm press; cis, yel-brn ndls (from alc or glac acet ac), mp 140° (Ref 4)

3,3′-Dinitrostilbene, trans, yel ndls (from glac acet ac), mp 240–42°, bp – sublimes at 175° under 0.001mm press (Ref 4)

3,4′-Dinitrostilbene, trans, yel ndls (from glac acet ac, Nitrobenz or pyridine), mp 220–22°, bp – sublimes at 170° under 0.001mm press; cis, yel ndls (from glac acet ac), mp 155° (Ref 5)

4,4′-Dinitrostilbene, trans, yel ndls (from glac acet ac or Nitrobenz), mp 292–95° & 303–04°; cis, yel crysts or tablets (from glac acet ac or acetone), mp 183–86° & 234–35° (Ref 6)

2,α′-Dinitrostilbene, $C_6H_5.C(NO_2):CH.C_6H_4.NO_2$, lt-yel ndls (from alc), mp 106° (Ref 7)

3,α′-Dinitrostilbene, crysts (from alc), mp 112° (Ref 7)

4,α′-Dinitrostilbene, cis, yel ndls (from ethyl acet + petr eth), mp 155–157.5° (Ref 8)

α,α′-Dinitrostilbene, $C_6H_4\,C(NO_2):C(NO_2).C_6H_4$, trans, lt-yel ndls or prisms (from alc), mp 185–86°; cis, yel pyramids (from alc), mp 104–08°, bp – dec on heating above 150° (Ref 9)

3,α-Dinitrostilbene, $C_6H_5.CH:C(NO_2).C_6H_4.NO_2$, crysts (from benz + petr eth), mp 179–80° (Ref 7)

Other props & methods of prepn are found in the Refs

Refs: 1) Beil **5**, 636, (305), [540] & {1970} 2) Beil **5**, {1970} 3) Beil **5**, 637, (306) & {1970} 4) Beil **5**, {1971} 5) Beil **5**, [541] & {1971} 6) Beil **5**, 637, (306), [541] & {1971} 7) Beil **5**, {1972} 8) Beil **5**, [541] & {1973} 9) Beil **5**, 637, [541, 542] & {1973}

Trinitrodiphenylethylene or **Trinitrostilbene**, $C_{14}H_9N_3O_6$; mw 315.24, N 13.33%. The following isomers are described in the literature:

2,4,6-Trinitrostilbene or *1-(2,4,6-Trinitrophenyl)-2-phenylethylene*, $C_6H_5.CH:CH.C_6H_2(NO_2)_3$, trans, yel ndls (from hot acet ac), or yel tablets + 1 mol benz (from benz), dec in light, mp 156–58°; sol in benz & Nitrobenz; v sl sol in CCl_4, glac acet ac, alc & eth; was prepd by heating 2,4,6-trinitrotoluene & benzaldehyde in the presence of piperidine on a water bath for ½ hr (Ref 1). Its sensitivity to impact is FI 109% PA and its power is less than that of PA (Ref 6)

2,4,2′-Trinitrostilbene or *1-(2,4-dinitrophenyl)-2-(2-nitrophenyl)-ethylene*, $O_2N.C_6H_4.CH:CH.C_6H_3(NO_2)_2$, trans, gm-yel crysts (from glac acet ac), mp 194–95°; was prepd by reacting 2,4-dinitrotoluene with 2-nitrobenzaldehyde in the presence of piperidine (Ref 2)

2,4,3′-Trinitrostilbene, yel ndls (from glac acet ac), mp 183–84°; v sl sol in hot alc; was prepd by heating 2,4-dinitrotoluene & 3-nitrobenzaldehyde in the presence of piperidine (Ref 3)

2,4,4′-Trinitrostilbene, trans, dk-orn ndls, mp 234–35° & 240°; sol in hot glac acet ac; was prepd by heating 2,4-dinitrotoluene & 4-nitrobenzaldehyde in the presence of piperidine (Ref 4)

3,4′,α-Trinitrostilbene or *1-(4-Nitrophenyl)-2-nitro-2-(3-Nitrophenyl)-ethylene*, $O_2N.C_6H_4.C:C(NO).C_6H_4.NO_2$, brownish crysts (from toluene), mp 236–37°; was prepd by heating 3-nitro-1-nitromethylbenzene & 4-nitrobenzaldehyde in the presence of alcoholic methylamine soln (Ref 5)

4,4′,α-Trinitrostilbene or *1-Nitro-1,2-di(4-nitrophenyl)-ethylene*, grn-yel crysts (from ethylacetate), mp 210.5–211°; was obtd with other products by nitrating 4,α′-Dinitrostilbene (mp 155°) with nitric acid (d 1.49) at minus 20° to 15° (Ref 5)

Other props & methods of prepn are given in the Refs

Refs: 1) Beil **5**, 638, [542] & {1973} 2) Beil **5**, 638 & {1973} 3) Beil **5**, 638 & [542] 4) Beil **5**, 638, (307), [542] & {1974} 5) Beil **5**, {1974} 6) A.H. Blatt, OSRD **2014** (1944)

Tetranitrodiphenylethylene or **Tetranitrostilbene**, $C_{14}H_8N_4O_8$; mw 360.24, N 15.55%. Five isomers are known:

2,4,6,2'-Tetranitrostilbene or *1-(2,4,6-Trinitrophenyl)-2-(2-nitrophenyl)-ethylene*, $O_2N.C_6H_4.CH{:}CH.C_6H_2(NO_2)_3$, yel-brn ndls (from alc), mp 181°; was prepd from 2,4,6-Trinitrotoluene & 2-nitrobenzaldehyde in the presence of piperidine in hot benzene (Refs 1 & 7)

2,4,6,3'-Tetranitrostilbene, trans, yel pltlts (from benz + alc), mp 159°; was prepd by reacting 2,4,6-TNT & 3-nitrobenzaldehyde in the presence of piperidine in boiling benz (Refs 1 & 7)

2,4,6,4'-Tetranitrostilbene, yel ndls, mp 196°; readily sol in boiling glac acet ac; sl sol in benz; v sl sol in alc; was prepd from reaction of 2,4,6-TNT & 4-nitrobenzaldehyde in alc and in presence of piperidine (Refs 2 & 5)

2,4,2',4'-Tetranitrostilbene or *1,2-Bis-(2,4-dinitrophenyl)-ethylene*, $(O_2N)_2C_6H_3.CH{:}CH.C_6H_3(NO_2)_2$, exists as high-melting form: yel ndls (from chlf, acet, Nitrobenz or glac acet ac), mp 269°(dec); was prepd by action of alcoholic KOH on 2,4-dinitrobenzylchloride; forms the lower-melting form on exposure to light: orn-colored crysts (from glac acet ac), mp 263°; stable in sunlight; sol in hot Nitrobenz & hot glac acet ac; insol in acet & chlf (Refs 3, 8 & 9)

2,6,2',6'-Tetranitrostilbene or *1,2-Bis-(2,6-dinitrophenyl)-ethylene*, lt-yel ndls (from Nitrobenz), mp 250°; sol in acet; insol in alc, eth & petr eth; was prepd by reacting alcoholic KOH on 2,6-dinitrobenzylbromide (Refs 4 & 6)

Other props are found in the Refs

Refs: 1) Beil **5**, [542] & {1974} 2) Beil **5**, 638 3) Beil **5**, 638, [543] & {1974} 4) Beil **5**, (307) 5) F. Ullmann & M. Gschwind, Ber **41**, 2297(1908) & CA **2**, 2933(1908) 6) S. Rich et al, Ber **45**, 3059(1912) & CA **7**, 1011(1913) 7) G. Bishop & O.L. Brady, JChemSoc **121**, 2367(1922) & CA **17**, 555 (1923) 8) A. Plissov, JChimUkraine **1**, 418(1925) & CA **20**, 2851(1926) 9) A.H. Blatt & A.W. Rytina, JACS **72**, 405(1950) & CA **45**, 1081(1951)

Pentanitrodiphenylethylene or **2,4,6,2',4'-Pentanitrostilbene** or **1-(2,4,6-Trinitrophenyl)-2-(2,4-dinitrophenyl)-ethylene**, $(O_2N)_2C_6H_3.CH{:}CH.C_6H_2(NO_2)_3$; mw 405.24 N 17.28%; grn-yel ndls (from benz + acetone), mp 198–99°; prepd by heating 2,4,6-trinitro-trans stilbene with nitric acid (d 1.41) & concd sulfuric acid at 100°. Oxidization of the pentanitro deriv with nitric acid (d 1.5) & concd sulfuric acid at 120–30° yielded *2,4,6,2',4'-Pentanitrobenzil* (See in Vol 2, p B65-L)

Pentanitrostilbene is an expl compd which has not been investigated for military applications

Refs: 1) Beil **5**, {1975} 2) F. Challenger & P.H. Clapham, JChemSoc **1948**, 1614 & CA **43**, 1733(1949)

Hexanitrodiphenylethylene, 2,4,6,2',4',6'-Hexanitrostilbene (HNS) or **1,2-Bis-(2,4,6-Trinitrophenyl)-ethylene**, $(O_2N)_3C_6H_2.CH{:}CH.C_6H_2(NO_2)_3$; mw 450.24, N 18.67%; yel ndls (from Nitrobenz), mp 316° (dec), mp – explodes; sol in dimethyl formamide; sl sol in hot acet, methyl ethyl ketone & glac acet acid. This compd was prepd by Shipp (Ref 5) from 2,4,6-Trinitrobenzyl chloride in methanol by heating on a steam bath with methanol contg KOH. Since this product melts at 105° higher than that reported by Reich et al (Refs 1 & 2), its identity was confirmed by mw detn, elemental analysis, and by an unambiguous independent synthesis. Its X-ray diffraction measurements were also made & its unit cell dimensions reported. The product of Reich et al [mp 211°(dec), correct value 218–20°] is probably 2,4,6,2',4',6'-Hexanitrobibenzil

HNS is a heat-resistant expl developed at NOL (Ref 5). Several industries, such as Northrup-Carolina Inc, Del Mar Engineering Laboratories, and Jet Research Inc, now manuf HNS under Navy Procurement Specification WS-5003C & D (Ref 7). Del Mar, anticipating wide & increasing use of this versatile expl, has designed and constructed pilot-plant scale synthesis capable of producing 300lb/month of both Type I & Type II HNS, Grade A & Grade B

Del Mar in its data sheet (Ref 8) reports the following physical, sensitivity & expl props of its product:

	Type I	Type II
Melting point, °C	316(dec)	319(dec)
°F	601(dec)	607(dec)
Particle size, microns	1–5	100–300
Bulk density, g/cc	0.32–0.45	0.45–1.0
Differential thermal analysis, onset of exotherm	315°C(599°F)	325°C(617°F)
Electrostatic spark sensitivity, fires above, μfd	0.001 @8kv	0.0001 @17kv
Friction sensitivity, 10/10 No fires	440 kg/cm	440 kg/cm
Impact sensitivity, 50% height, cm NOL-ERL Machine, Type 12 tools 5/0 Sandpaper, 2.5kg wt	44	61
Vacuum stability @260°C:		
1st 20mins, cc/g/hr	1.8	0.3
Addnl 2hrs, cc/g/hr	0.6	0.2
@280°	2.7	2.7
Velocity of deton, m/sec	7000	7000
density, g/cc	1.70	1.70
Autoignition point	325°C(618°F)	325°C(618°F)
Decomposition rate @260°	0.1%/hr	0.1%/hr
Heat of combustion, cal/g	3451	3451
Mean firing voltage for EBW, 1 μfd capacitor	12950 @ 0.9g/cm^3	12950 @ 0.9g/cm^3
Vapor pressure	2.9×10^{-8}mmHg @160°C(320°F)	2.9×10^{-8}mmHg @160°C(320°F)

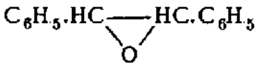
HNS has a uniquely small critical diam (~0.020 inch), is practically insensitive to electrostatic spark, is less sensitive to impact than Tetryl, is radiation resistant, and is the only commercially available expl meeting the expl sensitivity requirements of the Fuze Design Safety Criteria of all three services (Ref 8)

Uses: HNS is used as a heat-resistant booster expl. It is sufficiently insensitive to heat, impact, and electrostatic spark to be used in mild detonating fuse (MDF) end couplers & end boosters. It may find application in leads and in plastic-bonded HE's

Refs: 1) Beil **5**, (307) 2) S. Reich et al, Ber **45**, 3060(1912) & CA **7**, 1011(1913) 3) A.H. Blatt, OSRD **2014**(1944), not found 4) ADL Synth HE, Rept No 1(1949), p86 5) K.G. Shipp, JOrgChem **29**, 2620–23(1964) & CA **61**, 10609(1964) 6) K.G. Shipp, "Heat Resistant Explosives XVI – A New Synthesis of 2,2',4,4',6,6'-Hexanitrostilbene, HNS", NOLTR **64–34**(1964) (Conf, not used as a source of info) 7) Navy Dept, Bureau of Weapons, NOL, Md, "Purchase Description, HNS Explosive WS 5003C & D" (June 1965) (Unclassified) 8) Del Mar Engineering Laboratories, Los Angeles, Calif (no date)

N,N'-Diphenyl-ethylenediamine and Its Nitrated Derivatives. See Bis(anilino)-ethane and Derivatives in Vol 2, pB131-L. Its *Hexanitro* compd is code-named *Bitetryl, Ditetryl* or *Octyl* and is described on pp B131-R & B132-L

α,α'-Diphenyl-ethylene Ether or **Stilbene Oxide and Derivatives** (called α,α'-Diphenyl-äthylenoxyd or Stilbenoxyd in Ger),

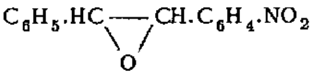


$C_6H_5.HC\!-\!\!-\!HC.C_6H_5$ (O)

mw 196.24, O 8.15%; exists as trans form, pltlts (from 70% alc), mp 69–70.3°; v sl volatile on a steam bath; sol in eth; was prepd by treating N,N-dimethyldiphenylhydroxyethylamine-iodomethylate with Ag oxide and boiling the base with water. The cis form, ndls (from 70% alc), mp 42° & 56°; was similarly prepd using the N,N-dimethylisodiphenyl deriv (Refs 1 & 2)

Refs: 1) Beil **17**, (31) & [72] 2) P. Rabe & J. Hallensleben, Ber **43**, 884, 885(1910) & CA **4**, 1749(1910)

Mononitro-α,α'-diphenyl-ethylene Ether or **Mononitrostilbene Oxide,**



$C_6H_5.HC\!-\!\!-\!CH.C_6H_4.NO_2$ (O)

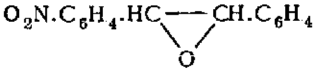
mw 241.24, N 5.80%. Two isomers are known: *2-Nitrostilbene Oxide*, high-melting form:

crysts (from alc), mp 101–02°; sol in hot alc, eth, acet & glac acet ac; less sol in the cold solvs; and low-melting form: crysts (from alc), mp 69°; sol same as h-melting form. Both forms obtd by treating benzaldehyde with 2-nitrobenzylchloride in alcoholic KOH (Refs 1 & 2)

4-Nitrostilbene Oxide, high-melting form, crysts (from alc or propyl alc), mp 125–26°; readily sol in chlf, eth, acet & glac acet ac; sl sol in alc; and low-melting form, crysts (from alc), mp 74–76°; more sol than the h-melting form. Both forms prepd by treating benzaldehyde with 4-nitrobenzylchloride in alc KOH (Refs 1 & 2)

Refs: 1) Beil **17**, [72, 73] 2) O. Chrzescinski, Dissertation, Strassburg (1911), 42, 43

Dinitro-α,α′-diphenyl-ethylene Ether or **Dinitrostilbene Oxide,**



$$O_2N.C_6H_4.\underbrace{HC{-}{-}CH}_{O}.C_6H_4$$

mw 286.24, N 9.79%. Three isomers are known: *2,4′-Dinitrostilbene Oxide*, high-melting form: crysts (from acet or ethyl acetate), mp 160–61°; readily sol in chlf, benz, acetone, glac acet ac & ethyl acetate; sl sol in alc & eth; v sl sol in w; and low-melting form: leaflets (from MeOH) & crysts (from alc), mp 127–28° & 112° (uncorrected); same solubility as h-melting form. Both forms prepd from either 2-nitrobenzylchloride & 4-nitrobenzaldehyde, or 4-nitrobenzylchloride & 2-nitrobenzaldehyde by treating with alcoholic KOH (Refs 1 & 2)

3,4′-Dinitrostilbene Oxide, high-melting form: prisms (from ethyl acetate), mp 148°; and low-melting form: crysts (from MeOH or propyl alc), mp 116°. Both forms obtd by heating 4-nitrobenzylchloride with 3-nitrobenzaldehyde in the presence of K_2CO_3 in MeOH (Refs 1 & 3)

4,4′-Dinitrostilbene Oxide, high-melting form: tablets (from glac acet ac) or crysts (from ethyl acetate), mp 200–01°; sol in hot Nitrobenz, sl sol in glac acet ac, alc, chlf, acet & dioxane; and low-melting form: ndls (from glac acet ac), mp 162–63°. Both forms obtd by treating 4-nitrobenzylchloride & 4-nitrobenzaldehyde with alcoholic KOH at RT, or by heating with K_2CO_3 in MeOH (Refs 1 & 3)

Refs: 1) Beil **17**, [73, 74] 2) O. Chrzescinski, Dissertation, Strassburg (1911), 38 3) E. Bergmann & J. Hervey, Ber **62**, 904–06 (1929) & CA **23**, 3919 (1929)

NOTE: Higher nitrated derivs of Stilbene Oxide, such as *Trinitro*, $C_{14}H_9N_3O_7$, *Tetranitro*, $C_{14}H_8N_4O_9$, *Pentanitro*, $C_{14}H_7N_5O_{11}$, and *Hexanitro*, $C_{14}H_6N_6O_{13}$, were not found in the literature thru 1966

Diphenyl-ethyleneglycol Ether or **Diphenoxyethane.** See Bis(phenyl)-ethyleneglycol Ether in Vol 2, p B153-R

1,1-Diphenyl-ethylene Peroxide, Polymeric, $\left[O.H_2C.C(C_6H_5)_2.O\right]_x$; mw $(212.24)_x$, O (15.08%); solid, mp – detonates on heating; can be prepd by exposing 1,1-diphenylethylene, $H_2C{:}C(C_6H_5)_2$ to air in the presence of ultraviolet light

Refs: 1) Beil, not found 2) H. Staudinger, Ber **58**, 1075 (1925) & CA **19**, 2658 (1925) 3) Tobolsky & Mesrobian (1954), 22 & 178

Diphenyl-ethylol and Derivatives

1,2-Diphenyl-1-ethylol; Diphenylethanol or *Diphenyl-hydroxyethane,*

$C_6H_5.CH(OH).CH_2.C_6H_5$; mw 198.25. Exists as inactive form: crysts (from eth + petr eth, dil alc or glac acet ac), mp 62–68°; its levorotatory form: crysts from eth + petr eth), mp 67°; and as dl-form: crysts, mp 66–69° (Ref). Other props & methods of prepn are given in Beil

Ref: Beil **6**, 683, (329), [637, 638] & {3390}

2-Nitro-1,2-diphenyl-1-ethanol,

$C_6H_5.CH(OH).CH(NO_2).C_6H_5$; mw 243.25, N 5.76%; crysts (from benz + petr eth), mp 97–98°; was first prepd by Dornow & Gellrich (Ref 2) by reaction of phenylnitromethane ($PhCH_2.NO_2$) in NaOH with benzaldehyde treated with $NaHSO_3$ in H_2O. See also Refs 3–6

Refs: 1) Beil, not found 2) A. Dornow &

M. Gellrich, Ann **594**, 183(1955) & CA **50**, 11346(1956) 3) J.J. Gardikes, Chem & Ind (London) **1958**, 632 & CA **53**, 3144(1959) 4) H. Shechter et al, JACS **81**, 5420(1959) & CA **54**, 8724(1960) 5) T.E. Stevens, JACS **81**, 3593(1959) & CA **54**, 22706(1960) 6) F.G. Bordwell & E.W. Garbisch Jr, JOrgChem **27**, 2322(1962) & CA **57**, 7142(1962)

3,4′-Dinitrodiphenyl-1-ethylol or *1-(3-nitrophenyl)-2-(4-nitrophenyl)-ethanol,* $O_2N.C_6H_4.CH(OH).CH_2.C_6H_4.NO_2$; mw 288.25, N 9.72%; crysts (from alc), mp 147–48°; sol in alc, glac acet ac, ethyl acetate & acetone; less sol in eth & benz; was prepd by heating 3-nitrobenzaldehyde & 4-nitrophenylacetic acid with piperidine present for 1 hour at 110° & 2 hrs at 150°, addn of water ppts the product which on heating with piperidine or treating with cold concd sulfuric acid yields 3,4′-Dinitrostilbene (See under Diphenylethylene and Derivatives in this Vol)
Refs: 1) Beil **6**, [638] 2) H.A. Harrison & H. Wood, JChemSoc **1926**, 580 & CA **20**, 1801(1926)

2-Nitro-1,2-diphenylethanol Nitrate, $C_6H_5.CH(ONO_2).CH(NO_2).C_6H_5$; mw 288.25, N 9.72%; crysts (from petr eth), mp 165–66° (dec); was obtd by action of dinitrogen pentoxide (N_2O_5) in methylene chloride on trans-stilbene in tetraethylammonium nitrate at minus 20° for 15 mins (Ref 2)
Refs: 1) Beil, not found 2) T.E. Stevens, JOrgChem **24**, 1137(1959) & JACS **81**, 3593 (1959); CA **54**, 5566 & 22706(1960)

Trinitrodiphenyl-ethylol, $C_{14}H_{11}N_3O_7$, not found in the literature thru 1966

Tetranitrodiphenyl-ethylol, $C_{14}H_{10}N_4O_9$, not found in the literature thru 1966

2,4,2′,4′,5′-Pentanitrodiphenyl-ethylol or **(±)2-(2,4-Dinitrophenyl)-1-(2,4,5-Trinitrophenyl)-2-ethanol,** $(O_2N)_2C_6H_3.CH(OH).CH_2.C_6H_2(NO_2)_3$, mw 423.25, N 16.55%, OB to CO_2 minus 81.3%; crysts (from toluene), mp 187°; bp – explodes at 360°; insol in w; sol in hot benz, toluene, acetone, 1,2-dichloroethane & 1,1,2,2-tetrachloroethane. It can be prepd by nitration of tetranitrodiphenylethane with mixed nitric-sulfuric acid at 85° for 4 days

Following props were detd at PicArsn:
Brisance by Sand Test – 0.4g sample detonated by 0.25g MF and 0.25g Tetryl crushed 21g sand vs 30g for TNT
Impact Sensitivity, with PicArsn apparatus, using 2kg wt, positive with 18 inches drop
Initiation Sensitivity – 0.4g could not be initiated with 0.25g MF alone but did initiate with 0.25g MF + 0.25g Tetryl (Refs 2, 3 & 4)
Refs: 1) Beil **6**, {3393} 2) H.A. Aaronson & W.H. Rinkenbach, JACS **52**, 5042(1930) 3) A.H. Blatt & F.C. Whitmore, OSRD Rept **1085**(1942), p6 4) A.H. Blatt, OSRD Rept **2014**(1944)

2,4,6,2′,4′,6′-Hexanitrodiphenyl-ethylol or *Dipicrylethanol,* $C_{14}H_8N_6O_{13}$, not found in the literature thru 1966

N,N′-Diphenylformamidine and Derivatives
N,N′-Diphenylformamidine or *Methenyl-diphenyl-diamine,* $C_6H_5.N:CH.NH.C_6H_5$; mw 196.24, N 14.28%; ndls (from alc or benz), mp 139–43°; bp – sl decomp at higher temp; very sol in chlf, sol in eth, sl sol in petr eth. It can be prepd by heating chloroform & aniline in a tube at 180–90°; forms many cryst addn compds (Refs 1 & 2). Other props & other methods of prepn are found in Beil
Refs: 1) Beil **12**, 236, (190) & [137] 2) A.W. Hofmann, CR **147**, 352(1858); AnnChim [3], **54**, 197(1858) & Jahresber Fortschr der Chemie **1858**, 354 3) W. Bradley & I. Wright, JChemSoc **1956**, 644 & CA **50**, 13806 (1956) (Metal derivs)

Azido, $C_{13}H_{11}N_5$, and *Diazido,* $C_{13}H_{10}N_8$, derivs of N,N′-Diphenylformamidine were not found in the literature thru 1966

Mononitrodiphenylformamidine, $C_{13}H_{11}N_3O_2$; mw 241.24, N 17.43%. The following derivs are found in the literature:

N-Phenyl-N′-(3-Nitrophenyl)-formamidine, $C_6H_5.N{:}CH.NH.C_6H_4.NO_2$ or $C_6H_5.NH.CH{:}N.C_6H_4.NO_2$; yel prisms (from abs alc), mp 145°; was prepd from reaction of 3-nitroisoformanilide-O-methylether, ($O_2N.C_6H_4.N{:}CH.O.CH_3$), & aniline (Refs 1, 2 & 3)
N-Phenyl-N′-(4-Nitrophenyl)-formamidine, yel crysts (from toluene & then from alc), mp 181–85°; was obtd when p-nitroaniline & ethyl-N-phenylformamidate, $[HC({:}N.C_6H_5)OC_2H_5]$ were heated in a distilling flask at 180° for 2 hrs, and the mixt poured while hot into dry toluene, pptg the product which was collected and recrystd (Refs 2, 3, 4, 5 & 6)

No expl props were reported
Refs: 1) Beil **12**, 703 2) R.M. Roberts et al, JACS **73**, 2277(1951) & CA **46**, 1477 (1952) 3) H.G. Mandel & A.J. Hill, JACS **76**, 3979(1954) & CA **49**, 10226(1955) 4) W. Bradley & I. Wright, JChemSoc **1956**, 640 & CA **50**, 13806(1956) (Metal derivs) 5) S.O. Onyiriuka & A.H. Rees, JChemSoc, Org **1966**, 504 & CA **64**, 11148(1966)

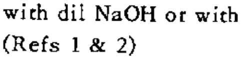
Dinitrodiphenylformamidine, $C_{13}H_{10}N_4O_4$; mw 286.24, N 19.58%. Three isomers are known:
N,N′-Bis(2-nitrophenyl)-formamidine, $O_2N.C_6H_4.N{:}CH.NH.C_6H_4.NO_2$, ndls, mp 160–61° [Beil, 124–25°, Ref 1]; was prepd by interaction of P pentachloride in a chlf medium upon o-nitroformamide & o-nitroaniline (Refs 1, 5, 7 & 9)
N,N′-Bis(3-nitrophenyl)-formamidine, ndls (from abs alc), mp 201–03°; sl sol in alc, eth & benz; insol in petr eth; was prepd from reaction of 3-nitro-isoformanilide & 3-nitroaniline (Ref 2), and by the method of Roberts (Ref 4) & of Mandel (Ref 5). See also Refs 6, 7 & 10
N,N′-Bis(4-nitrophenyl)-formamidine, leaflets, mp 239–40° & 245–46°; readily sol in warm acet; was prepd from 4-nitroaniline & dichloromethylformamidine hydrochloride in benz & in the presence of pyridine (Ref 3). Other methods of prepn are also given (Refs 1, 4, 5, 7, 8, 10 & 11)

No expl props of the Dinitro deriv are reported except that their metal derivs are unstable & decomp on heating (Ref 6)
Refs: 1) Beil **12**, 691 2) Beil **12**, 703 3) Beil **12**, 718 4) R.M. Roberts et al, JACS **73**, 2279(1951) & CA **46**, 1477(1952) 5) H.G. Mandel & A.J. Hill, JACS **76**, 3979 (1954) & CA **49**, 10266(1955) 6) W. Bradley & I. Wright, JChemSoc **1956**, 644 & CA **50**, 13806(1956) 7) P. Grammaticakis, CR **245**, 2307(1957) & CA **52**, 9761(1958) (UV absorption spectra) 8) H. Bredereck et al, ChemBer **92**, 837(1959) & CA **53**, 14996 (1959) 9) J.W. Clark-Lewis & M.J. Thompson, JChemSoc **1959**, 2401 & CA **54**, 2349 (1960) 10) R.H. DeWolfe, JACS **82**, 1585 (1960) & CA **54**, 18398(1960) (Hydrolysis in acid solns); Ibid, **86**, 864(1964) & CA **60**, 9115(1964) (Hydrolysis in alk dioxane solns) 11) P. Lochon & Mrs Jambu-Geoffroy, BullFrance **1965**, 393 & CA **62**, 14658(1965)

NOTE: Higher nitrated derivs of Diphenylformamidine, such as *Trinitro*, $C_{13}H_9N_5O_6$, *Tetranitro*, $C_{13}H_8N_6O_8$, *Pentanitro*, $C_{13}H_7N_7O_{10}$ and *Hexanitro*, $C_{13}H_6N_8O_{12}$, were not found in the literature thru 1966

Diphenylfuroxan and Derivatives

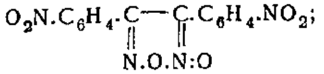
Diphenylfuroxan, Bis(phenyl)-furoxan or *3,4-Diphenylfurazan Oxide*,



$$\begin{array}{l} C_6H_5-C-C.C_6H_5 \\ \qquad\ \ \| \quad \| \\ \qquad\ N.O.N{:}O \end{array}$$

mw 238.24, N 11.76%; ndls or leaflets (from alc or glac acet ac), mp 112–15°; bp – decomp on heating rapidly into phenylisocyanate; sol in benz & chlf; sl sol in eth; v sl sol in cold alc; insol in w; was prepd by treating benzohydroxamicacid chloride with dil NaOH or with concd NaOH in eth (Refs 1 & 2)
Refs: 1) Beil **27**, 590, (577) & [645] 2) W. Steinkopf & B. Jürgens, JPraktChem [2] **84**, 692, 712(1911) & CA **6**, 988(1912)

Dinitro-3,4-diphenylfuroxan or **Bis(nitrophenyl)-furazan Oxide,**



$$\begin{array}{l} O_2N.C_6H_4.C—C.C_6H_4.NO_2; \\ \qquad\qquad \|\quad\ \| \\ \qquad\qquad N.O.N{:}O \end{array}$$

mw 328.24, N 17.07%. Two isomers are known:

Bis-(3-nitrophenyl)-furoxan, ndls (from glac acet ac or acet) & yel ndls (from xylene), mp 183–85°; readily sol in hot xylene, warm glac acet ac & acetone; insol in w, alc, eth, petr eth, benz & chlf; was prepd by oxidation of 3-nitro-α-benzaldoxime with isoamylnitrite in hot benz, or by shaking it with benzoic acid or m-nitrobenzoic acid Ag salt in eth; and by other methods (Refs 2, 3, 4, 5, 6, 7 & 8)

Bis-(4-nitrophenyl)-furoxan, ndls (from alc or glac acet ac), mp 197–98°; readily sol in warm glac acet ac; almost insol in w, alc, eth, benz & petr eth; was prepd by heating 4-nitrobenzohydroxime acid chloride with concd KOH, and by other methods (Ref 1)

Refs: 1) Beil **27**, 591 2) Beil **27**, 591 & [645] 3) O.L. Brady & F.H. Peakin, JChemSoc **1929**, 2269 & CA **24**, 604(1930) 4) ADLittle, SynthHE's, 3rd Rept (1953), 359 5) N.E. Boyer et al, JACS **77**, 420 (1955) & CA **49**, 15487(1955) (IR absorption spectrum) 6) ADL, SynthHE's, 4th Rept (1956), 59 7) N. Kornblum & W.M. Weaver, JACS **80**, 4333(1958) & CA **53**, 4261(1959) 8) R.H. Wiley & B.J. Wakefield, JOrgChem **25**, 546(1960) & CA **54**, 19657(1960)

NOTE: *Tetranitro*, $C_{14}H_6N_6O_{10}$, and *Hexanitro*, $C_{14}H_4N_8O_{14}$ derivs of Diphenylfuroxan were not found in the literature thru 1966

Diphenyl-glycerylether and Derivatives

α,α'-Diphenylglycerylether or *1,3-Diphenoxy-2-propanol*, $C_6H_5–O.CH_2.CH(OH).CH_2.O–C_6H_5$, mw 244.28, O 19.65%; pearly pltlts (from alc), mp 80–82°, bp 224.5° at 17.5mm Hg, sublimes on careful heating w/o decompn, sp gr 1.179 at 24/4°; insol in w; sol in eth, chlf, benz & hot alc; can be prepd by heating (±) epichlorohydrin with Na phenolate & phenol in dioxane at 100–05° and by other methods (Ref 1)

1,3-Bis-O-(2,4-dinitrophenyl) glycerin or *1,3-Bis(2,4-dinitrophenoxy)-2-propanol*, $(O_2N)_2C_6H_3.O.CH_2.CH(OH).CH_2.O.C_6H_3(NO_2)_2$; mw 424.28, N 13.21%; crysts (from acet), mp 173.5–174° (Pt block). It was obtd with other products by heating 4-chloro-1,3-dinitrobenzene with glycerin & NaOH (Refs 2 & 6)

No expl props are reported

1,3-Di-O-picrylglycerin; 1,3-Dipicrylhydroxy-2-propanol; or 1,3-Bis-(2,4,6-trinitrophenoxy)-2-propanol; $(O_2N)_3C_6H_2.O.CH_2.CH(OH).CH_2.O.C_6H_2(NO_2)_3$; mw 514.28, N 16.34%; crysts, mp 173–75°; a color reaction occurs by treating the compd in alc or acet soln with alc KOH or alc NH_3. It was prepd by heating 1,3-bis-(2,4-dinitrophenoxy)-2-propanol with aq nitric acid & concd sulfuric acid (Refs 3 & 6)

No expl props are reported

1,3-Dipicrylglycerylether-β-Nitrate or **2,4,6,-2',4',6'-Hexanitrodiphenyl-glycerylether Nitrate,** $(O_2N)_3C_6H_2–O.CH_2.CH(ONO_2).CH_2.O–C_6H_2(NO_2)_3$; mw 559.28, N 17.53%, OB to CO_2 minus 50%; dk-yel solid, mp 158–62° (dec). It can be prepd by nitrating the parent compd in sulfuric acid with the usual mixed acid (Refs 4 & 5)

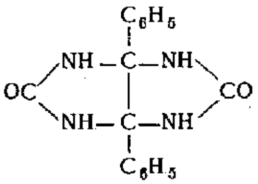
Its *Impact Sensitivity* with 2kg wt app is 115cm vs 40cm for Picric Acid. *Power by Trauzl Test* – 111% of TNT (Refs 7 & 8)

Refs: 1) Beil **6**, 149, (86), [152] & {582} 2) Beil **6**, (126) & {865} 3) Beil **6**, {971} 4) Beil **6**, (141) 5) Dynamite Nobel, BritP 24352(1914) & CA **10**, 1597(1916); GerP 294813(1916) 6) L. Desvergnes, AnnChimAnalAppl [2] **13**, 321(1931) & Chim et Ind **25**, 811, 817(1931) 7) A.H. Blatt & F.C. Whitmore, OSRD Rept **1085**(1942), p47 8) A.H. Blatt, OSRD **2014**(1944)

Diphenylglycolic Ether. See Bis(phenyl)-ethyleneglycol Ether in Vol 2, p B153-R

Diphenylglycoluril and Derivatives

7,8-Diphenylglycoluril; Diphenylacetylenediurein or *Diphenylacetyleneurea*,



mw 294.30, N 19.04%; prisms or pltlts (from glac acet ac), mp >310°(Ref 1); was prepd from benzil, $C_6H_5.CO.CO.C_6H_5$, and urea, $H_2N.CO.NH_2$ as described in Ref 3, p10

Trinitro-7,8-diphenylglycoluril or **Trinitrodiphenylacetylenediurein,** $C_{16}H_{11}N_7O_8$; mw 429.31, N 22.84%; lt yel ppt, mp – decomp at 215–225° and burns very rapidly, leaving a black ash; insol in w, eth or benz; sol in hot alc, hot acet, concd nitric acid and in glac AcOH. It can be prepd by nitrating diphenylglycoluril with mixed nitric-sulfuric acid. It is stable at 135° and would not explode even with a No 8 detonator

It was considered by Shriner as of no promise as a military explosive (Ref 3)

Refs: 1) Beil **26**, 506 & (148) 2) Beil, not found (Trinitro deriv) 3) R.L. Shriner, OSRD Rept **2054**(1943), pp 10ff

Diphenylglyoxal, Diphenyldiketone, Dibenzoyl or **Benzil.** See Vol 2, p B64-R

N,N′-Diphenyl-guanidine, Melaniline or **N,N′-Diphenyl-iminourea.** See Bis(phenyl)-guanidine in Vol 2, p B154-L. It was proposed by E. Wydler [SwissP 144148(1929); CA **25**, 5179(1931)] to be used as a stabilizer in smokeless propellants

In the US the Thiokol Chemical Corp, Trenton, NJ proposed it as a curing accelerator in some of their "Polysulfide Composite Propellants". The composition and properties of these proplnts are given in conf Proplnt Manual **SPIA/M2** (1962) (Unit Nos 618, 619, 620, 647, 64 & 533)

Nitrated compds: N,N′-Bis(mononitrophenyl)-guanidine and N,N′-Bis(2,4,6-trinitrophenyl)-guanidine or Hexanitrophenylguanidine are described in Vol 2, p B154

N,N′-Diphenylhydrazine and Derivatives

N,N′-Diphenylhydrazine or *Hydrazobenzene,* $C_6H_5.NH.NH.C_6H_5$; mw 184.23, N 15.21%; rhom bipyramidal tablets (from alc + eth), mp 126–27°, sp gr 1.15 at 16°; sol in alc; sl sol in benz, water & cold acet ac; was prepd by hydrogenizing of nitrobenzene in the presence of Ni in aq NaOH under pressure at 110–120°(Ref 1)

It is used as a reagent for aldehydes and ketones; was proposed by Snelling & Wyler as a sensitizer for Ammonium Nitrate in expl mixts (Ref 7)

Dinitro-N,N′-diphenylhydrazine or **Dinitrohydrazobenzene,** $C_{12}H_{10}N_4O_4$; mw 274.23, N 20.43%. The *2,2′-Dinitro-* and *4,4′-Dinitro-* and *2,4-Dinitro* derivs are described in Beil (Ref 2)

Tetranitro-N,N′-diphenylhydrazine or **Tetranitrohydrazobenzene,** $C_{12}H_8N_6O_8$; mw 364.23, N 23.08%. Two isomers are found in Beil:

2,4,2′,4′-Tetranitrohydrazobenzene, $(O_2N)_2C_6H_3.NH.NH.C_6H_3(NO_2)_2$, yel crysts (from acet + alc), mp 250°; sol in glac acet ac, concd sulfuric acid with yel color & in NaOH with blue color; insol in benz; was prepd by treating a suspension of 2,4,2′,4′-Tetranitroazobenzene in hot w with hydrogen sulfide (Ref 3, p146)

2,4,6,4′-Tetranitrohydrazobenzene or *N-(4-nitrophenyl) -N′-(2,4,6-trinitrophenyl)-hydrazine,* $(O_2N)_3C_6H_2.NH.NH.C_6H_4.NO_2$, yel crysts (from a little eth acet + alc), mp 210–14°; readily sol in glac acet ac, eth acet, alc, acet, alkali & concd sulfuric acid; can be prepd by reaction of 4-nitrophenylhydrazine & Picrylchloride in the presence of Ca carbonate in hot alc (Refs 3 & 6)

No expl props of the tetranitro derivs are reported

2,4,6,2′,4′-Pentanitrohydrazobenzene or **N-(2,4-dinitrophenyl)-N′-(2,4,6-trinitrophenyl)-hydrazine,** $(O_2N)_3C_6H_2.NH.NH.C_6H_4.NO_2$; mw 409.23, N 23.96%; yel ndls (from Nitrobenz + alc), mp 225–28°(dec); sol in acet, glac acet ac, alkali & ethyl acet; less sol in alc, eth & benz; was prepd by reaction of Picrylchloride & 2,4-dinitrophenylhydrazine in the presence of Ca carbonate in hot alc (Refs 4 & 6)

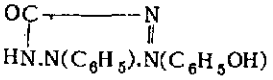
This compd is undoubtedly explosive

2,4,6,2′,4′,6′-Hexanitrohydrazobenzene; N,N′-Dipicrylhydrazine or N,N′-Bis-(2,4,6-trinitrophenyl)-hydrazine, $(O_2N)_3C_6H_2.NH.NH.C_6H_2(NO_2)_3$; mw 454.23, N 24.67%; lt-yel n dls (from glac acet ac), mp 201–02°; sl sol in eth, benz & petr eth with a yel color; somewhat more sol in alc, ethyl acet & acetone with a deep-red color. It was prepd by reaction of 1 mol hydrazine hydrate & 2 mols Picrylchloride in hot alc; or from reaction of equimolar mixt of Picrylhydrazine & Picrylchloride at 120°

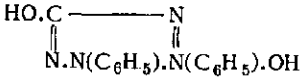
This compd forms a *Potassium salt*, $KC_{12}H_5N_8O_{12}$, green crysts having a metallic luster (from acet + alc), mp approx 188°, explodes on heating rapidly; sl sol in acet with a red color; v sl sol in alc; insol in w. It also forms a *Dipotassium salt*, $K_2C_{12}H_4N_8O_{12}$, grn pltlts, sol in acet & w with a blue to blue-violet color (Refs 4 & 5)

The expl props of the hexanitro deriv are reported by Blatt (Ref 8) as follows:
Power by Lead Block Expansion: 98–113% of PA
Reactivity: material is acid
Sensitivity to Impact: FI is 74% PA
Refs: 1) Beil **15**, 123, (28) & [52] 2) Beil **15**, 468, 489 & (127, 130) 3) Beil **15**, 494 & (146, 147) 4) Beil **15**, 494 & (147) 5) E. Grandmougin & H. Leemann, Ber **39**, 4384(1906) & Ber **41**, 1296(1908) 6) A.G. Green & F.M. Rowe, JChemSoc **101**, 2451 (1912) 7) W.O. Snelling, USP 1827675 (1931) & CA **26**, 601(1932) 8) A.H. Blatt, OSRD **2014**(1944) 9) CondChemDict (1961), 584-R (Hydrazobenzene)

2,3-Diphenyl-5-hydroxytetrazolium Hydroxide [called 2.3-Diphenyl-5-oxy-tetrazoliumhydroxyd or 2-Phenyl-tetrazolon-(5)-hydroxyphenylat-(3) in Ger]

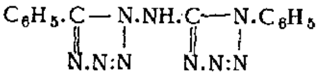


or



(See its Anhydride, **Diphenylcarbodiazone**, in this Vol)

1,5-Diphenyl-1′,5-iminobis(1,2,3,4-tetrazole) {called [1-Phenyl-tetrazolyl-(5)]-[5-phenyl-tetrazolyl-(1)]-amin in Ger}

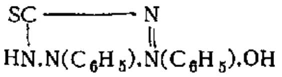


mw 305.30, N 41.29%; ndls (from alc), mp 192°(dec), deflgr on heating in a flame; insol in w; v sl sol in eth & petr eth; sl sol in alc, benz & chlf; mod sol in hot glac acet ac, toluene & xylene; stable in alkaline & mineral acid solns. It was prepd by heating bis(α-azidobenzylidene)-hydrazine with Na azide in alc & CO_2 stream. Its *Silver salt* is stable in light, sol in ammonium hydroxide, and deflgr on heating in a flame (Refs 1 & 2)
Refs: 1) Beil **26**, [344] 2) R. Stollé & A. Netz, Ber **55B**, 1301, 1303(1922) & CA **16**, 3899(1922)

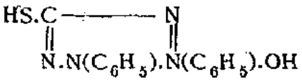
Diphenyl-iminourea. See Bis(phenyl)-guanidine in Vol 2, p B154-L

Diphenylketone. See Benzophenone in Vol 2, p B77-L

2,3-Diphenyl-5-mercaptotetrazolium Hydroxide,



or



(See its Anhydride, **Diphenylthiocarbodiazone**, in this Vol)

Diphenyl-methane and Derivatives

Diphenyl-methane, Methylene-diphenyl, Ditane or *Benzylbenzene*, $C_6H_5.CH_2.C_6H_5$; mw 168.23; col crysts, sp gr 1.001 at 26/4°, mp 26.6°, bp 265°; insol in w; v sol in alc & in eth. It can be prepd by condensation of benzylchloride with benzene in presence of Al chloride. Used in organic synthesis (Refs 1 & 10)

Azidodiphenylmethane, $(C_6H_5)_2CH.N_3$, mw 209.24, N 20.08%; liq, bp 135° at 4mm pres, n_D 1.5883 at 22°. It was obtd with other products when diphenylmethanol in chlf reacted with hydrazoic acid-sulfuric acid with subsequent addn of ice & warming (Ref 8)

No expl props are reported (Refs 7, 9, 11 & 12)

α,α-Diazidodiphenylmethane or **Benzophenone Diazide,** $C_6H_5.C(N_3)_2.C_6H_5$; mw 250.26, N 33.58%; crysts (from MeOH), mp 42°, vapor press 0.04mm at 80° & 0.10mm at 98°, sp gr 1.1568 at 50°, latent heat of vaporization 14.70kcal/mol, surface tension 37.34 dynes/cm at 50°; sensitivity to shock is only sl greater than that of PA; unchanged by boiling w or dil alkalies; reacts explosively with sulfuric acid even in the cold; sensitive to shock. It was prepd & isolated in 88% yield from reaction of dichlorodiphenylmethane & AgN_3 in ether soln (Refs 3, 4, 5 & 6)

See also **Benzophenonediazide** in Vol 2 of Encycl, p B77-R

Refs: 1) Beil **5**, 588, (277), [498] & {1785}; Beil **7**, [941] (Diphenylmethane) 2) Beil, not found (Azidodiphenylmethane) 3) Beil **5**, {1798} (Diazido deriv) 4) S. Götzky, Ber **64B**, 1555, 1558(1931) & CA **25**, 4543(1931) 5) G. Cronheim et al, ZPhysChem, Bodenstein-Festband **1931**, 785, 788 & CA **25**, 5847(1931) (Decomposes when exposed to X-rays) 6) G. Kornfeld & A. Stern, ZPhysChim **159A**, 40(1932) & CA **26**, 3172(1932) (Thermal decompn of Benzophenone Diazide) 7) I. Hechenbleikner, USP 2286169(1942) & CA **36**, 7224(1942) (Use of Azidodiphenylmethane in spray or dust against red spider) 8) C.L. Arcus & R.J. Mesley, JChemSoc **1953**, 181 & CA **47**, 11169(1953) (Prepn) 9) C.H. Gudmundsen & W.E. McEwen, JACS **79**, 332 (1957) & CA **51**, 9524(1957) (Prepn) 10) CondChemDict (1961), 407-R 11) R. Krecher & G. Jaeger, ZNaturforsch **19b** (7), 657(1964) & CA **61**, 10609(1964) (Decompn in benz & $AlCl_3$) 12) A.J. Papa, JOrgChem **31** (5), 1428(1966) & CA **64**, 19407(1966) (Prepn)

Diphenylmethane Hydroperoxide or **Benzohydryl Hydroperoxide,** $(C_6H_5)_2CH.OOH$; mw 200.23, O 15.98%; almost odorless ndls or columns (from benz + eth acet), mp 51°, bp – decomps at 135° (gas evolution), deflgr above 170°; even in absence of air & light it decomp in 6 weeks. It was obtd when diphenylmethane was shaken for 60 hrs with dry O at 65° under a Hg vapor lamp, and by other methods (See Refs)

It gives diphenylmethanol, mp 68°, almost quantitatively with Na_2SO_3 soln; and with 10% sulfuric acid, it is cleaved to phenol & benz (Ref 2)

Refs: 1) Beil, not found 2) H. Hock & S. Lang, Ber **77B**, 262(1944) & CA **39**, 3526 (1945) 3) W.T. Bishop et al, USP 2535344 (1950) & CA **45**, 2842(1951) (Use of Hydroperoxides for recovery of PbS 91.5, ZnO 97 & Fe 87% from ores, and reduction of ash content of coal to one-third) 4) F. Mashio & Y. Nakagawa, JChemSocJapan, IndChemSect **55**, 111–13(1952) & CA **48**, 6404(1954) (Formation of Ph_2CO as a thermal decompn product of Ph_2CHOOH) 5) A.G. Davis et al, JChemSoc **1953**, 1541 & CA **48**, 7537 (1954) (Prepn) 6) Stamicarbon N.V., DutchP 76525(1954) & CA **50**, 5028(1956); BritP 748565–6(1956) & CA **50**, 10771 (1956) (Prepn by decompn of the hydroperoxide) 7) M. Bassey et al, JChemSoc **1955**, 2471 & CA **50**, 4084(1956) (Prepn, isotraces studies on formation & decompn) 8) A.G. Davis & R. Feld, JChemSoc **1956**, 4669 & CA **51**, 4300(1957) (Prepn) 9) R. Hüttel & H. Ross, ChemBer **89**, 2644(1956) & CA **51**, 8679(1957) (Prepn by oxidation of Ph_2CHCl at 0–5° with 80–90% H_2O_2) 10) W.E. Weesner, USP 2792424 & 2792426 (1957) & CA **51**, 15572–73(1957) (Production of the hydroperoxide using α-aryl ketones or β-diketones as oxidation initiators) 11) J.I.G. Cadogan et al, JChemSoc **1958**, 4498 & CA **53**, 15003(1959) (Prepn) 12) Ruhrchemie Akt-Ges, GerP 1041960 (1958) & CA **55**, 11461(1961) (Prepn by oxidizing Ph_2CH_2 with Mg phthalocyanine at 105° in the presence of O for 10 hrs) 13) D.B. Sharp & B.S. Wildi, USP 2883427 (1959) & CA **53**, 17062(1959) (Prepn from Ph_2CH_2 & O at 140–80° for <3 hrs) 14) H. Hock & H. Kropf, JPraktChem **9**, 173(1959) & CA **54**, 7626(1960)(Prepn by autoxidation of Ph_2CH_2 using metal derivs phthalocyanines) 14a) F. Masuo & S. Kato, USP 2955996 (1960) & CA **55**, 8275 (1961) (Prepn from Ph_2CH_2 by treating with 1–10% TiO_2 & O at 25–130°) 15) F. Masuo & S. Kato, GerP 1129486(1962) & CA **58**, 7887(1963) (Same as Ref 14a)

Mononitrodiphenylmethane, $C_{13}H_{11}NO_2$; mw 213.23, N 6.57%. The following derivs are found in Beil:

a-Nitrodiphenylmethane, $(C_6H_5)_2CH.NO_2$, colorless oil, fr p – below –15°, sp gr 1.1727 at 20°(Ref 1)

a-aci-Nitrodiphenylmethane, $(C_6H_5)_2C{:}NO_2H$, prisms (from eth), mp 90°(dec), unstable even at RT (Ref 2)

2-Nitrodiphenylmethane, $C_6H_5.CH_2.C_6H_4.NO_2$, lt-yel liq, bp 183–84° at 10mm press (Ref 3)

3-Nitrodiphenylmethane, liq, not volatile on steam bath (Ref 4)

4-Nitrodiphenylmethane, long ndls (from petr eth), mp 31°, bp 202° at 11mm press, sp gr 1.278 at 0°(Ref 5)

Refs: 1) Beil **5**, 594 2) Beil **5**, 594, (279) & {1796} 3) Beil **5**, 593, (279), [503] & {1796} 4) Beil **5**, 593 5) Beil **5**, 593 & [503]

Dinitrodiphenylmethane, $C_{13}H_{10}N_2O_4$, mw 258.23, N 10.85%. Several isomers are known:

a,a-Dinitrodiphenylmethane, $(C_6H_5)_2C(NO_2)_2$, pltlts (from dil alc), mp 78–78.5°; readily sol in chlf, eth, benz & hot alc (Ref 1)

2,2'-Dinitrodiphenylmethane, $O_2N.C_6H_4.CH_2.C_6H_4.NO_2$, pltlts or ndls (from w), mp 83.5°; sl sol in alc & eth; insol in petr eth (Ref 2)

3,3'-Dinitrodiphenylmethane, pltlts (from glac acet ac), mp 174°; sol in alc, hot benz & glac acet ac; sl sol in eth (Ref 3)

4,4'-Dinitrodiphenylmethane, crysts (from petr eth or glac acet ac), mp 184–189.4°; sol in hot benz & glac acet ac; sl sol in eth; insol in cold alc (Ref 4)

2,4'-Dinitrodiphenylmethane, yel prisms (from benz), mp 118°; sol in benz; sl sol in eth; v sl sol in alc (Ref 4)

3,4'-Dinitrodiphenylmethane, ndls (from alc), mp 102–04°(Ref 4)

2,4-Dinitrodiphenylmethane, $C_6H_5.CH_2.C_6H_3(NO_2)_2$, not obtd pure (Ref 5)

3,5-Dinitrodiphenylmethane, ndls (from dil alc), mp 183–85°; sol in alc, acet & benz (Ref 6)

Other props & methods of prepn are found in Refs

Refs: 1) Beil **5**, 596 & {1797} 2) Beil **5**, 596 & {1796} 3) Beil **5**, 595, (280), [503] & {1797} 4) Beil **5**, 595 & {1797} 5) Beil **5**, {1796} 6) Beil **5**, [503] & {1797} 7) J.D. Margerum et al, JPhysChem **66**, 2435, 2438(1962) & CA **58**, 4082(1963)

Trinitrodiphenylmethane, $C_{13}H_9N_3O_6$; mw 303.23, N 13.86%. Three isomers are described in the literature:

x,x,x-Trinitrodiphenylmethane, $C_{13}H_9(NO_2)_3$, yel ndls (from alc), mp 109–10°; v sl sol in benz; was prepd by nitrating diphenylmethane with 6–7 parts of nitric acid (sp gr 1.53) at 50°(Ref 1)

2,4,2'-Trinitrodiphenylmethane, $(O_2N)_2C_6H_3.CH_2.C_6H_4(NO_2)$, crysts (from alc), mp 111–13°; was obtd when 2,2'-dinitrodiphenylmethane in concd sulfuric acid was nitrated with Na nitrate in concd sulfuric acid at 70° for 1.5 hrs (Ref 3)

2,4,4'-Trinitrodiphenylmethane, crysts (from alc), mp 107–10°; was prepd by nitrating 4,4'-dinitrodiphenylmethane in concd sulfuric acid with K nitrate in concd sulfuric at 70° for 1.5 hrs. This synthesis is reported to establish the structure as that of the x,x,x-Trinitro deriv prepd by Staedel (Ref 3)

No expl props are reported
Refs: 1) Beil **5**, 596 2) W. Staedel, Ann **283**, 155(1894) 3) J.D. Margerum et al, JChemPhys **66**, 2435, 2438(1962) & CA **58**, 4082(1963) 4) A.L. Blum et al, JOrgChem **29**, 636, 639(1964) & CA **60**, 10501(1964) (Prepn of 2,4,4'-deriv by decarboxylation of 2,4,4'-trinitrodiphenylacetic acid; activation energy & UV spectrum are reported)

Di(or Bis)-(2,4-dinitrophenyl)-methane or **2,4,2',4'-Tetranitro-diphenylmethane,** $(O_2N)_2C_6H_3.CH_2.C_6H_3(NO_2)_2$, mw 348.23, N 16.09%; crysts (from glac acet acid or benz), mp 173–81°. It was prepd by nitrating diphenylmethane with nitric acid (d 1.53) & concd sulfuric acid (Ref 1) and by decarboxylation of ethyl bis(2,4-dinitrophenyl-acetic acid (Ref 2). No expl props are reported
Refs: 1) Beil **5**, 596, [503] & {1798} 2) A.L. Blum et al, JOrgChem **29**, 636, 638, 639(1964) & CA **60**, 10501(1964)

2,4,6,2',4'-Pentanitrodiphenylmethane, $(O_2N)_3C_6H_2.CH_2.C_6H_3(NO_2)_2$; mw 393.23, N 17.81%; lt-yel tablets (from glac acet ac), mp 200°. It was prepd by nitrating the tetranitro deriv (above) with nitric acid (sp gr 1.5) & fuming sulfuric acid at 100° (Refs 1 & 2). No expl props are reported
Refs: 1) Beil **5**, {1798} 2) G.D. Parkes & R.H.H. Morley, JChemSoc **1936**, 1478 & CA **31**, 386(1937) 3) Not found in later refs thru 1966

Hexanitrodiphenylmethane, $(O_2N)_3C_6H_2.CH_2.C_6H_2(NO_2)_3$, not found in the literature thru 1966

Diphenylmethanol and Derivatives

Diphenylmethanol, Diphenylmethylol, Benzhydrol or *Diphenylcarbinol,* $(C_6H_5)_2CHOH$, mw 184.23; ndls, mp 68–69°, bp 298.5°; sl sol in w; v sol in alc, eth, chlf & CCl_4; insol in ligroin; can be prepd by reduction of benzophenone $(C_6H_5)_2CO$ with Mg or Zn dust and by other methods (Refs 1 & 2)
Refs: 1) Beil **6**, 678, (325), [631] & {3364} 2) CondChemDict (1961), p 138-R

Azido, $C_{13}H_{11}N_3O$, and *Diazido,* $C_{13}H_{10}N_6O$, derivs were not found in the literature thru 1966

4-Nitrosodiphenylmethanol (called 4-Nitroso-α-phenylbenzyl Alcohol in CA), $ON.C_6H_4.CH(OH).C_6H_5$; mw 213.23, N 6.57%; yel ndls, mp 114–17°; was obtd with 4,4'-dibenzoylazoxybenzene when β-(p-nitrodiphenylmethylthio)propionic acid was boiled with 5% NaOH for 1 min only, cooling on ice, diluting with ice-water, and separating the two fractions (Ref 2)
Refs: 1) Beil, not found 2) Y. Iskander & I. Tewfik, JChemSoc **1961**, 2400 & CA **55**, 25847(1961)

Diphenylmethylnitrite, $C_6H_5.CH(ONO).C_6H_5$; mw 213.23, N 6.57%; yel liq, bp – decompd on distillation even at 65° and 0.1mm press. It was prepd by using equimolar quantities of diphenylmethanol and pyridine in dry ether, and adding nitrosyl chloride until in sl excess; pyridine hydrochloride was filtered off, and ether removed by evaporation (Refs 2 & 3)
Refs: 1) Beil, not found 2) A.D. Allen, Nature **172**, 301(1953) & CA **48**, 3124(1954) 3) A.D. Allen, JChemSoc **1954**, 1972 & CA **48**, 9162(1954)

Diphenylmethyl Nitrate, $C_6H_5.CH(ONO_2).C_6H_5$; mw 229.23, N 6.11%; highly refracting, colorless crysts (from petr eth), mp 36.7–37.2°, bp 40–60°, unstable at RT & slowly decomp with evolution of brn fumes; on heating to 140–50° it smoothly decompd to benzophenone; its soln in methyl cyanide was hydrolyzed to diphenylmethanol by shaking with saturated bicarbonate soln for several hrs. It is too unstable to be isolated by ordinary methods, but can be obtd in soln, by heating under reflux diphenylmethyl chloride & an excess of powdered Ag nitrate in dry ether. The ethereal soln was evaporated in a vacuum (See Refs)

Refs: 1) Beil, not found 2) G.W.H. Cheeseman, Chem & Ind **1954**, 281 & CA **49**, 3910 (1955) 3) J.W. Baker & T.G. Heggs, Chem & Ind **1954**, 464 & CA **49**, 6872(1955) 4) R.T. Merrow & R.H. Boschan, JACS **76**, 4622(1954) & CA **49**, 13182(1955) 5) J.W. Baker & T.G. Heggs, JChemSoc **1955**, 616 & CA **50**, 7081(1956) (Solvolytic & alk hydrolytic decompn studies in aq alc at several temps) 6) G.W.H. Cheeseman, Chem & Ind **1956**, 1390 & CA **51**, 8054(1957) (Reactions with some basic reagents) 7) Ibid, **1957**, 115–23 & CA **51**, 9621–22 (1957) (Prepn & reactions of Benzhydrol Nitrate)

Mononitrodiphenylmethanol or **Mononitrobenzhydrol**, $C_6H_5.CH(OH).C_6H_4.NO_2$, mw 229.23, N 6.11%. Three isomers are known:
2-Nitrodiphenylmethanol, pltlts (from petr eth), mp 59–60° & 63–64° (Ref 6), bp 170–80° at 0.74mm press (Refs 2, 3, 7 & 8)
3-Nitrodiphenylmethanol, crysts, mp 68–71°, bp 208–12° at 6mm press (Refs 1, 2 & 6)
4-Nitrodiphenylmethanol, yel crysts (from benzin or benzol), mp 73–78° (Refs 1, 4, 5 & 6)

Methods of prepn & other props are found in Refs

Refs: 1) Beil **6**, {3378} 2) M.S. Newman & A.S. Smith, JOrgChem **13**, 592, 596(1948) & CA **43**, 565(1949) 3) D.H. Hey & R.D. Mulley, JChemSoc **1952**, 2276 & CA **47**, 1090(1953) 4) B.B. Smith & J.E. Leffler, JACS **77**, 2509(1955) & CA **50**, 3289(1956) 5) B.B. Stewart & H.A. Smith, JACS **77**, 5457(1957) & CA **52**, 4569(1958) 6) R.T. Puckowski & W.A. Ross, JChemSoc **1959**, 3555 & CA **54**, 8717–18(1960) 7) W.B. Dickinson, JACS **86**, 3580(1964) & CA **61**, 11915(1964) (Novel reactions) 8) A. Silberg & Z. Frenkel, RevRoumaineChim **10**, 1035(1965) (Fr); StudiiCercetariChim **13**, 1071(1965) (Rom) & CA **64**, 12641(1966) (Prepn & reactions)

Dinitrodiphenylmethanol or **Dinitrobenzhydrol**, $O_2N.C_6H_4.CH(OH).C_6H_4.NO_2$; mw 274.23, N 10.22%. Three isomers are known:
2,2′-Dinitrodiphenylmethanol or *Bis-(o-nitrophenyl)-methanol*, crysts (from cyclohexane + ether), mp 126°; was obtd when, to a stirred soln of 2,2′-dinitrobenzophenone in tetrahydrofuran at −35°, a soln of LiAl hydride in tetrahydrofuran was added dropwise during 15 mins. The soln was warmed to RT and neutralized with dil sulfuric acid and stored overnight. It was filtered, concd, and extracted with ether. The dried extract was chromatographed and the solid from the second fraction was sublimed at 35° & 10^{-3}mm press and crystallized (Ref 8)
3,3′-Dinitrodiphenylmethanol or *3,3′-Dinitrobenzhydrol*, wh crysts (from 60% MeOH), mp 106.5–107.6°; was obtd by reducing 3,3′-dinitrobenzophenone using Al isopropoxide or using 2-propanol & its Al salt (Refs 2a &8)
4,4′-Dinitrodiphenylmethanol or *4,4′-Dinitrobenzhydrol*, crysts (from benz), mp 168–69°; was obtd by reducing 4,4′-dinitrobenzphenone in toluene + iso-propylalcohol with Al-isopropyl alc under reflux for 10 hrs and addg 20% sulfuric acid to the mixt (Refs 2a, 3, 4 5, 6 & 7)

This compd was also prepd by nitrating benzhydrol with concd nitric & sulfuric acids, and treating the resulting product with NaOH in an org solvent to prep 4,4′-dinitrobenzophenone (Ref 9)

4,4′-Dinitrodiphenylmethyl Nitrate or *4,4′-Dinitrobenzhydrol Nitrate*, $O_2N.C_6H_4.CH(ONO_2).C_6H_4.NO_2$; mw 319.23, N 13.16%; wh ndls (from alc or glac acet ac), mp 160.5–162°; was prepd by adding portionwise benzhydrol to white fuming nitric acid held at 28–30°. The dk-red soln was allowed to stand 5 hrs, then poured into chopped ice & water. The mixt was filtered and the crude product recrystallized 4 times from glac acet ac and an addnl time from a large vol of alcohol (Ref 3a)

Refs: 1) Beil, not found (Dinitro derivs) 2) D.Y. Curtin & S. Leskowitz, JACS **73**, 2632(1951) & CA **46**, 2527(1952) 2a) N.C. Deno & A. Schriesheim, JACS **77**, 3052(1955) & CA **50**, 32(1956) 3) J.H. Gorvin, JChemSoc **1955**, 88 & CA **50**, 902–03(1956) 3a) I.

Moyer et al, JOrgChem **20**, 71(1955) & CA **49**, 13181(1955) 4) B.B. Stewart & H.A. Smith, JACS **79**, 5459(1957) & CA **52**, 4569 (1958) 5) R.T. Puckowski & W.A. Ross, JChemSoc **1959**, 3560 & CA **54**, 8718(1960) 6) Y. Iskander & I. Tewfik, JChemSoc **1961**, 2393 & CA **55**, 25847(1961) 7) E.F. Pratt & J.F. Van De Castle, JOrgChem **26**, 2973 (1961) 8) R.B. Jones & K.R. Markham, JChemSoc **1962**, 3716–17(1962) & CA **57**, 12493(1962) (Not found in abstract) 9) P.M. Kocherigin & G.A. Karpov, RussP 165433(1964) & CA **62**, 5231(1965)

Trinitro, $C_{13}H_9N_3O_7$, and *Tetranitro*, $C_{13}H_8N_4O_9$, derivs of Diphenylmethanol were not found in the literature thru 1966

Diphenyl-methylamine and Derivatives

Diphenyl-methylamine or *N-Methyl-diphenylamine*, $C_6H_5.N(CH_3).C_6H_5$; mw 183.24, colorless liq, sp gr 1.048 at 20°/4, fr p minus 7.6°, bp 295–96°; insol in w; sol in alc & eth (Ref 1)

2,4-Dinitrodiphenyl-methylamine, $C_6H_5.N(CH_3).C_6H_3(NO_2)_2$; mw 273.24, N 15.38%, mp 166–67°, d 1.406 at 10°; can be prepd by condensation of N-methylaniline with 2,4-dinitrochlorobenzene,

$C_6H_5.N\boxed{H}(CH_3)+\boxed{Cl.}C_6H_3(NO_2)_2$

It gives on nitration Hexanitrodiphenylmethylamine

2,4,6,2′,4′,6′-Hexanitrodiphenyl-methylamine; Dipicryl-methylamine or **Bis(2,4,6-trinitrophenyl)-methylamine,** $(O_2N)_3C_6H_2.N(CH_3).C_6H_2(NO_2)_3$; mw 453.24, N 21.63%, OB to CO_2 minus 123.5%; crysts, mp 236–37°; can be prepd by nitration of 2,4-dinitrodiphenyl-methylamine, or by condensing 2,4-dinitrochlorobenzene with monomethylaniline and nitrating the product (Ref 3)

Its expl props listed in Blatt (Ref 4) are as follows:

Impact Sensitivity – FI 92% Picric Acid

Power by Trauzl Test – 87% Picric Acid

Refs: 1) Beil **12**, 180, (166) & [105] 2) Beil **12**, 752, (362) & [409] (Dinitro) 3) Beil **12**, 766 (Hexanitro) 4) A.H. Blatt, OSRD Rept **2014**(1944) (Hexanitro)

Diphenylmethylenediamine and Derivatives

Diphenylmethylenediamine or *Methylenedianiline* $(C_6H_5.NH)_2CH_2$, mw 198.26, N 14.13%; pltlts, mp 65°, bp 208–09° (decomp); insol in w & petr eth; sol in alc or eth; can be prepd by heating aniline with methylene iodide in the presence of K_2CO_3 (Ref 1)

Its isomer:

4,4′-Methylenedianiline, $H_2N.C_6H_4.CH_2.C_6H_4.NH_2$, mw 198.26, N 14.13%; yel to lt brn crysts; mp 89° (min); almost insol in w & CCl_4; sol in acet & methanol; can be prepd by heating aniline & aniline HCl in w at 95° (Refs 2 & 4)

Dinitrodiphenylmethylenediamine or **Bis-(nitroanilino)-methane.** $O_2N.C_6H_4.NH.CH_2.NH.C_6H_4.NO_2$; mw 288.26, N 19.44%. Three isomers are known:

Bis (2-nitro-anilino)-methane, golden yel ndls (from alc), mp 195°; sol in eth; insol in cold alc & w; was prepd by treating a hot soln of 2-nitroaniline in 5–10% alc with an excess of 40% formaldehyde (Ref 3, p 690)

Bis (3-nitro-anilino)-methane, orn-yel ndls (from alc), mp 213°; almost insol in alc; insol in w, ether, chlf & benz. It was prepd by addg a 40% formaldehyde soln to a hot concd soln of 3-nitroaniline in alc. It forms a *Picrate salt*, $C_{13}H_{12}N_4O_4+2C_6H_3N_3O_7$, yel ndls, mp 120° (Ref 3, p 702)

Bis (4-nitro-anilino)-methane, citron-yel ndls, mp 232°; insol in w & eth; v sl sol in hot alc; was obtd by reacting formaldehyde with 4-nitroaniline in alc (Ref 3, p 717)

Refs: 1) Beil **12**, 184, (168) & [110] 2) Beil **12**, 185 & [110] 3) Beil **12**, 690, 702, 717 4) CondChemDict (1961), 739-L (4,4′-Methylenedianiline)

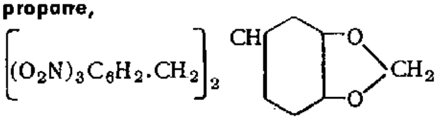
Tetranitrodiphenylmethylenediamine, $C_{13}H_{10}N_6O_8$, not found in the literature thru 1966

Hexanitrodiphenylmethylenediamine, $C_{13}H_8N_8O_{12}$, not found in the literature thru 1966

Diphenylmethylenedihydroxyphenylpropane and Derivatives

Diphenylmethylenedihydroxyphenylpropane, $C_{22}H_{20}O_2$, mw 316.38; may be considered as the parent compd of its Dipicryl deriv, although not used to prep it:

1,3-Dipicryl-2-(3,4-methylenedihydroxyphenyl)-propane,



mw 586.38, N 14.33%; yel crysts (from glac acet acid), mp 137–40°(dec). It was prepd from reaction of 1 mol piperonal & 2 mols TNT in the presence of piperizine or piperidine in pyridine; in addn to a little 2,4,6-trinitro-3′,4′-methylenedihydroxystilbene (Refs 1 & 2)

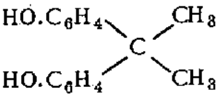
Refs: 1) Beil **19**, [48] 2) I.A. Pastak, BullSocChim (France) **39**, 81(1926) & CA **20**, 3000(1926)

Diphenylnitrosamine. See under Diphenylamine and Derivatives

Diphenyl-octatetrayne. See Diphenyl-tetraacetylene

Di(or Bis)-(phenylol)-dimethyl-methane and Derivatives

Di(phenylol)-dimethyl-methane or *Di(hydroxyphenyl)-dimethyl-methane*,

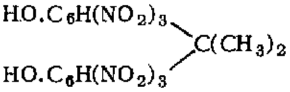


$$(HO.C_6H_4)_2C(CH_3)_2$$

mw 228.28; O 14.02%. This compd and its *Dinitro*, $C_{15}H_{14}N_2O_6$, and *Tetranitro*, $C_{15}H_{12}N_4O_{10}$, derivs are described in Vol 2 of Encycl, p B147-R under Bis (hydroxyphenyl)-propane. The structure of these derivs has been established as 2,2-Bis-(3-nitro-4-hydroxyphenyl)-propane (Ref 2) and 2,2-Bis-(3,5-dinitro-4-hydroxyphenyl)-propane (Ref 3). See Ref 4

Refs: 1) Beil **6**, 1011, (493), [978] & {5459} (Parent compd) 2) Beil **6**, {5462} (Dinitro deriv) 3) Ibid, (Tetranitro deriv) 4) J. Hackel et al, BiulWojskowejAkadTech **10**, No 108, 81(1961) & CA **57**, 11074(1962) [Prepd 1,3-Bis-(2,4-dinitrophenoxy)-propane, mp 185°, ignition temp 440° for 5 sec & 402° for 10 sec, Trauzl Pb block test 29 vs 100 for PA, insensitive to flame]

1,3-Bis (2,4,6-trinitrophenoxy)-propane; 1,3-(2,4,6,2′,4′,6′-Hexanitrodiphenoxy)-propane or **1,3-Bis (picrylhydroxy)-propane,**



$$(HO.C_6H(NO_2)_3)_2C(CH_3)_2$$

mw 498.28, N 16.87%; mp 145°; ignition temp 365° for 5 sec & 327° for 10 sec; Trauzl Pb block test 88 vs 100 for PA; insensitive to flame. It was prepd by nitrating the tetranitro deriv with 73.5/24.5-nitric acid/sulfuric acid (250% excess) at 20–50° and 1.5 hrs at 85°(Ref 2)

Refs: 1) Beil, not found 2) J. Hackel et al, BiulWojskowejAkadTech **10**, No 108, 81(1961) & CA **57**, 11074(1962)

NOTE: The above 1,3-Bis (2,4-dinitrophenoxy)-propane and 1,3-Bis (2,4,6-trinitrophenoxy)-propane were not known at the time of publication of Vol 2 of Encycl

Di(phenylol)-propane. See Bis(hydroxyphenyl)-propane in Vol 2, p B147-R

Di(phenylol)-sulfone. See Bis(hydroxyphenyl)-sulfone in Vol 2, p B148-L

Diphenyloxide. See Diphenylether

Diphenyloxamide and Derivatives

N,N′-Diphenyloxamide, $C_6H_5.NH.CO.CO.NH.C_6H_5$; mw 240.25, N 11.66%; pltlts (from benz or Nitrobenz), mp 247–54°; insol in cold alc, hot w & eth; sl sol in hot alc; sol in benz(Ref 1)

N,N′-Dinitrodiphenyloxamide; N,N′-Bis(nitrophenyl)-oxamide or **Dinitroöxanilide,** $O_2N.C_6H_4.NH.CO.CO.NH.C_6H_4.NO_2$; mw 330.25, N 16.97%. Three isomers are known:
N,N′-Bis (2-nitrophenyl)-oxamide, yel plates (from aniline), mp 331°(dec); insol in w, alc, eth, CS_2, benz & acet; sl sol in glac acet ac; sol in hot aniline (Ref 2)
N,N′-Bis (3-nitrophenyl)-oxamide, almost colorless ndls (from aniline), mp 309–10°(dec) & orn-colored rhombic crysts – mp 307°(dec); sl sol in alc, eth & benz; sol in aniline (Ref 3)
N,N′-Bis (4-nitrophenyl)-oxamide, yel ndls (from aniline), mp 358–59°(dec); v sl sol in hot alc, benz & $CHCl_2$; sl sol in hot glac acet ac

Methods of prepn & other props are found in Beil

N,N′-Bis-(2,4-dinitrophenyl)-oxamide or **2,4,2′,4′-Tetranitroöxanilide,**
$CO.NH.C_6H_3(NO_2)_2$
|
$CO.NH.C_6H_3(NO_2)_2$; mw 420.25, N 20.00%; yel ndls (from Nitrobenz), mp 306°(dec); sl sol in alc, benz & glac acet ac. It was prepd by reacting 2,4-dinitroaniline & oxalylchloride in boiling benz (Refs 5 & 8)

Pentanitrodiphenyloxamide, $C_{14}H_7N_7O_{12}$, not found in the literature thru 1966

2,4,6,2′,4′,6′-Hexanitrodiphenyloxamide; 2,4,6,2′,4′,6′-Hexanitroöxanilide or **N,N′-Dipicryloxamide,** $CO.NH.C_6H_2(NO_2)_3$
|
$CO.NH.C_6H_2(NO_2)_3$; mw 510.25, N 21.96%; crysts, mp 295–300°(dec); sl sol in hot alc & benz; very low expln temp. It was prepd by nitrating oxanilide. Its power & brisance are comparable with those values of TNT; Pb block expansion test – 70% Picric Acid. It is reported to be chemically unstable or reactive with moisture (Refs 6, 7, 8 & 9)
Refs: 1) Beil **12**, 284, (207) & [165] 2) Beil **12**, 693 & (342) 3) Beil **12**, 705 & (347) 4) Beil **12**, 721 & (353) 5) Beil **12**, 755 & (363) 6) Beil **12**, 767 7) SA d'Explosifs(Seine), FrP 391106(1910) & CA **4**, 2733 (1910) 8) J.T. Bomwater, Rec **31**, 118(1912) & CA **6**, 2743(1912) 9) Blatt, OSRD **2014** (1944) 10) K.K. Andreev & Y.A. Terebilina, "TeoriyaVzryvchatykhVeshchestv," SbStatei **1963**, 53 & CA **59**, 12585(1963) (Impact tester is described for detg relative sensitivity to mechanical shock by impact of a 10-kg wt falling 25cm on 2-, 10- & 50-mg samples. Relative sensitivity in terms of % explns and rebound of wt as a % of its fall are reported)

Di(phenyloxy)-ethylene or **Ethyleneglycol-diphenyl-Ether.** This compd and its *Dinitro,* $C_{14}H_{12}N_2O_6$, *Tetranitro,* $C_{14}H_{10}N_4O_{10}$, and *Hexanitro,* which should read:
$CH_2.O.C_6H_2(NO_2)_3$
|
$CH_2.O.C_6H_2(NO_2)_3$, derivs are described in Vol 2 of Encycl, p B153-R to B154-L under Bis(phenyl)-ethyleneglycol Ether and Derivatives

Their addnl refs are as follows:
Refs: 1) Beil **6**, {568}(Parent) 2) Beil **6**, {802, 823}(Dinitro derivs) 3) Beil **6**, {864} (Tetranitro deriv) 4) (?) Lippmann, CR **68**, 1269(1869) (Was first to prepare the parent compd by heating K phenolate with 1,2-dibromoethane on a water bath) 5) (?) Weddige, JPraktChem [2] **21**, 127(1880) & **27**, 201(1883) [Prepn of o-, m-, and p-dinitrocompounds, corresponding to formula : $(O_2N)C_6H_4.O.CH_2-CH_2.O.C_6H_4(NO_2)$ by interaction of ethylenebromide on two molecules of alkaline salts of corresponding mononitrophenols]

Di{[(phenyl-phenylamino)-ethyl] amino}-ethane. See 1,2-Bis{[(phenyl-phenylamino)-ethyl]-amino}-ethane in Vol 2, p B154-R

Diphenylphenylenediamine and Derivatives
N,N′-Diphenylphenylenediamine, $C_6H_5.NH.C_6H_4.NH.C_6H_5$; mw 260.32, N 10.76%. The N,N′-Diphenyl-o- (Ref 1); N,N′-Diphenyl-m- (Ref 2); and N,N′-Diphenyl-p-phenylenediamine (Ref 3) derivs are known
Refs: 1) Beil **13**, [13] 2) Beil **13**, 42 & [26] 3) Beil **13**, 80, (23) & [41]

Azido, $C_{18}H_{15}N_5$, and *Diazido*, $C_{18}H_{14}N_8$, derivs are not found in Beil

Dinitro-N,N′-diphenylphenylenediamine, $C_{18}H_{14}N_4O_4$, mw 350.32, N 16.00%. Several isomers are known:
N-Phenyl-N′-(2,4-dinitrophenyl)-o-phenylenediamine, $C_6H_5.NH.C_6H_4.NH.C_6H_3(NO_2)_2$, red crysts (from glac acet ac), mp 170–71°; almost insol in alc & eth (Refs 1 & 4)
N-Phenyl-N′-(2,6-dinitrophenyl)-o-phenylenediamine, resembling a lacquer (Refs 2 & 7)
4,6-Dinitro-N,N′-diphenyl-1,3-phenylenediamine, $C_6H_5.NH.C_6H_2(NO_2)_2.NH.C_6H_5$, red prisms (from alc), mp 186° (Refs 3 & 5)
N,N′-Bis (2-nitrophenyl)-p-phenylenediamine or *1,4-Bis (2-nitroanilino)-benzene*, $O_2N.C_6H_4.NH.C_6H_4.NH.C_6H_4.NO_2$, red prisms (from Nitrobenz), mp 233° (Refs 4 & 6)

Other props & methods of prepn are found in Refs

Refs: 1) Beil **13**, 18 2) Beil **13**, [13] 3) Beil **13**, 59 4) Beil **13**, [41] 4) F. Kehrmann & (?). Messinger, JPraktChem [2] **46**, 572(1896) 5) Jackson & Cohoe, AmChemJ **26**, 4(1901) 6) B.L. Manjunath, JIndianChemSoc **4**, 277(1917) & CA **21**, 3199(1927) 7) F. Kehrmann & I. Effront, Helv **4**, 520(1921) & CA **15**, 3449(1921)

Trinitro-N,N′-diphenylphenylenediamine, $C_{18}H_{13}N_5O_6$; mw 395.32, N 17.72%. Two isomers are found in Beil:
N-Phenyl-N′-(2,4,6-trinitrophenyl)-o-phenylenediamine or *N-Phenyl-N′-picryl-o-phenylenediamine*, $C_6H_5.NH.C_6H_4.NH.C_6H_2(NO_2)_3$, garnet-red pltlts (from alc), mp – decomp on heating to 120° or by heating with alc NaOH at 100°. It was prepd from reaction of picrylchloride & N-phenyl-o-phenylenediamine (Refs 1 & 3)
N,N′-Diphenyl-2,4,6-trinitro-1,3-phenylenediamine or *2,4,6-Trinitro-1,3-dianilinobenzene*, $C_6H_5.NH.C_6H(NO_2)_3.NH.C_6H_5$, red ndls (from glac acet ac), mp 217°. It was prepd by heating aniline with N-nitro-N,N′,N′-trimethyl-2,4,6-trinitro-1,3-phenylenediamine in alc, with N-nitro-N-methyl-N′-phenyl-2,4,6-trinitro-1,3-phenylenediamine in benz, or with 2,4,6-trinitro-1,3-bis-(methylnitramino-benzene) in benz (Refs 2 & 4)

Refs: 1) Beil **13**, 18 2) Beil **13**, (17) 3) F. Kehrmann & (?). Kramer, Ber **33**, 3074, 3075(1900) 4) C.F. van Duin, Rec **38**, 93 (1919)

Tetranitrodiphenylphenylenediamine, $C_{18}H_{12}N_6O_8$; mw 440.32, N 19.09%:
N-Phenyl-N′-picryl-4-nitro-1,2-phenylenediamine or **5,2′,4′,6′-Tetranitro-2-anilinodiphenylamine,** $C_6H_5.NH.C_6H_3(NO_2).NH.C_6H_2(NO_2)_3$, brn-yel crysts (from glac acet ac), mp – dec on heating above 174°; sl sol in alc; insol in w; obtd by heating picrylchloride with 4-nitro-2-aminodiphenylamine in alc (Refs 1 & 2)
N,N′-Bis(2,4-dinitrophenyl)-1,4-phenylenediamine, $(O_2N)_2C_6H_3.NH.C_6H_4.NH.C_6H_3(NO_2)_2$, crysts (from MeOH or acet), mp 289°. It was prepd by warming a mixt of p-phenylenediamine in acet with aq $NaHCO_3$ & 2,4-dinitrofluorobenzene, cooling & acidifying with AcOH (Ref 4)
N,N′-Bis(2,5-dinitrophenyl)-1,4-phenylenediamine, brn-red crysts (from Nitrobenz), mp 288–288.5°. It was obtd when a mixt of 2,5-dinitro-4′-aminodiphenylamine & bromo-p-dinitrobenzene, intimately mixed by grinding was heated for 2 hrs at 155–60°, and extracting the product by refluxing with 95% alc (Ref 3)

The tetranitro derivs are probably expl

Refs: 1) Beil **13**, (10) 2) F. Kehrmann et al, Ber **44**, 2623(1911) 3) C.K. Sunde & W.M. Lauer, JOrgChem **17**, 611(1952) & CA **47**,1642(1953) 4) H. Bräuniger & K. Spangenberg, Pharmazie **12**, 335(1957) (Ger) & CA **52**, 11764(1958); CA **54**, 22668(1960)

Pentanitro-N,N′-diphenylphenylenediamine, $C_{18}H_{11}N_7O_{10}$, not found in the literature thru 1966

Hexanitro-N,N′-diphenylphenylenediamine; N,N′-Dipicryl-1,4-phenylenediamine; or N,N′-Bis(2,4,6-trinitrophenyl)-p-phenylenediamine, $(O_2N)_2C_6H_2.NH.C_6H_4.NH.C_6H_2(NO_2)_2$

mw 530.33, N 21.13%; red crysts (from Nitrobenz), mp 320°; insol in common solvs; sol in aq alkalies. It was prepd by heating p-phenylenediamine & picrylchloride in toluene, in the presence of Na acetate; and by reaction of picrylpyridinium chloride & phenylenediamine in aq soln at 60–140°(Refs 1, 2 & 3) This compd is expl

Refs: 1) Beil **13**, 80 2) G.T. Morgan & F.M.G. Miklethwait, JChemSoc **93**, 609(1908) & CA **2**, 1568(1908) 3) K. Okoń, BullAcadPolnSci, SérSciChemGéol et Géograph **6**, 319 (1958) (English) & CA **52**, 20153(1958)

N,N′-Dipicryl-5-nitro-1,3-phenylenediamine or **5-Nitro-1,3 bis(2,4,6-trinitroanilino)-benzene,** $(O_2N)_3C_6H_2NH.C_6H_3(NO_2).NH.C_6H_2(NO_2)_3$; mw 575.33, N 21.91%; orn-red crysts (from alc), mp – darkens on heating to 240° & melts with decompn at 260°. Prepd by heating 5-nitro-1,3-phenylenediamine with picrylchloride & Na acetate (Refs 1 & 2)

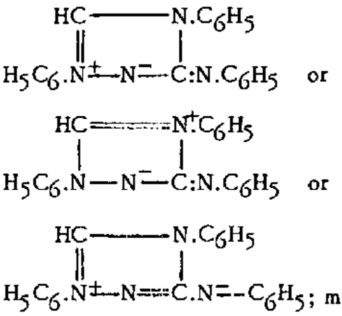
N,N′-Bis(2,4-dinitrophenyl)-2,4,6-trinitro-1,3-phenylenediamine; or **2,4,6-Trinitro-1,5-bis(2,4-dinitrophenylamino)-benzene,** $(O_2N)_2C_6H_3NH.C_6H(NO_2)_3.NH.C_6H_3(NO_2)_2$, no info on this compd, except a report of its synthesis as a reagent for K, is found in the abstract

Both of the above compds are expl

Refs: 1) Beil **13**, [31] 2) A. Korczyński & S. Plasecki, AnzAkadKrakau [A] **1917**, 179 & CA **16**, 2124(1922) 3) H. Ueda et al, NipponKagakuZasshi **86**(8), 865(1965) (Jap) & CA **63**, 17116(1965)

Di(phenyl)-phenylhydrazine. See Triphenylhydrazine in future volume

1,4-Diphenyl-3,5-phenylimino-1,2,4-triazole; 1,4-Diphenyl-3,5-endoanilino-4,5-dihydro-1,2,4-triazole; 1,4-Diphenyl-3,5-endoanilino-1,2,4-triazoline or Nitron [called Anhydro(1.4-diphenyl-3-phenylimino-dihydro-1.2.4-triazoliumhydroxid) or Anhydro (1.4-diphenyl-3-anilino-1.2.4-triazolium hydroxid) in Ger],



; mw 312.36, N 17.94%; lemon-yel fine ndls (from chlf + petr eth) or yel plts (from alc), mp 189° (dec); sol in chlf, acet & eth acet; sl sol in eth & alc; can be prepd by heating triphenylaminoguanidine & formic acid, diluting with water, filtering, and pptg the product with NH_4OH; forms numerous salts, some of which decomp on heating (Ref 1)

Nitron is used as a reagent for detecting the nitrate ion (NO_3^-) in very dil solns (Ref 2)

Refs: 1) Beil **26**, 344, (110) & [76] 2) CondChemDict (1961), 806

Diphenylphthalate (DPhPht), $C_6H_4(COOC_6H_5)_2$; mw 318.31; colorless prisms, sp gr 1.28 at 20°; mp 73°, bp 405° at 759mm; flash p 435°F; insol in w; sl sol in alc or eth; sol in acet. It can be prepd from phthalylchloride & phenol or from phthalyic acid monophenyl ester, phenol & phosphorpentoxide in benz (Refs 1, 2 & 3)

Médard & Thomas (Ref 4) determined its heat of combstn at C_v as 2245.9kcal/mol and heat of formation as 116.9kcal/mol

Technical DPhPht has been used as a plasticizer for NC in the manuf of proplnts. The US Armed Forces requirements and tests listed in Specification MIL-D-50006(Ord) (Nov 1958) are as follows:

1) *Material* – crystalline loose form or solid form

2) *Assay* – 99%. Weigh 0.5g sample to the nearest mg into a dry 250ml Erlenm flask with a ground joint and add 20ml of benzene. Dissolve by warming on an electric hot plate, remove from the plate and add to the warm soln 125ml of 0,5N alcoholic KOH soln. Reflux at 60° for 2 hrs, using a water condenser. Stopper the flask, cool to RT, add 75ml of absolute

ether and allow to stand for 30–60 mins. Filter thru a tared sintered glass crucible of medium porosity and rinse the flask with six 10ml portions of 50/50-ether/alcohol (with help of policeman), transferring the washings to the crucible. Do not allow the ppt to become completely dry during the filtering and washings. Heat the crucible for a few mins at 80° and then at 150° for 3 hours. Weigh as dipotassium phthalate and calculate:

$$\%\text{Diphenylphthalate} = \frac{131.36P}{W}$$

where: P = weight of ppt in the crucible
W = weight of sample

3) *Acidity* (as phthalic acid) – not to exceed 0.05%. Neutralize 100ml of chloroform, placed in a flask, with 0.1N NaOH (in presence of 3 drops of phpht indicator) to a faint pink color. Add an accurately weighed 15g portion of sample to chlf and titrate the mixt with 0.1N NaOH soln

$$\%\text{Phthalic Acid} = \frac{8.3VN}{W}$$

where: V = ml of NaOH soln used in titration
N = normality of NaOH soln
W = weight of sample

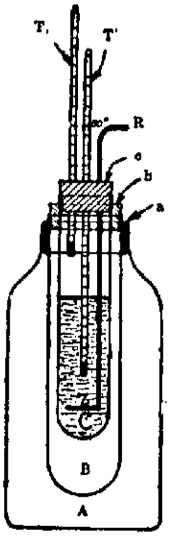
4) *Ash* – not greater than 0.2%. Weigh accurately a 1 g sample in a tared porcelain dish and add ca 1 g of paraffin conforming to MIL-P-3324. Heat on a hot plate until nearly all the material has volatilized. Place the dish in a 650° muffle oven for 30 mins, cool and weigh. Run a blank using 1 g paraffin

$$\%\text{Ash} = \frac{\text{Wt of ash}}{\text{Wt of sample}} \times 100$$

5) *Loss in Weight at 105°* – not more than 0.5% Heat a 3 to 5g sample in a tared shallow dish at 105° to constant weight

$$\%\text{Loss} = \frac{\text{Loss of wt in the dish}}{\text{Wt of sample}} \times 100$$

6) *Setting Point* – 69° to 70°. Use the apparatus shown in Fig. The bottle A is of 1 liter capacity, such as a Kerr Mason Jar No 12, and the Pyrex test tube B (1.5 x 7 inch) is held in place by means of tape (a) around the top. A cork stopper (b) serves for holding Pyrex test tube C, (1 x 6 inch). Cork stopper (c), inserted in tube C, has three holes, of which the center hole serves for "standardized" thermometer T; one of the side holes serves for "correction" thermometer T_1, while the other for the stirring rod R [Nichrome or Al wire ca 1½mm diam with a ring (r)]



SETTING POINT APPARATUS

Procedure. Transfer to the tube C ca 40g of the sample, previously dried for 4hrs at 40° and then heated to ca 80°. Stopper the tube and adjust thermometer T so that the mercury bulb is equally spaced between the bottom of the tube and the surface of molten sample. Adjust thermometer T_1 in the middle of the air space between the surface of liquid and the middle of cork (c). Record number of degrees of the exposed mercury column. Stir the molten sample thruout the test by making ca 120 complete up and down movements of the rod R per minute. Watch the thermometer closely and, as soon as the temp, after falling steadily, starts to rise, stop stirring. Note now the temp every 15 secs (using special reading lens) until the maximum is reached. Record this temp as the "uncorrected setting point" and record at the same time the temp of the correction thermometer T_1. Correct the maximum temp for emergent stem by adding the value calculated from the formula:

$0.000159N(t-t_1)$,

where: 0.000159 = coefficient of expansion of Hg in glass
N = number of degrees in the exposed mercury column of T
t = uncorrected solidification point
t_1 = temp of the exposed Hg column detd by means of thermometer T_1 suspended so that its bulb is in the mid point of the exposed column

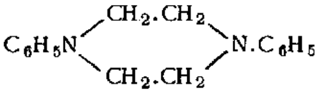
Record the corrected temp as the setting point

Refs: 1) Beil **9**, 801, (360), [594] & {4157} 2) Schreder, Ber **7**, 705(1874) 3) Pawlewski, Ber **28**, 108(1894) 4) L. Médard & M. Thomas, MP **39**, 350, 355(1957) 5) CondChemDict (1961), p 408-R

Di(phenylpicrylhydrazino)-ethane. See *α,β-*Bis(*α*-phenyl-*β*-picryl-hydrazino)-ethane in Vol 2, p B154-R

Diphenylpiperazine and Derivatives

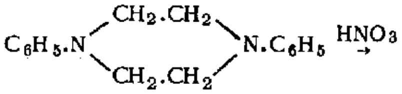
N,N′-Diphenylpiperazine,



mw 238.32, N 11.76%, ndls, mp 163–64°; bp 230–35° at 12mm; insol in w; sol in benz, acet or eth; sl sol in petr eth. It was prepd in 1889 by Bischoff (Refs 1 & 2) by heating equimolecular amounts of aniline and ethylenebromide in the presence of anhydrous Na carbonate. Schouten (Ref 3) prepd it by heating a mixture of 1,2-bis(phenylamino)-ethane with 1,2-dibromoethane and Na acetate for 6 hrs at 150°

Schouten found out that a violent reaction took place when diphenylpiperazine was treated with absol nitric acid even at low temps, such as −10°. After the evolution of nitrous fumes ceased, the resulting solid substance proved to be *1,2-Bis-[N-(2,4,6-trinitrophenyl) -nitramino]-ethane*, code named *Bitetryl* or *Ditetryl* (US) and *Octyl* (GtBritain). Properties and prepn of this compd are described in Vol 2 of Encycl, p B131-R

The reaction observed by Schouten proceeded as follows:



$(O_2N)_3C_6H_2.N(NO_2).CH_2.CH_2.N(NO_2).C_6H_2(NO_2)_3$

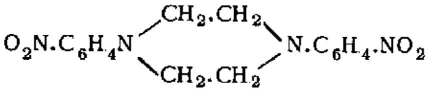
(Ditetryl), which proves that the piperazine ring was opened when abs nitric acid was used No opening of piperazine ring was observed with weaker acid (such as of sp gr 1.42), or when strong nitric acid was mixed with AcOH

Refs: 1) Beil **23**, 8, (5) & [5] 2) C.A. Bischoff, Ber **22**, 1778(1889) 3) A.E. Schouten, Rec **56**, 870(1937) & CA **31**, 7046 (1937)

N,N′-Bis(4-nitrosophenyl)-piperazine, $ON.C_6H_4.N:C_4H_8:N.C_6H_4.NO$; mw 296.32, N 18.91%, grn-yel ppt or dk ndls (from acet-alc), mp – decomp completely at 180°. It was prepd by reaction of Na nitrite & N,N′-diphenylpiperazine in dil HCl (Refs 1 & 2)

Refs: 1) Beil **23**, 8 2) Morley, Ber **12**, 1795(1879) 3) M.W. Swaney & S.B. Lippincott, USP 2545176(1951) & CA **45**, 5357 (1951) (Used as a fungicide) 4) L. Convert & R. Fabre, FrP 1311908(1962) & CA **58**, 10383(1963) (Use as a promoter for heat treatment of butyl rubber & as a vulcanization activator) 5) R. Fabre & G. Bertrand, RevGenCaoutchoucPlastiques **42**(3), 405 (1965) (Fr) & CA **63**, 10149(1965) (Use same as Ref 4)

N,N′-Dinitrodiphenylpiperazine,



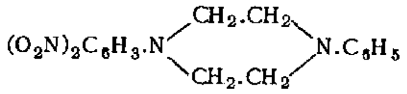
mw 328.32, N 17.07%. Four isomers are known:

N,N′-Bis(2-nitrophenyl)-piperazine, orn pltlts, mp 193–94°(Ref 2)

N,N′-Bis(3-nitrophenyl)-piperazine, brn-yel ndls (from acet), mp 220°(Ref 1) & orn crysts, mp 206°(Ref 3)

N,N′-Bis(4-nitrophenyl) -piperazine, crysts (from Nitrobenz), mp 261°(dec) (Ref 5) & 248°(dec) (Ref 1)

N-(2,4-Dinitrophenyl)-N'-phenylpiperazine,

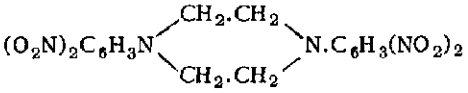


no description of this compd is found in CA (Ref 4)

Other props & methods of prepn are found in the Refs

Refs: 1) Beil **23**, 8 2) R.J.W. LeFèvre, JChemSoc **1930**, 149 & CA **24**, 1863(1930) 3) M.G. Holdsworth & F. Lions, JProcRoySoc, NS Wales **66**, 459(1933) & CA **27**, 2956(1933) 4) V. Prelog & Z. Blažek, CollCzechChem-Communications **6**, 549(1934) & CA **29**, 2959 (1935) 5) M. Aroney & R.J.W. LeFèvre, JChemSoc **1960**, 2163 & CA **54**, 21109(1960)

N,N'-Tetranitrodiphenylpiperazine,



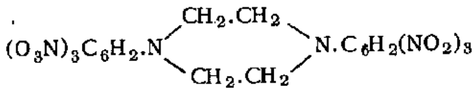
mw 418.32, N 20.09%. Two isomers are known: *N,N'-Bis(2,4-dinitrophenyl)-piperazine,* orn-red ndls (from Nitrobenz), mp – dec with effervescence at 240°; sl sol in glac acet ac, pyridine, acet & chlf; can be prepd by heating piperazine hydrate with 3 mols 4-chloro-1,3-dinitrobenzene (Refs 1, 2, 3 & 4)

N,N'-(2,6-Dinitrophenyl)-piperazine, crysts, mp 269°. It was prepd by refluxing for several hrs a mixt of halo-dinitrobenzene, piperazine, Na carbonate & anhyd alc (Ref 4)

Other props & methods of prepn are found in the Refs

Refs: 1) Beil **23**, [6] 2) R.J.W. LeFèvre & E.E. Turner, JChemSoc **1927**, 1121 & CA **21**, 2681(1927) 3) T.I. Orlova & N.I. Gavrilov, ZhurObshchKhim **27**, 3314(1957) & CA **52**, 9151(1958) [Prepn from piperazine, $NaHCO_3$ & $FC_6H_3(NO_2)_2$-2,4 in alc gave 98% yield, mp 255–58°(dec)] 4) S.P. Gupta et al, JIndianChemSoc **43**(5), 356(1966) (Engl) & CA **65**, 8907(1966)

N,N'-Bis(2,4,6-trinitrophenyl)-piperazine or **Dipicrylpiperazine,**



mw 508.32, N 22.05%; orn-yel crysts (from Nitrobenz), mp – dec at 287°. It was prepd by reacting piperazine with 3 mols of picryl-chloride, or by nitrating N,N-bis[2-(or 4)-nitrophenyl]-piperazine with nitric acid (Refs 1, 2 & 3)

This compd is a mild expl

Refs: 1) Beil **23**, 8 & [6] 2) R.J.W. LeFèvre & E.E. Turner, JChemSoc **1927**, 1121 & CA **21**, 2681(1927) 3) S.P. Gupta et al, JIndianChemSoc **43**(5), 356(1966) (Engl) & CA **65**, 8907(1966)

Diphenylpropane. Same as Dibenzylmethane

Diphenylpropylenediamine and **2,4,6,2',4',6'-Hexanitrodiphenylpropylenediamine** (Code name **Methylene Ditetryl**) are described under Bis(anilino)-propane and Derivatives in Vol 2, pp B132-R to B133-L

Diphenyl-quinomethane and Derivatives

Diphenyl-quinomethane or *Fuchsone,* $(C_6H_5)_2C:C_6H_4:O$, mw 258.30, brn-yel ndls, mp 168–69°, insol in w, sl sol in eth, sol in acet; can be prepd by heating diphenyl(4-hydroxyphenyl)-methanol in glac acet ac & concentrating the soln at 90°(Refs 1 & 4)

Mononitrodiphenyl-quinomethane, $(C_6H_5)_2C:C_6H_4(NO_2):O$, mw 303.30, N 4.62%. Only one isomer is found in the literature: *3-Nitrofuchsone,* shiny brownish powd, mp 100°; readily sol in benz, chlf, alc & ethyl benzoate; insol in petr eth. It was prepd by heating 3-nitro-4-methoxytriphenylchloromethane for 3 hrs at 180°(Refs 2 & 3)

Dinitro, $C_{19}H_{12}N_2O_5$, *Tetranitro,* $C_{19}H_{11}N_3O_7$, and *Hexanitro,* $C_{19}H_{10}N_4O_9$, derivs were not found in the literature thru 1966

Refs: 1) Beil **7**, 520(290), [478] & {2726} 2) Beil **7**, [478] 3) P. Demont, Dissert (Freiburg, Schweiz) (1919), 19 4) I.S.

Ioffe & Z.Ya. Khavin, ZhurObshchKhim **19**, 917(1949) & CA **43**, 8683(1949)

Diphenylselenide and Derivatives

Diphenylselenide or *Seleniumdiphenyl*, $(C_6H_5)_2Se$, mw 233.16; oil, sp gr 1.338 at 16/4°, fr p 2.5°, bp 301–02°; miscible with alc or eth. It can be prepd by treating diphenylselenium oxide with Zn & alcoholic ammonia, or by warming dibromodiphenylselenide with Na ethylate in alc on a w bath (Refs 1 & 2)
Refs: 1) Beil **6**, 345, (164), [318] & {1106} 2) P. Donzelot & M. Chaix, CR **201**, 501 (1935) & CA **29**, 7191(1935)

Dinitrodiphenylselenide or **Bis (Nitrophenyl)-selenide**, $O_2N.C_6H_4Se.C_6H_4.NO_2$; mw 323.16, N 8.67%. Three isomers are known:
2,2′-Dinitrodiphenyl-, prisms (from toluene or ethyl acet), mp 143° (Refs 1 & 4)
4,4′-Dinitrodiphenyl-, pale-yel prisms (from ethyl acet), mp 170–75° (Refs 2, 3, 5, 6, 7 & 8) and
2,4-Dinitrophenyl-phenylselenide, $C_6H_5SeC_6H_3(NO_2)_2$, no description of compd is found in abstract (Ref 6)
Refs: 1) Beil **6**, {1116} 2) Beil **6**, {1120} 3) J.W. Baker & W.G. Moffitt, JChemSoc **1930**, 1722 & CA **24**, 5291(1930) 4) J Matti, BullFr **7**, 617, 619(1940) & CA **36**, 2850(1942) 5) P.L.N. Rao, JIndianChemSoc **18**, 1(1941) & CA **35**, 7945(1941) 6) L. Bartolotti & R. Passerini, RicercaSci **25**, 1095(1955) & CA **50**, 224(1956) 7) A. Arcoria, BollSeduteAccadGioeniaSciNatCatania [4] **3**, 314(1956) & CA **52**, 9980 (1958) 8) L.M. Litvinenko & R.S. Cheshko, ZhurObshchKhim **30**, 3682(1960) & CA **55**, 19847(1961)

2,4,2′,4′-Tetranitrodiphenylselenide or **Bis(2,4-Dinitrophenyl)-selenide**, $(O_2N)_2C_6H_3SeC_6H_3(NO_2)_2$; mw 413.16, N 13.56%; golden-yel ndls (from ethyl acet + alc), mp 194–96°, bp – dec at 240° & explodes at higher temp. It can be prepd by reacting 4-bromo-1,3-dinitrobenzene with ½ mol K monoselenothiosulfate (K_2SeSO_3) in aq alc, or from 4-chloro-1,3-dinitrobenzene & hydrogen selenide in boiling alcoholic acetic acid (Refs 1, 2 & 3)
Refs: 1) Beil **6**, (165) & [322] 2) D.F. Twiss, JChemSoc **105**, 1677(1914) & CA **8**, 3031(1914) 3) O. Behaghel & M. Rollman, Ber **62**, 2699(1929) & CA **24**, 1344(1930) 4) Not found in later refs thru 1966

Pentanitrodiphenylselenide, $C_{12}H_5N_5O_{10}Se$, not found in the literature thru 1966

2,4,6,2′,4′,6′-Hexanitrodiphenylselenide or **Dipicrylselenide**, $(O_2N)_3C_6H_2.Se.C_6H_2(NO_2)_3$; mw 503.16, N 16.70%, lt-brn crysts (from ethyl acetate + alc), mp 242° (dec); explodes & deposits selenium; sol in hot ethyl acetate & acet ac; v sl sol in hot alc. It can be prepd from reaction of picrylchloride & K monoselenothiosulfate (K_2SeSO_3) in dil alc & in presence of Na carbonate (Refs 1 & 2), and from KSeCN & 2,4,6-$(O_2N)_2C_6H_2Cl$ in alc (Ref 3)
Refs: 1) Beil **6**, (165) 2) D.F. Twiss, JChemSoc **105**, 1676(1914) & CA **8**, 3031 (1914) 3) M. Giua & R. Bianco, GazzChimItal **89**, 693(1959) & CA **54**, 22454(1960)

Diphenylsulfide and Derivatives

Diphenylsulfide or *Bis(phenyl)-sulfide*, $(C_6H_5)_2S$, mw 186.26, colorless liq, sp gr 1.119 at 15/15°, fr p minus 25.9°, bp 296–97°; insol in w; sol in benz, eth & CS_2; sol in hot alc. It can be prepd by reaction of benzene & sulfur in the presence of Al chloride (Ref 1) (See also Diphenyldisulfide)

Mononitrodiphenylsulfide, $C_6H_5.S.C_6H_4.NO_2$; mw 231.27, N 6.06%. Three isomers are known:
2-Nitro-, yel-orn crysts (from alc + eth), mp 79–82°
3-Nitro,- lt-yel crysts, mp 42.5° and
4-Nitrodiphenyl sulfide, lt-yel prisms (from petr eth), mp 55°

Other props & methods of prepn are found in Beil
Ref: Beil **6**, 337, 339, (154, 159), [305,311] & {1058, 1066, 1070}

Dinitrodiphenylsulfide, $O_2N.C_6H_4.S.C_6H_4.NO_2$; mw 276.27, N 10.14%. Five isomers are known:
2,2'-Dinitrodiphenylsulfide, yel ndls (from eth acet + alc), mp 122–23°, bp – sublimes with partial decompn (Ref 1)
2,4'-Dinitrodiphenylsulfide, crysts (from glac acet ac), mp 163–64° & 158–59° (Ref 2)
3,4'-Dinitrodiphenylsulfide, yel crysts (from alc), mp 125° (Ref 3)
4,4'-Dinitrodiphenylsulfide, orn-colored pltlts (from glac acet ac), mp 156–61° (Ref 4)
2,4-Dinitrodiphenylsulfide, $(O_2N)_2C_6H_3.S.C_6H_5$, lt-yel ndls (from acet or benz + alc), mp 119–21° (Ref 5)

Other props & method of prepn are found in Beil

Refs: 1) Beil **6**, 337, [305] & {1059} 2) Beil **6**, {1070} 3) Beil **6**, {1071} 4) Beil **6**, 339, [311] & {1071} 5) Beil **6**, [315] & {1094}

Trinitrodiphenylsulfide, $C_{12}H_7N_3O_6S$; mw 321.27, N 13.08%. Three isomers are known:
2,4,2'-Trinitrodiphenylsulfide, $(O_2N)_2C_6H_3.S.C_6H_4.NO_2$, yel tablets (from alc), mp 131–33° (Ref 1)
2,4,4'-Trinitrodiphenylsulfide, $(O_2N)_2C_6H_3.S.C_6H_4.NO_2$, yel tablets (from acet ac), mp 158–60° (Ref 1)
2,4,6-Trinitrodiphenylsulfide, $(O_2N)_3C_6H_2.S.C_6H_5$, crysts, mp 120° (dec) (Refs 2 & 3)

Other props & methods of prepn are found in Refs

Refs: 1) Beil **6**, 343 & {1095} 2) Beil **6**, {1103} 3) A. Mangini & R. Passerini, RAL (8) **7**, 324, 327 (1949)

2,4,2',4'-Tetranitrodiphenylsulfide or **Bis(2,4-Dinitrophenyl)-sulfide,** $(O_2N)_2C_6H_3.S.C_6H_3(NO_2)_2$; mw 366.27, N 15.29%, crysts (from glac acet ac), mp 196–97°, bp – explodes at 280°; sol in nitrobenz & pyridine. It can be prepd by reacting 4-chloro-1,3-dinitrobenzene with Na_2S or $Na_2S_2O_3$ in aq alc, and by other methods (Refs 1, 2 & 3)

Refs: 1) Beil **6**, 343, (163), [315] & {1095} 2) H.H. Hodgson & D.P. Dodgson, JChemSoc **1948**, 1002 & CA **43**, 222 (1949) 3) G. Leandri & A. Tundo, AnnChim (Rome) **44**, 330 (1954) & CA **49**, 15784 (1955)

2,4,6,2',4'-Pentanitrodiphenylsulfide, $(O_2N)_3C_6H_2.S.C_6H_3(NO_2)_2$; mw 411.27, N 17.03%; large, thick yel crysts (from glac acet ac), or small almost colorless pltlts (from acet + w), mp 217°, bp – explodes; sol in glac acet ac, benz & acet; almost insol in petr eth. It can be prepd by reacting an alcoholic soln of K_2S, 4-chloro-1,3-dinitrobenzene & picryl chloride (Refs 1 & 2)

Refs: 1) Beil **6**, 343 2) Willgerodt, Ber **8**, 305 (1875) 3) Not found in later Refs thru 1966

2,4,6,2',4',6'-Hexanitrodiphenylsulfide (HNDPhS), Bis(2,4,6-trinitrophenyl)-sulfide or **Dipicrylsulfide,** $(O_2N)_3C_6H_2.S.C_6H_2(NO_2)_3$; mw 456.27, N 18.42%, OB to CO_2 & SO_2 minus 56.1%; yel solid, mp 234° (corr); resolidifies at 213° and remelts at 234°; bp detonates at 320°; sp gr 1.70 at 3000kg/sq cm; insol in w; sl sol in CS_2, petr eth, alc, ether or chlf; sol in acet or glac AcOH. It was first prepd and patented in Germany in 1912 (Refs 1 & 2) and proposed for use in mixts with other expls for filling mines, torpedoes and bombs. Then it was patented in GtBritain (Ref 3), Canada (Ref 9) and Sweden (Ref 5). Accdg to Pepin Lehalleur (Ref 11), HNDPhS was used during WWI in mixts with TNT for loading some French aerial bombs and accdg to Stettbacher (Ref 10) and Davis (Ref 15) it was used in German WWI bombs mixed with equal parts of TNT. Accdg to Bebie (Ref 13) it was also used during WWII in Ger drop bombs. Davis stated the fact that expln of HNDPhS produces obnoxious sulfur dioxide has commended it for use in projectiles intended to make closed spaces, such as casemates, holds of ships, etc untenable

Following methods of prepn of HNDPhS are described in the literature:
a) Treating 2 mols of Chloro-2,4,6-trinitrobenzene (Picrylchloride) with 1 mol of Na thiosulfate in water, followed by neutraliza-

tion of liberated acid

$2C_6H_2(NO_2)_3Cl + Na_2S_2O_3 + H_2O \rightarrow$

$2NaCl + H_2SO_4 + [C_6H_2(NO_2)_3]S$ (Refs 6, 7 & 8)

Neutralization can be achieved if the reaction is conducted in the presence of Mg carbonate or conducting reaction in alcohol instead of water (Refs 4 & 14)

b) Treating 2 mols of Picrylchloride with 1 mol K sulfide in 1% alc soln

$2C_6H_2(NO_2)_3Cl + K_2S \rightarrow 2KCl + [C_6H_2(NO_2)_3]S$ (Ref 1)

c) Giua (Ref 9) proposed to warm Picrylchloride with thiourea in 90–92% alcohol:

$2C_6H_2(NO_2)_3Cl + CS(NH_2) + C_2H_5OH \rightarrow$

$2HCl + [C_6H_2(NO_2)_3]S + HN{:}C(OC_2H_5).NH_2$

(See also Ref 14)

Following are properties of HNDPhS:

Eutectic with 86.5% TNT – mp 78°

Explosion Point – 319° if temp is raised at the rate of 20°/min and 302° at 5°/min

Heat of Combstn – 1425cal/mol at C_v(Ref 16)

Heat of Formation – minus 30Kcal/kg or minus 14 Kcal/mol [Ref A. Schmidt, SS **9**, 263(1934)]

Impact Sensitivity: less sensitive than PA, (FI 83% PA) (Ref 16); more sensitive than Tetryl: Kast app with 2kg fall for 0/6 shots is 36–39cm vs 49–51cm for Tetryl (Ref 7)

Power by Trauzl Test – 107% PA (Ref 8); ADL Rept 1 gives 101% PA & 110% TNT

Thermal Stability at 95° – no decompn in 30 eight hour days of heating (Ref 7)

Toxicity. The substance is not toxic, but its vapors cause intense vesication (Ref 15)

Uses: Besides its uses in mixts with TNT and other nitrohydrocarbons for filling bombs, shells, mines, torpedos, etc listed above, it was proposed for use in primers and detonators (Refs 4, 13 & 15)

Refs: 1) Beil **6**, 344, (163), [316] & {1103} 2) Sprengstoff AG Carbonit, GerP's 275037 and 286543(1912) & CA **9**, 381(1915) and **10**, 1272(1916) 3) C. Hartmann, BritP 18355 (1913); SS **9**, 195(1914) & CA **9**, 381(1915) 4) Canadian Explosives Ltd, CanadP's 183396, 183498, 183501 and 183505(1918) & CA **12**, 1348(1918) 5) Sprengstoff AG Carbonit, SwedP 43977(1918) & CA **12**, 2689(1918) 6) Colver (1918), 384 7) C.F. van Duin & B.C.R. van Lennep, Rec **39**, 145 (1920) 8) B. Flürscheim, JSCI **40T**, 97 (1921) 9) M. Giua, GiornChimIndustrAppl **6**, 166–67(1924) & CA **18**, 3273(1924) 10) Stettbacher(1933), 294 11) Pepin Lehalleur (1935), 275 (Sulfure de picryl) 12) Thorpe **4** (1940), 484 (Use of HNDPhA for filling aerial bombs) 13) Bebie(1943), 83–84 14) Clift & Fedoroff, **2** (1943), pH5 15) Davis (1943), 187 16) A.H. Blatt, OSRD Rept **2014**(1944) 17) Pérez Ara (1945), 536 (Sulfuro de picrilo) 18) Vivas, Feigenspan & Ladreda **2**(1946), 179–81 (Sulfuro-hexanitrodifenilico) 19) ADL PureExplCompds Pt **1** (1947), p 109 20) ADL PureExplCompds Pt **2** (1947), p 247 21) G. Leandri et al, Gazz **84**, 3(1954) & CA **50**, 222(1956) (UV absorption spectrum & chromatographic props) 22) S.S. Joshi & T.C. Mathur, JIndianChemSoc **40**(11), 934(1963) & CA **60**, 5376(1964) 23) Urbański **1**(1964), p 553 24) V.G. Pesin & I.G. Vitenberg, ZhObshchKhim **26**(7), 1268 (1966) & CA **65**, 16957(1966)

Diphenylsulfocarbamide. See Diphenylthiocarbamide

Diphenylsulfocarbazone. See Diphenylthiocarbazone

Diphenylsulfocarbodiazone. See Diphenylthiocarbodiazone

Diphenylsulfone and Derivatives

Diphenylsulfone or *Sulfobenzide*, $(C_6H_5)_2SO_2$, mw 218.26; ndls, sp gr 1.248 at 25/4°, mp 128–29°, bp 379°; sl sol in hot w; sol in benz and in hot alc. It can be prepd from reaction of benzenesulfonyl chloride & benzene in the presence of $FeCl_3$ at 80–110°, and by several other methods (Ref)

Ref: Beil **6**, 300, (144), [290] & {992}

4,4′-Diazidodiphenylsulfone or **Bis(4-Azido phenyl)-sulfone,** $N_3C_6H_4.SO_2.C_6H_4N_3$; mw 300.30, N 28.03%, crysts (from acet), mp 156–57°(explosion point). It was prepd by diazotizing bis(4-aminophenyl)-sulfone in aq sulfuric acid and treating the diazonium soln with a cold soln of hydrazine hydrate & Na acetate (Refs 1 & 2)

Refs: 1) Beil 6, {1104} 2) C.K. Banks & O.M. Gruhzit, JACS **70**, 1268(1948)

Mononitrodiphenylsulfone or **Phenyl-(nitrophenyl)- sulfone,** $O_2N.C_6H_4.SO_2.C_6H_5$; mw 263.27, N 5.32%. The three possible isomers are known:

2-Nitrodiphenylsulfone, colorless crysts (from alc), mp 147°; sol in warm alc, benz & glac acet ac; insol in w (Ref 1)

3-Nitrodiphenylsulfone, ndls (from alc), mp 81–85°(Ref 2)

4-Nitrodiphenylsulfone, pltlts (from alc or glac acet ac), mp 140–43°(Ref 3)

Other props & methods of prepn are found in Beil.

Refs: 1) Beil 6, 338, (154) & {1059} 2) Beil **6**, (158) & {1066} 3) Beil 6, 339, (159), [311] & {1071}

Dinitrodiphenylsulfone or **Bis(nitrophenyl)-sulfone,** $O_2N.C_6H_4.SO_2.C_6H_4.NO_2$; mw 308.27 N 9.30%. Five isomers are known:

2,2′-Dinitrodiphenylsulfone, crysts (from glac acet ac), mp 163°(Ref 1)

3,3′-Dinitrodiphenylsulfone, tablets, mp 198–203 °; sol in hot glac acet ac; sl sol in alc & eth (Ref 2)

3,4′-Dinitrodiphenylsulfone, crysts (from glac acet ac), mp 181°(Ref 3)

4,4′-Dinitrodiphenylsulfone, crysts (from glac acet ac), mp 282–90°(dec); insol in w; v sl sol in alc; sl sol in hot 80% acet ac (Ref 4)

2,4-Dinitrodiphenylsulfone, $(O_2N)_2C_6H_3.SO_2.C_6H_5$, ndls (from alc or glac acet ac), mp 161°; insol in eth; sl sol in alc; mod sol in hot glac acet ac & benz (Ref 5)

Other props & methods of prepn are found in Beil

Refs: 1) Beil 6, (154) & {1059} 2) Beil **6**, (158), [309] & {1066} 3) Beil 6, {1072} 4) Beil **6**, 340, (311) & {1072} 5) Beil **6**, 343 & {1096}

Trinitrodiphenylsulfone or **Nitrophenyl-(dinitrophenyl)-sulfone,** $(O_2N)_2C_6H_3.SO_2.C_6H_4.NO_2$; mw 353.27, N 11.89%. Two isomers are known:

2,4,3′-Trinitrodiphenylsulfone, crysts (from glac acet ac), mp 196–97°; was prepd by heating 4-chloro-1,3-dinitrobenzene or 2,4-dinitrophenyl-p-toluenesulfonate with Na 3-nitrobenzene-1-sulfinate in aq alc (Refs 1 & 3)

3,5,4′-Trinitrodiphenylsulfone, ndls, mp 199°; prepd by heating 3-nitrophenyl-(4-nitrophenyl)-sulfone with a mixt of fuming nitric acid & sulfuric acid at 150°(Refs 2 & 4)

Refs: 1) Beil **6**, {1097} 2) Beil **6**, {1102} 3) J.D. Loudon, JChemSoc **1935**, 538 4) Y Takubo, JPharmSocJapan **62**, 518(1942) & CA **45**, 2893(1951)

2,4,2′,4′-Tetranitrodiphenylsulfone or **Bis(2,4-Dinitrophenyl)-sulfone,** $(O_2N)_2C_6H_3.SO_2.C_6H_3(NO_2)_2$; mw 398.27, N 14.40%; crysts (from aq acet), mp 174–75°; sol in acet; mod sol in glac acet ac; insol in w; dec in alkalies. Can be prepd from bis(2,4-dinitrophenyl) -sulfide by heating for 6 hrs with an excess of CrO_3 in glac acet ac or by heating with $K_2Cr_2O_7$ in 87% sulfuric acid to 120° or in 57% sulfuric acid at boiling temp. It was also prepd by heating bis(2,4-dinitrophenyl)-sulfoxide with $K_2Cr_2O_7$ in 57% sulfuric acid at its boiling temp (Refs)

Refs: 1) Beil **6**, {1097} 2) R.C. Fuson & S. Melamed, JOrgChem **13**, 690, 695(1948) & CA **43**, 3803(1949) 3) T. Matsukawa et al, JPharmSocJapan **70**, 77(1950) & CA **44**, 5322(1950) 4) H. Bradbury, JChemSoc **1953**, 1184 & CA **48**, 3288(1954) 5) K. Okoń, BiulWjskowejAkadTech im JaroslawaDabrowskiego, PractChem **8**, 15(1959) (Engl summary) & CA **54**, 22466(1960) 6) C.W. Muth et al, JOrgChem **25**, 738(1960) & CA **54**, 24770(1960) (Compd incorrectly listed in CA, should read 2,4-Dinitrodiphenylsulfone) 7) M. Nakadate et al, ChemPharmBull(Tokyo) **12**(10), 1138 (1964) (Engl) & CA **62**, 809(1965)

2,4,2′,4′,6′-Pentanitrodiphenyl-sulfone (PNDPhSo) or **Thional,**
$(O_2N)_2C_6H_3.SO_2.C_6H_2(NO_2)_3$; mw 443.27, N 15.80%, OB to CO_2 & SO_2 minus 59.5%' lt yel crysts, mp 240°. Its method of prepn is not reported

It is an expl which was examined during WWII in GtBritain and the USA and found to be less powerful than TNT or Picric Acid (75% of PA by Trauzl Test) and more sensitive to impact (FI 117% PA) (Ref 10, p 74)

2,4,6,2′,4′,6′-Hexanitrodiphenyl-sulfone (HNDPhSo), Bis(2,4,6-trinitrophenyl)-sulfone or **Dipicrylsulfone,**
$(O_2N)_3C_6H_2.SO_2.C_6H_2(NO_2)_3$, mw 488.27, N 17.21%, OB to SO_2 & CO_2 minus 45.9%; lt yel crysts, mp 226°(Ref 8); 307°(Davis & Blatt); >254°(Refs 2 & 6); bp – explodes at 290°(Ref 8); diff sol in w; sol in acet + alc. It can be prepd by oxidation of Hexanitrodiphenyl sulfide with Cr trioxide in nitric acid suspension (Ref 2)

It was described as "peroxide" in original patent (Ref 3). Formulation as "sulfone" was suggested by Stettbacher (Ref 5)

Following are properties of HNDPhSo:
Compatibility with Metals – does not attack the metals and does not form any metallic salts (Ref 10)
Explosion Point – 297° when heated at the rate of 5°/min (Refs 4 & 10)
Impact Sensitivity: less sensitive than HNDPhS and Picric Acid and more sensitive than Tetryl: FI 70% PA and Kast app with 2kg wt the max fall for 0/6 shots 43cm vs 49–51cm for Tetryl (Ref 10)
Power by Trauzl Test – 107% PA (Ref 10)
Thermal Stability – no decompn took place in thirty 8hr days at 95°(Ref 4); did not decomp after being stored for a long time at temps as high as 130°(Ref 8)
Toxicity – Produces on expln toxic gases contg sulfur dioxide (Ref 5)
Velocity of Detonation – 5210m/sec (Ref 15)

It was patented by Sprengstoff AG Carbonit (Ref 3)(either alone or in component expls) for filling shells, bombs and torpedoes, as well as for blasting purposes. It might be suitable for use in primers and detonators (Refs 6, 8, 11 & 12). Germans used it in mixts with TNT for filling aerial bombs(Ref 5)

Refs: 1) Beil, not found (Pentanitro) 2) Beil **6**, (163) & [317] (Hexanitro) 3) Sprengstoff AG Carbonit, GerP 269826 (1912) & CA **8**, 2253(1914) 4) C.F. van Duin & B.C.R. van Lennep, Rec **39**, 145(1920) & CA **14**, 2708(1920) 5) Stettbacher (1933). 294–95 6) Pepin Lehalleur (1935), 271 7) A.H. Blatt & F.C. Whitmore, OSRD Rept **1085**(1942), 50 & 74 8) Clift & Fedoroff **2**(1943), p H5 9) Davis (1943), 187–88 10) A.H. Blatt, OSRD **2014**(1944) 11) Pérez Ara (1945), 537 12) Vivas, Feigenspan & Ladreda **2** (1946), 181 (Hexanitrodifenilsulfona) 13) E. von Herz Sr & E. von Herz Jr, GerP 859868(1952) & CA **52**, 14171(1958) 14) K. Okoń, BiulWojskowejAkadTechnicznej im JDabrowskiego **7**, No 38, 6 (1957) & CA **53**, 4181(1959) 15) J.E. Hughes & D.N. Thatcher, USP 2952708(1960) & CA **55**, 3062 (1961) (Dipicrylsulfone expls) 16) DuPont, BritP 858084(1961) & CA **55**, 12360(1961) (Monoclinic Dipicrylsulfone vs orthorhombic form) 17) DuPont, GerP 1078920(1961) & CA **56**, 624(1962) (Dipicrylsulfone, prepn & use in elec fuzes) 18) Urbański 1(1964), 554

Diphenylsulfourea. See Diphenylthiourea in this Vol

Diphenyltetraacetylene or **1,8-Diphenyl-1,3,5,7-octatetrayne,**
$C_6H_5C{\vdots}C.C{\vdots}C.C{\vdots}C.C{\vdots}C.C_6H_5$; mw 250.28, yel ndls, mp 115–16°(browning), stable at RT for 13 months in the dark; when placed on a hot metallic plate it decompd explosively with much soot. It shows no color reaction with sulfuric acid; is more sol than tolan & $(C_6H_5C{\vdots}C)_2$ in polar solvs such as MeOH, alc & acetone. It was prepd by oxidative coupling of cuprous acetylide, $C_6H_5.C{\vdots}C.C{\vdots}C.Cu$, with $CuCl_2$ (Refs)
Refs: 1) Beil, not found 2) M. Nakagawa, Kagaku no Ryôiki (JJapanChem) **4**, 564;

ProcJapanAcad **26**, (10) 38 (1950) (Engl) & CA **45**, 7081–82(1951) 3) H.H. Schlubach & V. Franzen, Ann **572**, 116(1951) & CA **45**, 10206(1951) 4) Ibid, Ann **573**, 110(1951) & CA **45**, 1956(1952) (UV absorption spectrum) 5) M. Nakagawa, JChemSocJapan, PureChemSect **72**, 561(1951) & CA **46**, 6602–03(1952) (Prepn) 6) Ibid, **72**, 993 (1951) & CA **47**, 2740(1953) (Prepn) 7) Ibid, **73**, 141(1952) & CA **47**, 10511–12 (1953) (A new synthesis) 8) E.R.H. Jones, RecordChemProg **14**, No 1, 1–5(1953) & CA **48**, 9895(1954) 9) J.B. Armitage et al, JChemSoc **1954**, 147 & CA **49**, 1672(1955) 10) H. Günter et al, ChemBer **92**, 3064(1959) & CA **54**, 6505(1960) (Prepn) 11) T. Watanabé et al, ActaCryst **12**, 347(1959) (Engl) & CA **54**, 16097(1960) (Crystal data) 12) I.L. Kotlyarevskii & L.I. Vereshchagin, IzvAkadNauk SSSR, OtdKhimNauk **1962**, 162 & CA **57**, 11155(1962) (Prepn) 13) K. Ernst et al, Ber **95**, 1943(1962) & CA **57**, 15047(1962) (Prepn) 14) V.N. Andrievskii et al, IzvAkadNauk SSSR, SerKhim **1966** (5), 882 (Russ) & CA **65**, 13580(1966) (Prepn)

Diphenyl Tetraozonide. See Biphenyl Tetraozonide in Vol 2, p B126-R

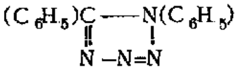
I, III-Diphenyl-tetrazene (1); 1,3-Diphenylisotetrazene or **Diazobenzenephenylhydrazide** [1,3-Diphenyl-tetrazen(1) in Ger], $C_6H_5.N{:}N.N(C_6H_5).NH_2$; mw 212.25, N 26.40%; pale yel crysts (from eth); mp 71° (with decompn); bp explodes at higher temp; insol in w, dil acids & alkalies; sl sol in alc; very sol in eth, benz & glac AcOH. It can be prepd by several methods, eg, by the action of iodine on phenylhydrazine in Na carbonate soln and in atm of CO_2:

$$2C_6H_5.NH.NH_2+2I_2{=}4HI+C_6H_5.N{:}N.N(C_6H_5)NH_2$$

Refs: 1) Beil **16**, 746 2) R. Stollé, JPraktChem [2] **66**, 336(1902) & JCS **84** (Abs 2), 100(1903)

Diphenyltetrazole and Derivatives

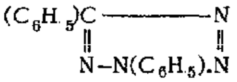
1,5-Diphenyl-1H(or a)-tetrazole or *1,5-Diphenyl-1,2,3,4-tetrazole*,



$$\begin{array}{l}(C_6H_5)C\text{——}N(C_6H_5)\\ \qquad\;\|\qquad\;|\\ \qquad N-N{=}N\end{array}$$

mw 222.24, N 25.21%; ndls (from alc), mp 146°. It was prepd by heating benzophenone chloride with Na azide in diisoamyl ether and in a CO_2 stream at 140–60° (Refs 1 & 2) *Refs:* 1) Beil. **26**, 362 & (112) 2) G. Schroeter, Ber **42**, 2341–43, 3359–60(1909)

2,5-Diphenyl-1,2,3,4-tetrazole,

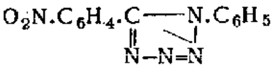


$$\begin{array}{l}(C_6H_5)C\text{————}N\\ \qquad\;\|\qquad\quad\;\;\|\\ \qquad N-N(C_6H_5).N\end{array}$$

ndls (from alc or petr eth), mp 101.5–102°; sol in common org solvs; insol in w. It was prepd by heating benzaldehyde-phenylhydrazone with phenyl azide in the presence of Na methylate soln in a tube on a w bath (Refs 1 & 2) *Refs:* 1) Beil **26**, 362 & (112) 2) O. Dimroth & S. Merzbacher, Ber **40**, 2402–04(1907) & CA **1**, 2610(1907)

Mononitrodiphenyltetrazole, $C_{13}H_9N_5O_2$; mw 267.24, N 26.21%.

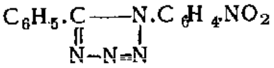
1-Phenyl-5-(4-nitrophenyl)-tetrazole,



$$\begin{array}{l}O_2N.C_6H_4.C\text{——}N.C_6H_5\\ \qquad\qquad\;\|\qquad\;|\\ \qquad\qquad N-N{=}N\end{array}$$

lt-yel pltlts, mp 177–78°. It was prepd by heating phenyl-(4-nitrophenyl)-dichloromethane with Ag azide in diisoamyl ether (Refs 1 & 4)

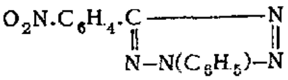
1-(4-nitrophenyl)-5-phenyl-tetrazole,



$$\begin{array}{l}C_6H_5.C\text{——}N.C_6H_4NO_2\\ \qquad\;\|\qquad\;|\\ \qquad N-N{=}N\end{array}$$

yel prisms (from ethyl acetate+alc), mp 149°; insol in w & petr eth; sl sol in alc; readily sol in ethyl acet. It was prepd by heating 1-(4-nitrophenyl)-4-benzoyl-1-tetrazene with aq NaOH (Refs 2 & 5)

2-Phenyl-5(4-nitrophenyl)-tetrazole,

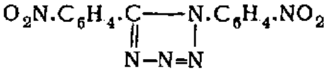


$$\begin{array}{l}O_2N.C_6H_4.C\text{————}N\\ \qquad\qquad\;\|\qquad\quad\;\;\|\\ \qquad\qquad N-N(C_6H_5)-N\end{array}$$

crysts (from acet), mp 199–200°; sol in chlf, acet, glac acet ac & benz; sl sol in alc, eth & petr eth. It was prepd by carefully heating

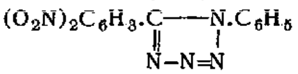
N-phenyl-N'-guanyl-C-(4-nitrophenyl)-formazan with concd nitric acid at 70°(Refs 1 & 3)
Refs: 1) Beil **26**, 365 2) Beil **26**, (112) 3) E. Wedekind, Ber **31**, 475, 478(1898) 4) G. Schroeter, Ber **42**, 3360(1909) 5) O. Dimroth & G. de Montmollin, Ber **43**, 2914 (1910) & CA **5**, 494(1911)

Dinitrodiphenyltetrazoles, $C_{13}H_8N_6O_4$;
1,5-Bis(nitrophenyl)-1,2,3,4-Tetrazole,
mw 312.24, N 26.92%.



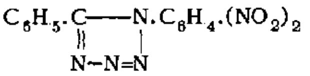
$$\begin{array}{l} O_2N.C_6H_4.C\text{——}N.C_6H_4.NO_2 \\ \qquad\qquad\quad \| \qquad\;\; | \\ \qquad\qquad\quad N-N=N \end{array}$$

The 1,5-Bis (o-), mp 209°, (m-), mp 244°, and (p-), mp 262° derivs were prepd by Braun & Rudolph (Ref 2) by nitrating the parent compd with a 1/1 mixt of fuming nitric acid + sulfuric acid on a water bath. Horwitz & Grakauskas (Ref 4) prepd the 1,5-Bis(p-dinitro) deriv by coupling diazotized p-nitroaniline with 1-acetyl-2-(4'-bromobenzoyl) hydrazine, separating the tetrazole following cyclization
1-(m-Nitrophenyl)-5-(p-nitrophenyl)-tetrazole, crysts, mp 170° (Ref 2)
5-(2,4-Dinitrophenyl) -1-(phenyl) -tetrazole,



$$\begin{array}{l} (O_2N)_2C_6H_3.C\text{——}N.C_6H_5 \\ \qquad\qquad\quad \| \qquad\;\; | \\ \qquad\qquad\quad N-N=N \end{array}$$

crysts, mp 164°(Refs 2 & 3)
1-(2,4-Dinitrophenyl)-5-(phenyl) -tetrazole,

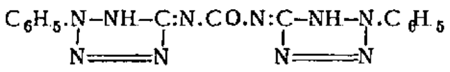


$$\begin{array}{l} C_6H_5.C\text{——}N.C_6H_4.(NO_2)_2 \\ \qquad\;\; \| \qquad\;\; | \\ \qquad\;\; N-N=N \end{array}$$

crysts, mp 183°; was prepd by stirring a suspension of $C_6H_5C.Cl:N.C_6H_3(NO_2)$-2,4 in acet with Na azide in alc for 3 hrs at RT (Refs 3 & 5)
Refs: 1) Beil, not found 2) J.V. Braun & W. Rudolph, Ber **74B**, 267, 271(1941) & CA **35**, 2892(1941) 3) F.L. Scott et al, JACS **75**, 5310(1953) & CA **49**, 169(1955) 4) J.P. Horwitz & V.A. Grakauskas, JOrgChem **19**, 199–200(1954) & CA **49**, 3174(1955) 5) G. Bianchetti et al, Gazz **94** (3–4), 210(1964) & CA **61**, 5633(1964)

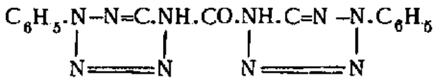
NOTE: No higher nitro derivs of Diphenyltetrazole were found in the literature thru 1966

N,N'-Di(2-phenyl-5-tetrazolyl)-urea {called N.N'-Bis-[2-phenyl-tetrazolyl-(5)]-harnstoff or Bis-[2-phenyl-tetrazolinyliden-(5)]-harnstoff in Ger}



$$\begin{array}{l} C_6H_5.N-NH-C:N.CO.N:C-NH-N.C_6H_5 \\ \qquad\;\;\, | \qquad\qquad\;\, | \qquad\qquad\;\; | \qquad\quad\;\; | \\ \qquad\;\;\, N\text{=====}N \qquad\quad N\text{=====}N \end{array}$$

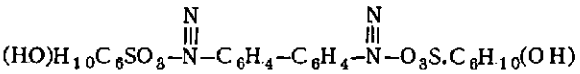
or



$$\begin{array}{l} C_6H_5.N-N=C.NH.CO.NH.C=N-N.C_6H_5 \\ \qquad\;\;\, | \qquad\quad\;\; | \qquad\qquad\;\; | \qquad\quad\;\; | \\ \qquad\;\;\, N\text{=====}N \qquad\quad N\text{=====}N \end{array}$$

mw 348.32, N 40.19%, yel ndls (from glac acet ac), mp 216°(dec); insol in w; v sl sol in eth; sl sol in hot alc; sol in hot glac acet ac. It was prepd by heating 2-phenyltetrazole-5-carbonylazide in xylene. Its expl props were not reported
Refs: 1) Beil **26**, [246] 2) R. Stollé & O. Orth, Ber **58**, 2100, 2103(1925) & CA **20**, 763(1926)

p-Diphenyltetrazonium-2,1-naphthol-sulfonate,



$$(HO)H_{10}C_6SO_3-\overset{\overset{N}{|||}}{N}-C_6H_4-C_6H_4-\overset{\overset{N}{|||}}{N}-O_3S.C_6H_{10}(OH)$$

mw 566.65, N 9.89%, brownish-yel powd, mp – explodes; diffc sol in w & neutral solvs; sol in alkalies. It was obtd when benzidine, $H_2N.C_6H_4-C_6H_4.NH_2$, was diazotized and then treated with 2,1-naphthol-sulfonic acid, 2,1-$C_{10}H_6(OH)SO_3H$, in HCl (Ref 2)
Refs: 1) Beil, not found 2) H.T. Bucherer & C. Tama, JPraktChem **127**, 58(1930) & CA **24**, 4280(1930)

NOTE: No nitrated derivs of this compd were found in the literature thru 1966

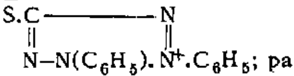
Diphenylthiocarbamide. Same as Diphenylthiourea

N,N'-Diphenylthiocarbazone or **Diphenylsulfocarbazone,**
$C_6H_5.N:N.CS.NH.NH.C_6H_5$; mw 256.32,
or
$C_6H_5.N:N.C(SH):N.NH.C_6H_5$ N 21.86%
dk-violet ndls (from chlf +petr), mp 178°; dec

when heated rapidly & at 169° when heated slowly; insol in w & dil acids; readily sol in org solvs, aliphatic hydrocarbons & in carbon disulfide gives a grn soln. It can be prepd by the action of light on a soln of 1,5-diphenyl-thiocarbohydrazide in aq alcoholic NaOH, and by other methods (Refs 1, 2 & 3)

It forms numerous salts of which the *Lead salt*, $Pb(C_{13}H_{11}N_4S)_2$, red ndls, mp 230°(dec) and *Mercury salt*, crysts, mp 220°(dec) appear to be the least stable

Oxidation of the compd gives the anhydride of 2,3-Diphenyl-5-mercapto-tetrazolium-hydroxide or Diphenyl-tetrazolium-thiobetane,



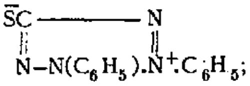
$$\begin{matrix}\bar{S}.C & \text{———} & N \\ \| & & \| \\ N-N(C_6H_5). & & N^+.C_6H_5\end{matrix}$$

; pale yel crysts, exploding at 180.5°, depending on rate of heating (Ref 2)

Refs: 1) Beil **16**, 26 & [10]; **27**, 869 2) E. Bamberger et al, Ann **446**, 270, 290(1926) & CA **20**, 1223(1926) 3) J.H. Billman & E.S. Cleland, OrgSynth **25**, 38(1945)

NOTE: Compare with **Diphenylcarbazone** in this Vol

2,3-Diphenyl-5-thiocarbodiazone (called ''Diphenyltetrazolium-thiobetain'' in Ger). It is the *Anhydride of 2,3-Diphenyl-5-thiotetrazolium Hydroxide* (qv):



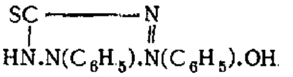
$$\begin{matrix}\bar{S}C & \text{———} & N \\ \| & & \| \\ N-N(C_6H_5). & & N^+.C_6H_5\end{matrix}$$

; mw 254.31, N 22.03%; red ndls (from alc); mp – explodes at 180.5° (depending on rate of heating w/o melting); sol in chlf, concd sulfuric acid, warm w and hot alc; diff sol in eth & benz; insol in alkalies. It was first prepd in 1882 by Fischer & Besthorn (Refs 1 & 2) by heating on a water N,N'-diphenyl-C-mercaptoformazan (listed in Beil **16**, 26),
$C_6H_5.N{:}N.CS.NH.NH.C_6H_5$ or
$C_6H_5.N{:}N.C(SH){:}N.NH.C_6H_5$, with manganodioxyhydrate, $Mn(OH)_3$, in the presence of alcoholic KOH. Another method is to heat N,N'-diphenyl-C-nitroformazan,
$C_6H_5.N{:}N.C(NO_2){:}N.NH.C_6H_5$, in an ethereal soln with isoamylnitrite introduced dropwise

Refs: 1) Beil **26**, 409, [250] 2) E. Fischer & E. Besthorn, Ann **212**, 321(1882) 3) E. Bamberger, Ber **44**, 3745(1911) 4) E. Bamberger et al, Ann **441**, 281–82(1925); **446**, 292(1926) & CA **20**, 1224(1926)

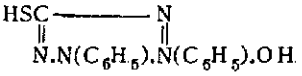
Di(phenylthio)-dimethylbenzene. See Bis-(phenylthio)-dimethylbenzene in Vol 2, p B155-L

2,3-Diphenyl-5-thio-tetrazolium Hydroxide (called 2,3-Diphenyl-5-mercapto-tetrazolium Hydroxid in Ger)



$$\begin{matrix}SC & \text{———} & N \\ | & & \| \\ HN.N(C_6H_5). & & N(C_6H_5).OH\end{matrix}$$

or



$$\begin{matrix}HSC & \text{———} & N \\ \| & & \| \\ N.N(C_6H_5). & & N(C_6H_5).OH\end{matrix}$$

Exists as its *Anhydride.* See Diphenylthiocarbodiazone

Diphenylthiourea and Derivatives

N,N'-Diphenylthiourea, N,N'-Diphenylsulfourea or *Thiocarbanilide,*
$(C_6H_5.NH)_2CS$, mw 228.30; crysts, sp gr 1.32 at 24°, mp 154°, bp – dec; insol in w; v sol in alc & in eth. It can be prepd by the interaction of aniline and carbon disulfide in alc and in the presence of sulfur, and by other methods

It is used as vulcanization accelerator and in flotation (Refs 1 & 3)

N,N-Diphenylthiourea, $(C_6H_5)_2N.CS.NH_2$, flat prisms (from alc), mp 212–13° with frothing to a grn liq. It can be prepd by heating N,N-diphenyl-N'-acetyl-thiourea with alcoholic NaOH (Ref 2)

Refs: 1) Beil **12**, 394, (245) & [227] 2) Beil **12**, 432, (258) & [242] 3) CondChemDict (1961), 1134-R (Thiocarbanilide)

Azido, $C_{13}H_{11}N_5S$, and *Diazido,* $C_{13}H_{10}N_8S$, derivs were not found in the literature thru 1966

Mononitrodiphenylthiourea or **Nitrothiocarbanilide,** $O_2N.C_6H_4.NH.CS.NH.C_6H_5$; mw 273.31, N 15.37%. Three isomers are known:

N-Phenyl-N'-(2-nitrophenyl)-thiourea or *2-Nitrothiocarbanilide*, yel crysts (from glac acet ac), mp 142°; insol in eth; sol in alc & in alkalies with a yel color. It can be prepd by reacting 2-nitrophenyl-mustard oil, $O_2N.C_6H_4.N:CS$, and aniline in the presence of aniline hydrochloride by briefly heating on a w bath (Ref 1)

N-Phenyl-N'-(3-nitrophenyl)-thiourea or *3-Nitrothiocarbanilide*, ndls or yel scales, mp 155° & 160°; sol in warm alc; sl sol in benz, chlf, carbon disulfide & cold alc. It can be prepd by heating 3-nitroaniline & phenyl mustard oil, $C_6H_5N:CS$ (Refs 2, 3 & 4)

N-Phenyl-N'-(4-nitrophenyl)-thiourea or *4-Nitrothiocarbanilide*, crysts, mp 100°. No addn info found in abstract (Ref 5)

Refs: 1) Beil **12**, (343) 2) Beil **12**, 708 & [381] 3) H. Engel & W. Simon, GerP 954988(1956) & CA **53**, 7578(1959) (Use of 3-nitro deriv as an additive to lubricating oil) 4) M.I. Bruce & J.A. Zwar, ProcRoySoc (London), SerB **165** (999), 245(1966) & CA **65**, 12792(1966) 5) J.P. Trivedi et al, JIndianChemSoc **43** (4), 265(1966) & CA **65**, 16958(1966)

Dinitrodiphenylthiourea or **Dinitrothiocarbanilide,** $O_2N.C_6H_4.NH.CS.NH.C_6H_4.NO_2$; mw 318.31, N 17.60%. Two isomers are known:

N,N'-Bis(3-nitrophenyl)-thiourea or *3,3'-Dinitrothiocarbanilide*, yel ndls, mp 160–61° & 168°. It can be prepd from reaction of 3-nitroaniline & carbon disulfide in the presence of sulfuryl chloride, and by other methods (Refs 1, 3, 4, 5, 6 & 8)

N,N'-Bis(4-nitrophenyl)-thiourea or *4,4'-Dinitrothiocarbanilide*, orn-brn crysts, mp 195–96°. It was prepd by heating 2 mols 4-Nitroaniline with 1 mol thiophosgene in water (Refs 2, 7 & 9)

Refs: 1) Beil **12**, 708, (348) & [382] 2) Beil **12**, [393] 3) R.T.C. Loh & W.M. Dehn, JACS **48**, 2958(1926) & CA **21**, 67 (1927) 4) E. Dyer & T.B. Johnson, JACS **54**, 778(1932) & CA **26**, 1587(1932) 5) G.M. Dyson, JChemSoc **1934**, 176 & CA **28**, 2688(1934) 6) P.P.T. Sah & H-H. Lei, JChineseChemSoc **2**, 153(1934) & CA **29**, 461(1935) 7) R. Mecke et al, ZNaturforsch **10b**, 367(1955) & CA **50**, 4650(1956) (IR spectrum) 8) H. Engel & W. Simon, GerP 954988(1956) & CA **53**, 7578(1959) (Use of 3,3'-Dinitrothiocarbanilide as an additive to lubricating oil); GerP 1011107(1957) & CA **54**, 8063(1960) (Ibid) 9) E.P. Nesynov & P.S. Pel'kis, ZhObshchKhim **34**(10), 3469 (1964) & CA **62**, 3960(1965) (Compd not found in abstract)

N-Phenyl-S-picryl-isothiourea, $C_6H_5.NH.C(:NH).S.C_6H_2(NO_2)_3$ or $C_6H_5.N:C(NH_2).S.C_6H_2(NO_2)_3$; mw 363.31, N 19.28%; obtd with other products by uniting of Picryl chloride & N-phenylthiourea in cold acetone or better in glac acet ac. It was isolated as the *Picrate*, mp 82°, explodes at higher temp (Refs 1 & 2)

Refs: 1) Beil **12**, [231] 2) J. Taylor & A.E. Dixson, JChemSoc **125**, 249(1924) & CA **18**, 978(1924) 3) Not found in later refs thru 1966

N,N'-Bis(3,5-dinitrophenyl)-thiourea or **3,5,3',5'-Tetranitrothiocarbanilide,** $(O_2N)_2C_6H_3.NH.CS.NH.C_6H_3(NO_2)_3$; mw 408.31, N 20.58%, yel prisms (from petr eth + glac acet ac), mp 160°. It was prepd by treating 3,5-dinitroaniline with thiophosgene in water for ½ hr (Refs 1 & 2)

Refs: 1) Beil **12**, [414] 2) G.M. Dyson et al, JChemSoc **1927**, 444 & CA **21**, 1637(1927) 3) Not found in later refs thru 1966

Pentanitro, $C_{13}H_7N_7O_{10}S$, and *Hexanitro*, $C_{13}H_6N_8O_{12}S$, derivs of Diphenylthiourea were not found in the literature thru 1966

Diphenyltolylpropane and Derivatives

1,3-Diphenyl-2-p-tolylpropane or *p-Tolyldibenzylmethane*, $CH_3.C_6H_4.CH(CH_2.C_6H_5)_2$, may be considered as the parent compd (Ref 1) of its hexanitro deriv, although not used to prep it:

1,3-Dipicryl-2-p-tolylpropane- 2,4,6,2′,4′,6′-Hexanitrodiphenyltolylpropane or **1,3 Bis-(2,4,6-trinitrophenyl)-2-p-tolylpropane,** $(O_2N)_3C_6H_2.CH_2.CH(C_6H_4.CH_3).CH_2.C_6H_2(NO_2)_3$; mw 556.40, N 15.11%, citron-yel ndls (from glac acet ac), turns brn in the light, mp 183–86°. It was prepd by reaction of p-toluylaldehyde, $CH_3.C_6H_4.CHO$, & 2,4,6,-TNT in pyridine in the presence of piperidine at RT (Refs 2 & 4). No expl props are reported.

Refs: 1) Beil **5**, not found (Parent) 2) Beil **5**, [624] 3) I.A. Pastak, BullFr **39**, 77(1926) & CA **20**, 3000(1926)

NOTE: No lower nitrated derivs of Diphenyltolylpropane were found in the literature thru 1966

1,3-Diphenyltriazene is described in this Vol, p D1156-R, as **Diazoaminobenzene**

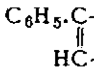
It was described and code named **PAD-2** in ADL, SynthHE's, 4th Rept (1956), p 107

Di(phenyltriazeno)-benzene. See 1,3-Bis-(phenyltriazeno)-benzene in Vol 2, p B155-R

Diphenyltriazole and Derivatives

Diphenyltriazole, $C_{14}H_{11}N_3$; mw 221.25, N 18.99%. Seven isomers are found in Beil:

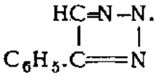
1,5-Diphenyl-1,2,3-triazole,



$$\begin{array}{l}C_6H_5.C{-}N(C_6H_5){-}N\\ \quad\ \| \qquad\qquad\quad \|\\ \quad HC{-\!-\!-\!-\!-\!-}N\end{array}$$

; tablets (from alc), mp 113–14°(Ref, p 68)

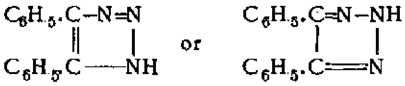
2,4-Diphenyl-1,2,3-triazole,



$$\begin{array}{l}\quad HC{=}N{-}N.C_6H_5\\ \quad\ |\qquad |\\ C_6H_5.C{=\!=}N\end{array}$$

; pltlts or ndls (from alc), mp 56–57°(Ref, p 34)

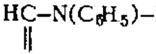
4,5-Diphenyl-1,2,3-triazole,



$$\begin{array}{lcl}C_6H_5.C{-}N{=}N & & C_6H_5.C{=}N{-}NH\\ \quad\ \|\quad\ | & \text{or} & \qquad\quad\ |\quad\ |\\ C_6H_5C{-\!-}NH & & C_6H_5.C{=\!=}N\end{array}$$

; ndls (from dil alc), mp 139°. It forms a Silver salt, $AgC_{14}H_{10}N_3$, light-sensitive powd (Ref, p 79)

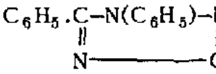
1,3-Diphenyl-1,2,4-triazole,



$$\begin{array}{l}HC{-}N(C_6H_5){-}N\\ \ \|\qquad\qquad\ \|\\ N{-\!-\!-\!-\!-\!-}C.C_6H_5\end{array}$$

; ndls (from dil alc), mp 96–97°, distills undecomposed. Forms a Hydrochloride salt, ndls (from eth acet), mp 176–80°(dec); a Chloroplatinate, dk-yel tablets(from alc), mp 218°(dec); and a Picrate, tablets (from alc), mp 148°(Ref, p 68)

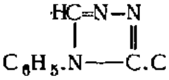
1,5-Diphenyl-1,2,4-triazole,



$$\begin{array}{l}C_6H_5.C{-}N(C_6H_5){-}N\\ \qquad\ \|\qquad\qquad\ \|\\ \qquad N{-\!-\!-\!-\!-\!-}CH\end{array}$$

; ndls (from w or dil. alc), prisms or scales (from petr eth), mp 91°, bp – sublimes, volatile on steam bath. It forms salts (Ref, p 68)

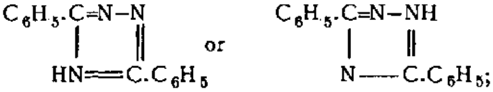
3,4-Diphenyl-1,2,4-triazole,



$$\begin{array}{l}\qquad HC{=}N{-}N\\ \qquad\ |\qquad \|\\ C_6H_5.N{-\!-}C.C_6H_5\end{array}$$

; crysts (from dil alc or petr eth), mp 142°. It forms a Pt chloride salt, $2C_{14}H_{11}N_3{+}2HCl{+}PtCl_4$, yel crysts, decomp 160° & a Picrate, $C_{14}H_{11}N_3{+}C_6H_3N_3O_7$, yel crysts, mp 174°(Ref, p 69)

3,5-Diphenyl-1,2,4-triazole,



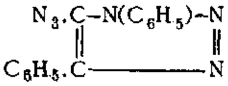
$$\begin{array}{lcl}C_6H_5.C{=}N{-}N & & C_6H_5.C{=}N{-}NH\\ \qquad\ |\quad \| & \text{or} & \qquad\quad\ |\quad \|\\ HN{=\!=}C.C_6H_5 & & \qquad N{-\!-}C.C_6H_5\end{array}$$

; prisms or pltlts + water (from dil alc or benz), mp 190–92°(becoming anhyd), bp – dec at 280°. It forms a Silver addn compd, $AgC_{14}H_{10}N_3{+}C_{14}H_{11}N_3$, ndls (from dil HNO_3), mp – softens at 140° & dec ca 170°; and a *Silver salt,* $AgC_{14}H_{10}N_3$, crysts, mp – dec 246°(Ref, pp 81 & 21)

Other props & methods of prepn are found in Ref

Refs: Beil **26**, 68, 69, 79, 81, (21) & [34]

5-Azido-1,4-diphenyl-1,2,3-triazole,



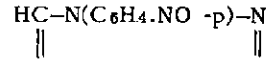
$$\begin{array}{l}\quad N_3.C{-}N(C_6H_5){-}N\\ \qquad\ \|\qquad\qquad\ \|\\ C_6H_5.C{-\!-\!-\!-\!-\!-}N\end{array}$$

; mw 262.27, N 32.05%; nearly colorless ndls (from acet + w or dimethylformamide + w, or from cold eth), mp 70°(dec) forming a blood-red melt with foaming; in inert solvs its soln is red and evolves N gas at temps as low as 50°. It was synthesized from 1,4-diphenyl-5-aminotetrazole in HCl by diazotizing it with Na nitrite in water at 0°(Ref 2)

Refs: 1) Beil, not found 2) P.A.S. Smith et al, JACS **86**, 2025, 2029(1964) & CA **61**, 654(1964)

Mononitrophenyl-phenyl-triazole, $C_{14}H_{10}N_4O_2$, mw 266.25, N 21.04%. Several isomers are described in the literature:

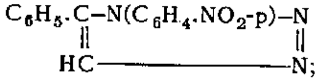
1-(4-nitrophenyl)-4-phenyl-1,2,3-triazole,



$$\begin{array}{l} HC{-}N(C_6H_4.NO\ \text{-p}){-}N \\ \| \qquad\qquad\qquad \| \\ C_6H_5.C{-}{-}{-}{-}{-}N \end{array}$$

; yel pltlts (from DMF + alc), mp 252–53°; prepd from 4-nitrophenylazide & phenylacetylene in toluene by refluxing for 24 hrs (Ref 9)

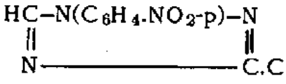
1-(4-nitrophenyl)-5-phenyl-1,2,3-triazole,



$$\begin{array}{l} C_6H_5.C{-}N(C_6H_4.NO_2\text{-p}){-}N \\ \quad \| \qquad\qquad\qquad \| \\ \quad HC{-}{-}{-}{-}{-}N \end{array}$$

; lt-yel pltlts (from benz + petr eth), mp 163–164.5° (Ref 9) & 159° (Ref 5); was prepd by refluxing 4-nitrophenylazide & benzoylethylacetate, cooling & decarboxylating the soln with acetonitrile (Ref 9)

1-(4-nitrophenyl)-3-phenyl-1,2,4-triazole,



$$\begin{array}{l} HC{-}N(C_6H_4.NO_2\text{-p}){-}N \\ \| \qquad\qquad\qquad \| \\ N{-}{-}{-}{-}{-}C.C_6H_5 \end{array}$$

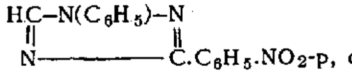
; crysts (from benz + petr eth), mp 202–03°; prepd by treating N-(4-nitrophenyl)-C, N'-diphenylformazan in DMF with 2N NaOH & aq CH_2O, heating the soln, and treating again with 2N NaOH, heating, diluting with water, and pptg repeatedly with DMF (Ref 7)

3-(4-nitrophenyl)-5-phenyl)-1,2,4-triazole,

$$\begin{array}{l} C_6H_5.C{=}N{-}NH \\ \quad | \qquad | \\ \quad N{=}C.C_6H_4.NO_2\text{-p} \end{array}$$

, wh ndls (from methanol or acet & then petr eth), mp 240°. It was obtd when phenyl cyanide & p-nitrobenzhydrazide benzenesulfonate were heated at 200° for 3 hrs, cooled & the crystalline melt extracted with cold NaOH soln & the extract acidified with concd HCl (Ref 2) and by other methods (Refs 3, 4, 6 & 8)

3-(4-nitrophenyl)-1-phenyl-1,2,4-triazole,

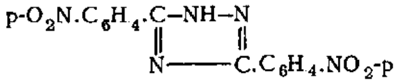


$$\begin{array}{l} HC{-}N(C_6H_5){-}N \\ \| \qquad\qquad \| \\ N{-}{-}{-}{-}C.C_6H_5.NO_2\text{-p} \end{array}$$

, crysts (from benz), mp 188–89°; prepd by treating C-(4-nitrophenyl)-N,N'-diphenylformazan in DMF with 2N NaOH & aq CH_2O for 6 hrs at 20° and diluting with water (Ref 7)

Refs: 1) Beil, not found 2) K.T. Potts, JChemSoc **1954**, 3462–63 & CA **49**, 13227(1955) 3) H. Weidinger & J. Kranz, GerP 1076136 (1960) & CA **55**, 13450(1961) (Prepn) 4) J. Kranz & H. Weidinger, Festschrift Carl Wurster zum 60. Geburtstag **1960**, 119 & CA **56**, 10132(1962) 5) R. Fusco et al, Gazz **91**, 864(1961) & CA **56**, 14020(1962) 6) H. Weidinger & J. Kranz, Ber **96**, 1064(1963) & CA **59**, 6387(1963) 7) R. Kuhn & H. Trischmann, Monatsh **95**(2), 457(1964) & CA **61**, 5626(1964) 8) H. Eilingsfeld, ChemBer **98**(4), 1308(1965) (Ger) & CA **62**, 16232(1965) 9) R. Huisgen et al, ChemBer **98**(4), 1146–47 (1965) & CA **62**, 16247–48(1965)

3,5-Bis(4-nitrophenyl)-1,2,4-triazole,

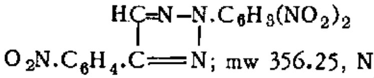


$$\begin{array}{l} p\text{-}O_2N.C_6H_4.C{-}NH{-}N \\ \qquad\qquad \| \qquad \| \\ \qquad\qquad N{-}{-}{-}C.C_6H_4.NO_2\text{-p} \end{array}$$

; mw 311.25, N 22.52%, fawn ndls (from aq alc), mp 250–51°. It was obtd when p-nitrophenyl cyanide & p-nitrobenzhydrazide benzenesulfonate were heated at 200° for hrs, and the cryst product was extracted with cold NaOH & the extract acidified with cold HCl (Ref 2). No expl props are reported

Refs: 1) Beil, not found 2) K.T. Potts, JChemSoc **1954**, 3462 & CA **49**, 13227(1955) 3) Not found in later refs thru 1966

Trinitro-2,4-diphenyl-2,1,3-triazole,

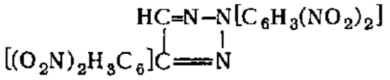


$$\begin{array}{l} \qquad\qquad HC{=}N{-}N.C_6H_3(NO_2)_2 \\ \qquad\qquad\ | \qquad | \\ O_2N.C_6H_4.C{=}N \end{array}$$

; mw 356.25, N 23.59%, crysts (from acet ac), mp 238–39°. It was obtd on long standing of the mother liquor resulting from refluxing 2,4-diphenyl-2,1,3-triazole & nitric acid & pptg the tetranitro deriv by pouring into wwter (Ref 2). No expl props are reported

Refs: 1) Beil, not found 2) E. Ghigi & T. Pozzo-Balbi, Gazz **71**, 228(1941) & CA **36**, 2863(1942) 3) Not found in later refs thru 1966

Tetranitro-2,4-diphenyl-2,1,3-triazole or **2,4-Bis(dinitrophenyl)-2,1,3-triazole,**



$$\begin{array}{l} HC{=}N{-}N[C_6H_3(NO_2)_2] \\ [(O_2N)_2H_3C_6]C{=}N \end{array}$$

mw 401.25, N 24.44%, straw colored crysts (from acet ac), mp 178–79°. It can be prepd by treating 2,4-diphenyl-2,1,3-triazole or its trinitro deriv with nitric acid (d 1.52), refluxing for 3 hrs and pouring into water to ppt product (Ref 2). Its expl props are not reported

Refs: 1) Beil, not found 2) E. Ghigi & T. Pozzo-Balbi, Gazz **71**. 228(1941) & CA **36**, 2862(1942) 3) Not found in later refs thru 1966

NOTE: Penta, $C_{14}H_6N_8O_{10}$, and *Hexa,* $C_{14}H_5N_9O_{12}$, nitro derivs of Diphenyltetrazole were not found in the literature thru 1966.

N,N′-Diphenyl-trimethylenediamine. See 1,3-Bis(anilino)-propane in Vol 2, p B132-R

asym-**Diphenylurea** or **N,N-Diphenylurea.** See Acardite 1 in Vol 1, p A7-R

sym-**Diphenylurea; N,N′-Diphenylurea** or **Carbanilide.** See N,N′-Bis(phenyl)-urea and Derivatives in Vol 2, p B155-R

In the description of **N,N′-Bis(2,4,6-trinitrophenyl) -urea** or **Dipicrylurea** given on p B156-L there was not included the information on explosive properties given in A.H. Blatt, OSRD Rept **2014**(1944) and ADL PureExplCompds, Pt 1 (1947), because these, originally conf repts, were not declassified at the time of compiling Vol 2 of Encycl

The props given in Blatt and in ADL repts are as follows:

Brisance by Sand Test – 0.4g crushed 36.6g sand vs 48.0 by TNT
Explosion Temperature – 345°
Impact Sensitivity – comparable with Tetryl
Initiation – 0.4g required 0.19 MF
Power by Ballistic Mortar – 76% TNT
Power by Trauzl Test – 80% TNT
Stability, Chemical – unstable; decompd by moisture
Stability, Thermal – good

Diphenylurethane and Derivatives

Diphenylurethane or *N,N-Diphenyl-ethyl-carbamate,* $(C_6H_5)_2N.CO_2C_2H_5$; mw 241.28, N 5.81%; prisms (from ligroin), mp 72°, bp >360°; sol in w, petr eth & benz; v sol in alc & eth (See also Refs 1 & 3). Schmidt (Ref 2) gave its heat of combstn as 7697 cal/g at C_v and heat of formation 287cal/g at C_v. Tavernier (Ref 4) gave heat of formation at C_v as 245cal/g and 267 at C_p

Diphenylurethane was prepd by heating diphenylamine with 1 mol of the ethyl ester of formic acid in solvent naphtha or xylene (Ref 1)

It was used by the Germans during WWII as a stabilizer of NGu (Nitroguanidine) contg propellants, known as *Gudolpulvern* (Ref 5, p Ger 37-L & Ger 81)

Refs: 1) Beil. **12**, 427, (253) & [240] 2) A. Schmidt, SS **29**, 263(1934) 3) R. Dalbert & J. Tranchant, MP **30**, 338(1948) 4) P. Tavernier, MP **38**, 306, 328(1956) 5) Fedoroff et al, PATR **2510** (1958), pp Ger 37-L & Ger 81

NOTE: Azido, $C_{15}H_{14}N_4O_2$, and *Diazido,* $C_{15}H_{13}N_7O_2$, derivs of Diphenylurethane were not found in Beil.

Mononitrodiphenylurethane, $C_{15}H_{14}N_2O_4$, mw 286.28, N 9.79%, Only one isomer is found in Beil

4-Nitrodiphenylurethane,
$O_2N.C_6H_4.N(C_6H_5).CO_2.C_2H_5$, prisms (from alc), mp 68°; readily sol in eth, alc, chlf, benz & acet; sl sol in petr eth It was prepd by treating diphenylurethane with cold nitric acid (d 1.42) or with 3 mols fuming nitric acid in chlf (Refs 1 & 2)

Refs: 1) Beil **12**, [394] 2) H. Ryan & A. Donnellen, SciProcRoyDublinSoc **17**, 114, 116 (1923) & CA **17**, 1791(1923)

Dinitrodiphenylurethane, $C_{15}H_{13}N_3O_6$, mw 331.28, N 12.69%. Two isomers are found in Beil:

2,4′-Dinitrodiphenylurethane,
$(O_2N.C_6H_4)_2N.CO_2.C_2H_5$, syrup, fr p – not reported; sol in alc & benz; obtd with the

4,4′-dinitro deriv on treating diphenylurethane with fuming nitric acid in carbon tetrachloride or with N_2O_4 in CCl_4 (Refs 1 & 2)
4,4′-Dinitrodiphenylurethane, yel ndls (from alc), mp 133–34°; readily sol in benz, acet, eth & chlf; sl sol in alc; almost insol in petr eth (Refs 1 & 2). Its prepn is given above
Refs: 1) Beil **12**, 724 & [394] 2) H. Ryan & A. Donnellen, SciProcRoy DublinSoc **17**, 115(1923) & CA **17**, 1791(1923)

Trinitrodiphenylurethane, $C_{15}H_{12}N_4O_8$; mw 376.28, N 14.89%. Not found in the literature thru 1966

2,4,2′,4′-Tetranitrodiphenylurethane or **Bis(2,4-dinitrophenyl)-ethyl-carbamate,** $[(O_2N)_2C_6H_3]_2N.CO_2.C_2H_5$; mw 421.28; N 16.63%, yel prisms (from chlf & alc), mp 184–85°; sol in chlf, benz & alc; sl sol in petr eth. It was prepd by nitrating diphenylurethane or 4-Nitrodiphenylurethane with mixed nitric-sulfuric acids (Refs 1 & 2). No expl props reported
Refs: 1) Beil. **12**, [412] 2) H. Ryan & A. Donnellen, SciProcRoyDublinSoc **17**, 115, 117(1923) & CA **17**, 1791(1923) 3) Not found in later refs thru 1966

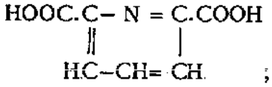
NOTE: Pentanitro, $C_{15}H_{10}N_6O_{12}$, and *Hexanitro*, $C_{15}H_9N_7O_{14}$, derivs of Diphenylurethane were not found in the literature thru 1966

Diphenylylamine; Xenylamine or **Phenylaniline.** See Aminobiphenyl in Vol 1, p 191-L

Diphosgene. One of the Chemical Warfare Agents, code named **DP**, briefly described in Vol 2, p C167-R

Diphthalic Acid Peroxide or **Diphthaloyl Peroxide.** See Bis(phthalic acid)-peroxide in Vol 2, p B156-L

Dipicolinic Acid and Derivatives
Dipicolinic Acid or Pyridine-2,6-dicarboxylic Acid,



$$\begin{array}{l} HOOC.C-N=C.COOH \\ \qquad\ \ \ \| \qquad\quad\ | \\ \qquad HC-CH=CH \end{array}$$

; mw 167.12, N 8.38%, prisms + water or hair-shaped ndls + 1½ water, mp 226–37°(dec); sl sol in w, alc, eth & glac acet ac. It was prepd by oxidation of 2,6-dimethylpyridine with K permanganate or by oxidation of 6-methylpyridine-2-carboxylic acid. It forms numerous crystalline addn salts with metals (Refs 1 & 3)

Dipicolinic Acid Diazide or **Di(picolinoyl) diazide,** $C_5H_3.N[CO(N_3)_2]_2$; mw 217.15 N 45.16%, prisms (from ether), mp 110–11° (dec, followed by expln); very irritating to mucous membranes. It was prepd by action of Na nitrite & cold dil HCl on dipicolinic acid dihydrazide, $NC_5H_3(CO.NH.NH_2)_2$ (Refs 2 & 3)
Refs: 1) Beil **22**, 154, (534) & [106] 2) Beil **22**, (534) (Diazide) 3) H. Meyer & J. Mally, Monatsh **33**, 403, 406(1912) & CA **6**, 2073(1912)

Di(β-picolino)-diazido-copper Complex, $[(N_3)_2Cu(C_6H_7N)_2]$; mw 270.29, N 41.46%; solid; mp – exploded violently when dropped on a metallic block preheated to 207–15°; it burned in flame with crepitation and exploded by impact of 1 kg weight dropped from 80cm height. It was prepd by Cirulis & Straumanis (Ref 2) by addg NaN_3 to a dipicolinic acid soln of the Cu^{++} salt (Ref 2)
Refs: 1) Beil, not found 2) A. Cirulis & M. Straumanis, JPrakChem **162**, 311(1943) & CA **38**, 1969(1944)

Dipicrylamine. Same as 2,4,6,2′,4′,6′-Hexanitrodiphenylamine described under DIPHENYLAMINE AND DERIVATIVES in this Volume

Di(picrylamino)-anthraquinone. See Bis-(trinitroanilino)-anthraquinone under Bis-(anilino)-anthraquinone in Vol 2, p B131-L

Dipicrylaminoethyl Nitrate. See under Diphenylaminoethyl Nitrate in this Vol

Dipicrylarginine. See under Diphenyl(amino-4-guanidino-valeric Acid) and Derivatives in this Vol

Dipicrylcarboxamide-acetylene. See under Di(N-phenylcarboxamide)-acetylene and Derivatives

Dipicryldianthranilide. See under Diphenyldianthranilide and Derivatives in this Vol

Dipicryldisulfide. See under Diphenyldisulfide and Derivatives in this Vol

Dipicrylethane. See 2,4,6,2′,4′,6′-Hexanitro-sym-diphenylethane, Vol 2, p B111-L, under Bibenzyl and Derivatives

Dipicrylethanol. See under Diphenylethylol

Dipicrylether. See under Diphenylethers

N,N′-Dipicryl-ethylenediamine. See 1,2-Bis(2′,4′,6′-trinitroanilino)-ethane in Vol 2, p B131-R

N,N′-Dipicryl-ethylenedinitramine or **N,N′-Dinitro-N,N′-bis(2,4,6-trinitrophenyl)-ethylenediamine** (Code named in US **Bitetryl** or **Ditetryl** and in GtBrit **Octyl**). See 1,2-Bis(2′,4′,6′-trinitroanilino)-ethane in Vol 2, pB131-R and under Diphenylpiperazine in this Vol

Dipicrylglycerylether Nitrate. See under Diphenylglycerylether and Derivatives

Dipicrylguanidine. See under Bis(phenyl)-guanidine in Vol 2, p B154-L

Dipicrylmethane. See under Diphenylmethane and Derivatives

Dipicryl-methylamine. See under Diphenylmethylamine and Derivatives

Dipicrylmethylenedihydroxyphenylpropane. See under Diphenylmethylenedihydroxyphenylpropane and Derivatives

Dipicryloxamide. See under Diphenyloxamide and Derivatives

Dipicrylphenylenediamine and its **5-Nitro Derivative.** See under Diphenylphenylenediamine and Derivatives

Dipicrylpiperazine. See under Diphenylpiperazine and Derivatives

Dipicrylpropylenediamine (Code named **Methylene Ditetryl).** See under Bis(anilino)-propane in Vol 2, p B133-L

Dipicrylselenide. See under Diphenylselenide and Derivatives

Dipicrylsulfide. See under Diphenylsulfide and Derivatives

Dipicrylsulfone. See under Diphenylsulfone and Derivatives

Di(picrylthiol)-ethylbenzene. See 2,4-Bis-(2′,4′,6′-trinitrophenylthio)-1-ethylbenzene in Vol 2, p B155-R

Di(picrylthiol)-xylene. See Bis(2,4,6-trinitrophenylthio)-dimethylbenzene in Vol 2, p B155-L

Dipicryltolylpropane. See under Diphenyltolylpropane and Derivatives

sym-**Dipicrylurea** or **Hexanitrocarbanilide.** See N,N′-Bis(2,4,6-trinitrophenylurea) in Vol 2, p B156-L and under sym-Diphenylurea in this Vol

Dipiperidine or Bipiperidine and Derivatives
Dipiperidine, $HN.H_9C_5.C_5H_9NH$; mw 168.28, N 16.65%. The 2,2′-, 2,3′-, 3,3′-, 3,4′- & 4,4′-derivs are found in the literature (Ref 1)

Dipiperidino-diazido-copper, $[(N_3)_2Cu(C_5H_{11}N)_2]$, mw 317.88, N 35.25; solid, mp 109°, decomp 160–65°. It was prepd by addg NaN_3 to a dipiperidine soln of the Cu^{++} salt (Ref 4)

Dipiperidino-tetrazido-cuprate or **Dipiperidine-tetrazido-copper,** $(C_5H_{12}N)_2[Cu(N_3)_4]$; mw 403.94, N 48.54%; solid; mp 125°, bp – expl at 200–205° when thrown on the preheated metallic block; burns in flame with a crackling noise; very insensitive to impact (100cm with 1 kg wt). It can be prepd by dissolving Cu azide in dipiperidine (Ref 4)

Dinitrosodipiperidine, $ON.NC_5H_9.C_5H_9.NO$; mw 226.28, N 24.76%. Five derivs are known:
1,1′-Dinitroso-2,2′-dipiperidine, crysts (from alc), mp 159° (Ref 3, p37)
1,1′-Dinitroso-2,3′-dipiperidine, crysts (from eth + a little alc), mp 88° (Ref 3, pp 38 & 32)
1,1′-Dinitroso-3,3′-dipiperidine, crysts, mp 102° (Ref 3, p 32)
1,1′-Dinitroso-3,4′-dipiperidine, crysts, mp 149° (Ref 3, p 32)
1,1′-Dinitroso-4,4′-dipiperidine, yel crysts (from alc), mp 141–43° & 150° (Ref 3, pp 38 & 33)
Refs: 1) Beil **23**, 37, 38, (15) & [31,32]
2) Beil, not found (Diazido-copper & Tetrazido-cuprate) 3) Beil **23**, 37,38 & [32, 33]
4) A. Cirulis & M. Straumanis, JPraktChem **162**, 312(1943); Ber **76B**, 825(1943) & CA **38**, 1969 (Diazodo-Cu) & 1971 (Tetrazido-Cu) (1944)

NOTE: No nitro derivs found in the literature thru 1966

Diplosal or **Salysal.** See Disalicylic Acid in this Vol

Diplumbic Dinitroresorcinol. See Basic Lead Salt of 4,6-Dinitro-1,3-dihydroxybenzene or Lead 4,6-Dinitroresorcinate, p D1275-R

Dipole Moment (Moment électrique in French) The *dipole* of a neutral diatomic molecule consists of two equal electrical charges a short distance apart and opposite in sign. Its magnitude is measured by its *moment*, which is the product of either charge times the distance between the two charges. Since the moment acts in the direction of the line joining the two charges (that is the axis of the dipole), it is a vector quantity. The hydrogen chloride molecule, HCl, is a simple example of a molecular dipole which, in this case, lies along the intermolecular axis with its positive & negative ends toward H & Cl, respectively

In general, a polyatomic molecule has a dipole moment when the centroid of positive charges, due to atomic nuclei, is displaced with respect to the centroid of negative charges due to the electrons

Dipole moments have been employed principally in the determination of the shapes of molecules and their electron distributions. Various experimental procedures for determining dipole moment are discussed in Ref 10, and an extensive tabulation of values is given in Ref 7. The accompanying table lists experimental & theoretical values for a variety of expl molecules. The theoretical values were obtd via quantum mechanical procedures

Dipole Moments of Explosive Molecules

Inorganic Compds	State or Solvent	Expt'l Dipole Moment Debyes	Ref	Theoretical Dipole Moment Debyes	Ref
Hydrogen Azide *	gas	0.84	5, 8	1.08**	11
Hydrogen Fulminate	gas	3.06	9	2.58**	11
Nitroaliphatic Compd					
Nitromethane	gas	3.54	10		
	benzene	3.13	10		
Nitroaromatic Compds					
Nitrobenzene	gas	4.2	10		
	benzene	4.0	10		
o-Dinitrobenzene	–	6.0	10		
m-Dinitrobenzene	–	3.88	10		
p-Dinitrobenzene	–	0.00	10		
2,3-Dinitrotoluene	benzene	5.81	7		
2,4-Dinitrotoluene	benzene	4.33	7		
2,5-Dinitrotoluene	benzene	0.58	7		
2,4,6-Trinitrotoluene	benzene	1.16	7		
2,4,6-Trinitroanisole	benzene	2.00	7		
Nitramine Compds					
Dimethylnitramine	dioxane	4.61	7		
RDX (Cyclotrimethylene Trinitramine)	dioxane	5.78	7	5.48***	6
Nitrate Compds					
PETN (Pentaerythritol Tetranitrate)	dioxane	2.48	7		
NG (Nitroglycerin or Glycerol Trinitrate)	benzene	3.38	7		

* The experimental & theoretical values refer to the component of the dipole moment along the unique axis

** Calcd in an exact manner via the Iterative Extended Hückel Method in quantum mechanics

*** Calcd via the Intermediate Neglect of Differential Orbital Method in quantum mechanics

See also Dielectrics and Dielectric Constants in this Vol, p D1222-R

Refs: 1) E. Errera, "Le moment électrique en chimie et en physique, Le moment électrique et structure moléculaire", Hermann, Paris (1935) 2) L. Pauling, "The Nature of the Chemical Bond", Cornell Univ Press, Ithaca (1940) 3) R.J.W. Le Fèvre, "Dipole Moments; Their Measurements and Application", Methuen, London (1948) 4) A. Weissberger, "Physical Methods of Organic Chemistry", Interscience, NY, Vol 1(1949), pp 1611–50, Article by C.P. Smyth, "Determination of Dipole Moments and of Dielectric Constants" (60 refs) 5) E. Amble & B.P. Dailey, JChemPhys **18**, 1422(1950) 6) M.V. George & V.F. Wright, JACS **80**, 1200(1958) 7) A.L. McClellan, "Tables

of Experimental Dipole Moments", Freeman & Co, San Francisco, Calif (1963) 8) K.J. White & R.L. Cook, JChemPhys **46**, 143(1966) 9) M. Winnewisser & H.K. Bodenseh, ZNaturforsch **22A**, 1724(1967) 10) V.I. Minkin, O.A. Osipov & Y.A. Zhdanov, "Dipole Moments in Organic Chemistry", English translation by B.J. Hazzard, Plenum Press, NY(1970) 11) J. Alster, Molecular Physics **23**, 1235(1972)

Dipping. A process applied to treatment of ammunition and its component metal parts in an effort to protect & prevent corrosion of the surfaces. Various chromate dippings (such as Cronak, Irridite, Yellow & Black Anodize have been used especially when plating is first applied. The usual requirement of a dipping process is that the 24-hour salt spray test must be met
Ref: Ohart (1946), 14

Dipping of Cellulose in Tanks used in DuPont's Mechanical Dipper Process of Manufacture of Nitrocellulose is briefly described in Vol 2 of Encycl, p C102-R, under "Industrial Manufacture of NC". Schematic arrangement of DuPont's Process is shown in Vol 3, p C539 (See also "Direct Dipping Process or Nobel's Method of Manufacture of Cordite")

Dipotassium Hydrazobenzene-Azobenzene. See Azobenzene-Dipotassium Hydrazobenzene in Vol 1, p A647-L

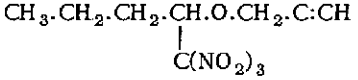
Dipotassium Nitroacetate (DKNA). See under Acetic Acid in Vol 1, p A27-R

Dipropanol-amine or Bis(hydroxypropyl)-amine. See Di(propylol) -amine

Di(iso-propanol)-amine. See Di(iso-propyl)-amine

Di(propanol)-ethylenediamine. See Di(propylol)-ethylenediamine

Dipropargylbutyral-nitroform Product,



$$CH_3.CH_2.CH_2.\underset{\displaystyle |\atop \displaystyle C(NO_2)_3}{CH}.O.CH_2.C{:}CH$$

mw 261.19, N 16.09%; solid; a sensitive and powerful explosive; unstable in storage giving off nitrogen oxides. It can be prepd by action of nitroform on dipropargyl butyral. The product is **1,1,1-Trinitro-2-pentyl-propargyl Ether** (Ref 2)
Refs: 1) Beil, not found 2) R.A. Odum et al, US Rubber Co Final Rept, Oct 24–June 15, 1956. Contract **NORD 10129**. Attached Rept by C.S. Rondestvedt Jr, EngrgResInst, Univ of Michigan, Ann Arbor, Final Rept (May 1956, p 9)

Dipropionylperoxide or **Propionic Peroxide,** $CH_3.CH_2.CO{-}O.O{-}CO.CH_2.CH_3$; mw 146.14, O 43.79%; fr p remained liq at minus 20°, bp – decomp at 80°; explodes on rapid heating above 80°; sol in w (with slow hydrolysis to propionic acid); sol in ordinary org solvents. It can be prepd by the action of Ba peroxide on an ethereal soln of propionic aldehyde at ca 0°. Another method is electrochemical oxidation on a platinum anode of a soln of propionic acid or its salts
Refs: 1) Beil **2**, 243, (108) & {540} 2) A.M. Clover & G.F. Richmond, AmChemJ **29**, 191 (1903) & JCS **84**, I, 396(1903) 3) F. Fichter & E. Krummenacher, Helv **1**, 153(1918) & JCS **114**, I, 369(1918) 4) Kirk & Othmer **10**(1953), 73; 2nd edit **14**(1967), p 801(Table)

Di-n-propyladipate, $H_7C_3O_2C(CH_4)_4CO_2C_3H_7$; mw 230.30, colorless liq, sp gr 0.979 at 20/4°; fr p minus 20.3°, bp 143–145° at 10mm Hg; insol in w; sol in alc & in eth. It can be prepd, in addn to Monopropyladipate, by cautiously heating adipic acid & propyl alcohol in the presence of sulfuric acid. They have been used as a plasticizer in rocket proplnts Unit Nos 386, 390, 391 & 550 described in conf Manual (Ref 2)

Refs: 1) Beil **2**, [574] & {1713} 2) Proplnt-Manual SPIA/M2(1962)(C)

Dipropylamine and Derivatives

Di-n-propylamine, $C_3H_7.NH.C_3H_7$, mw 101.19, N 13.84%; colorless liq, sp gr 0.722 at 22°, fr p minus 61°, bp 83.5° at 743mm; very flammable; sol in w and in alc. It can be prepd by introducing propyl alcohol vapor & ammonia over silica gel at ca 420°, or by reduction of propionaldiazine with Al amalgam in dil alc & a little propylamine (Ref 1). It forms numerous addn salts with both org & inorg compds, some of which are unstable when heated:

Azide, $C_6H_{15}N.HN_3$, leaflets (thru sublimation), mp 101°

Hydrogen Iodide, $C_6H_{15}N+HI$, ndls (from alc), mp 230–35°(dec)

Perchlorate, $C_6H_{15}N+HClO_4$, crysts, explodes at 308°

Tetraiodoethane, $C_6H_{15}N+2I_2C.C.I_2$, ndls or pltlts (from chlf), sinters at 116°

Di(n-propylamino)-diazido-copper,
$[(N_3)_2Cu(C_3H_7NH_2)_2]$, mw 265.81, N 42.16%; green crysts; mp 109–110°; bp — expl when dropped on metallic block preheated to 187°; burns in flame with noise. It can be prepd by dissolving $Cu(N_3)_2$ in di-n-propylamine soln (Ref 3). Does not expl by impact

Refs: 1) Beil **4**, 138, (362), [622] & {255} 2) Beil, not found (Diazido-copper) 3) A. Cirulis & M. Straumanis, JAnorgChem **251**, 346(1943) & CA **37**, 6574(1943)

Azido, $C_6H_{14}N_4$, and *Diazido*, $C_6H_{13}N_7$, derivs of Dipropylamine, were not found in the literature thru 1966

N-Nitrosodipropylamine or *Dipropylnitrosamine*, $H_5C_2.CH_2.N(NO).CH_2.C_2H_5$; mw 130.19, N 21.52%; lt-yel oil, fr p — not reported, bp 89° at 13mm press; sp gr 0.9160 at 20° & 0.8624 at 86.9°; n_D 1.4437 at 20°; almost insol in w. It was prepd by action of HNO_2 on dipropylamine and by other methods. It forms a salt, $2C_6H_{14}N_2O+2HBr+PtBr_4$, red prisms (from alc), mp — not reported (Refs 1 & 2)

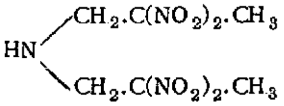
Refs: 1) Beil **4**, 146, (367), [628] & {264} 2) A.I. Vogel, JChemSoc **1948**, 1852

N-Nitrodipropylamine or *Dipropylnitramine*, $H_5C_2.CH_2.N(NO_2).CH_2.C_2H_5$; mw 146.19, N 19.17%; oil, fr p 1.0–1.6°, bp 103–04° at 10mm press, n_D 1.4559 at 20°; stable in 70% sulfuric acid at 25° It was prepd by nitrating di-n-propylamine with 99% nitric acid in the presence of acetyl chloride & acetic anhydride in glac acet ac (Refs 1 & 2)

Refs: 1) Beil **4**, {264} 2) W.R. Kingdom & G.F. Wright, JACS **72**, 1031(1950)

Dinitro, $C_6H_{13}N_3O_4$, and *Trinitro*, $C_6H_{12}N_4O_6$, derivs of Di-n-propylamine were not found in the literature thru 1966

2,2,2',2'-Tetranitro-dipropylamine or **Bis(2,2-dinitropropyl)-amine,**



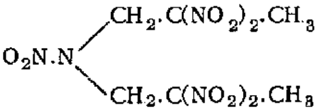
$$HN\begin{cases}CH_2.C(NO_2)_2.CH_3\\CH_2.C(NO_2)_2.CH_3\end{cases}$$

mw 281.19, N 24.91%; crysts (from isopropyl alc), mp 66–68°. It was obtd in 90.4% yield on condensing 2,2-dinitro-1-propanol in water with Amm acetate at 60° for 20 mins, cooling & filtering the pptd product (Ref 2). This compd was patented for use as an expl, or as an O donor in propellant fuels (Ref 3)

Refs: 1) Beil, not found 2) M.B. Frankel & K. Klager, JACS **79**, 2955(1957) & CA **51**, 14716(1957) 3) Ibid, USP 2978507(1961) & CA **55**, 15934(1961)

Pentanitrodipropylamine, $C_6H_{10}N_6O_{10}$; mw 326.19, N 25.77%. Two isomers are known:

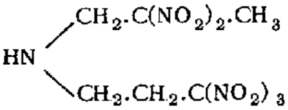
N,2,2,2',2'-Pentanitrodiphenylamine or **Bis(2,2-dinitropropyl)-nitramine,**



$$O_2N.N\begin{cases}CH_2.C(NO_2)_2.CH_3\\CH_2.C(NO_2)_2.CH_3\end{cases}$$

crysts (from MeOH), mp 187–89°. It was prepd by dropwise addn of 100% nitric acid to a soln of the above tetranitro deriv in ethyl acetate (Refs 3 & 6). The IR absorption bands were detd (Ref 6)

2',2',3,3,3-Pentanitrodipropylamine,



$$HN\begin{cases}CH_2.C(NO_2)_2.CH_3\\CH_2.CH_2.C(NO_2)_3\end{cases}$$

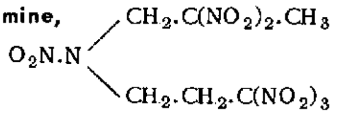
oil, fr p — not reported. It was obtd in quantitative yield by addg dropwise 1.136 N

NaOH soln at RT to a stirred soln of 3,3,3-trinitropropyl amine, $(O_2N)_3C.CH_2.CH_2.NH_2$, and 2,2-dinitropropanol in water, and cooling the mixt in an ice bath (Refs 2, 4, 5 & 6)

These compds are used as expl ingredients, or as O donors in propellant fuels (See Refs)

Refs: 1) Beil, not found 2) M.B. Frankel & K. Klager, USP 2978057(1961) & CA **55**, 15934(1961) 3) Ibid, USP 3000949(1962) & CA **56**, 8562(1962) 4) Ibid, JChemEngData **7**, 412(1962) & CA **57**, 10933(1962) 5) M.B. Frankel, Tetrahedron **19**, Suppl 1, 216(1963) & CA **59**, 11236(1963) 6) H.E. Ungnade & L.W. Kissinger, JOrgChem **30**(2), 359(1965) & CA **62**, 9043(1965)

N,2′,2′,3,3,3-Hexanitrodipropylamine or **N-(3,3,3-Trinitropropyl)-N-(2,2-dinitropropy)-nitramine,**



$$O_2N.N\begin{cases}CH_2.C(NO_2)_2.CH_3\\CH_2.CH_2.C(NO_2)_3\end{cases}$$

mw 371.19, N 26.42%; crysts (from CCl_4), mp 109–109.5°(dec). It was prepd by nitrating the Pentanitro deriv by using abs nitric acid & acetic anhydride at 5–10°, or by 100% nitric acid & concd sulfuric acid and a soln of 3,3,3-trinitropropyl amine dihydrochloride & 2,2-dinitropropanol in water with 4.877N NaOH added dropwise (Refs). This compd is sensitive to impact & useful as an expl additive

Refs: 1) Beil, not found 2) M.B. Frankel & K. Klager, USP 3000949(1962) & CA **56**, 8562 (1962) 3) Ibid, JChemEngData **7**, 412(1962) & CA **57**, 10933(1962) 4) M.B. Frankel, Tetrahedron **19**, Suppl 1, 216(1963) & CA **59**, 11236(1963)

Dipropylaniline and Derivatives

N,N-Dipropylaniline, $C_6H_5.N(C_3H_7)_2$, mw 177.28, N 7.90%; yel oil; sp gr 0.9104 at 20.4°, fr p (?), bp 245.4°; insol in w; sol in alc & eth. It can be prepd by reacting aniline with propyl iodide, treating the product with KOH soln and continued treating the separated base with propyl iodide

Ref: Beil. **12**, 167, (159) & [95]

Azido, $C_{12}H_{18}N_4$, and *Diazido,* $C_{12}H_{17}N_7$, derivs were not found in Beil

4-(or p)-Nitroso-N,N-dipropylaniline, $ON.C_6H_4.N(C_3H_7)_2$; mw 206.28; N 13.58%; emerald-grn triclinic crysts (from petr eth); mp 42°; sol in alc, eth, chlf & benz; decomp by heating in KOH soln to p-nitrosophenol & dipropylamine. It can be prepd by treating an ice-cold HCl soln of dipropylaniline with Na nitrite. It forms a *Hydrochloride salt,* $C_{12}H_{18}N_2O+HCl$, greenish microcryst powd, dec on heating rapidly at 160–65°

Ref: Beil **12**, 685, (338) & [366]

Mononitro-N,N-dipropylaniline, $O_2N.C_6H_4.N(C_3H_7)_2$; mw 222.28, N 12.60%:

2-Nitro-N,N-dipropylaniline, orn-yel, unpleasant acrid smelling oil, fr p – below solid CO_2 temp, bp – dec on heating; sl sol in w; sol in common org solvs. It was prepd by reacting 2-chloro-1-nitrobenzene & dipropylamine in the presence of Cu powd in alc at 100°(Refs 1 & 4). It forms cryst addn salts, some of which are unstable:

Chloride, $C_{12}H_{18}N_2O_2+HCl+AuCl_3$, yel prisms (from alc), dec on heating in alc

Cyanate, $2C_{12}H_{18}N_2O_2+H_4Fe(CN)_6+2H_2O$, brn crysts (from alc), dec on exposure to light

Cyanate, $2C_{12}H_{18}N_2O_2+H_3Fe(CN)$ $+2H_2O$, yel crysts, unstable in light

Iodide, $C_{12}H_{18}N_2O_2+HI$, ndls, decomp in light

Picrate, $C_{12}H_{18}N_2O_2+C_6H_3N_3O_7$, golden shiny pltlts (from abs alc), mp 93–94°, deflgr on heating rapidly (Ref 1)

3-Nitro-N,N-dipropylamine, not found in the literature thru 1966

4-Nitro-N,N-dipropylaniline, lt-green crysts, mp 59°. It was prepd by heating 4-bromo-1-nitrobenzene with dipropylamine in a tube at 183°(Refs 2 & 3)

Refs: 1) Beil **12**, (341) 2) Beil **12**, 715 3) Nogornow, JRussPhysChemGesellschaft **29**, 699(1898) & ChemZtr **1898** I, 886 4) M. Le Guyader & D. Peltier, BullFr **1966** (8), 2695(Fr) & CA **65**, 19686(1966)

Dinitro-N,N-dipropylaniline, $(O_2N)_2C_6H_3.N(C_3H_7)_2$ mw 267.28; N 15.72%. Only two isomers have been prepd:

2,4-Dinitro-N,N-dipropylaniline, brn-yel rhomb bipyramides (from acet), mp 40° or yel crysts, mp 41°; d 1.231 at 20°. It was prepd by re-

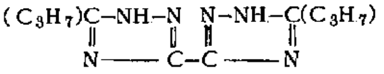
acting 4-bromo- or 4-chloro-1,3-dinitrobenzene with dipropylamine (Refs 1, 2, 4 & 6)
2,6-Dinitro-N,N-dipropylaniline, crysts, mp 50–52°; was prepd by reaction of 2,6-dinitrochlorobenzene & dipropylamine w or w/o a solv. This compd was effective against germinating weed grasses (Refs 5 & 7)
3,5-Dinitro-N,N-dipropylaniline, attempted prepn by heating 3,5-dinitro-n-propylaniline & n-propyl iodide for 100 hrs in a sealed tube at 100° yielded an insufficient quantity of product for characterization (Ref 3)
Refs: 1) Beil **12**, 750 & (362) 2) A. Hantzsch, Ber **43**, 1675(1928) 3) J.C. Roberts & K. Selby, JChemSoc **1949**, 2788 & CA **44**, 3922 (1950) 4) H. Bräuniger & K. Spangenberg, Pharmazie **12**, 335(1957) & CA **54**, 22668(1960) (Report mp of 95°) 5) Q.F. Soper et al, ProcSouthernWeedConf, 14th **1961**, 86 & CA **55**, 25137(1961) 6) A.T. Bottini & R.E. Olsen, JOrgChem **27**, 452(1962) & CA **57**, 708(1962) 7) Q.F. Soper, BritP 917253 (1963) & CA **59**, 9889(1963); USP 3257190 (1966) & CA **65**, 13606(1966)

2,4,6-Trinitro-N,N-dipropylaniline, $(O_2N)_3C_6H_2.N(C_3H_7)_2$; mw 312.28, N 17.94%; orn-red triclinic crysts (from acet + chlf), mp 38°, sp gr 1.373 at 10°. No method of prepn is given (Refs 1 & 2). It can probably be prepd from picryl chloride & dipropylamine in alc
Refs: 1) Beil **12**, 764 2) F.M. Jaeger, ZKryst u Mineral **42**, 361(1907) 3) Not found in later refs thru 1966

NOTE: No higher nitrated derivs of N,N-Dipropylaniline were found in the literature thru 1966

3,3′-Dipropyl-5,5′-azo-1,2,4-triazole. See 5,5′-Azobis(3-propyl-sym-triazole) in Vol 1, p A654-R

5,5′-Dipropyl-3,3′-bi-s-triazole,



$$\begin{array}{c}(C_3H_7)C-NH-N\quad N-NH-C(C_3H_7)\\ \| \qquad\qquad \| \quad \| \qquad\qquad \| \\ N ——— C-C ——— N\end{array}$$

mw 220.28; N 38.16%; wh crysts, mp – sublimes; sol in acet & chlf; sol in acids; sl sol in hot w & ammonia. It was prepd by sustained heating of cyanophenylhydrazine, $[C(:N.NH.C_6H_5)NH_2]_2$, & propionic anhydride, $(C_3H_7CO)_2O$

No nitrated derivs are reported in the literature thru 1966
Refs: 1) Beil, not found 2) G. Dedichen, AvhandlNorskeVidenskaps-Akad, Oslo, I, Mat-NaturvKlasse **1936**, No 5, 42pp & CA **31**, 4987(1937) 3) Not found in later refs thru 1966

Dipropylbenzidine and Derivatives

N,N′-Dipropylbenzidine; N,N′-Dipropyldiphenylene or *Dipropyldiaminobiphenyl*, $H_7C_3.HN.H_4C_6-C_6H_4.NH.C_3H_7$; mw 268.39, N 10.44%. It may be considered as the parent compd (Ref 1) of its nitrated derivs, although not used to prep them:
Dinitrodipropylbenzidine, $C_{18}H_{22}N_4O_4$, not found in the literature thru 1966
3,5,3′,5′-Tetranitro-N,N′-dipropylbenzidine, $H_7C_3.HN.C_6H_2(NO_2)_2-C_6H_2(NO_2)_2-NH.C_3H_7$; mw 448.39, N 18.74%; dk-red ndls (from methyl salicylate), mp 200°. It was prepd by heating propylamine with tetranitrobiphenol in alc in a sealed tube at 100° (Refs 2 & 4). It can be nitrated to a hexanitro deriv (qv)
N,N′-3,5,3′,5′-Hexanitro-N,N-dipropylbenzidine, $H_7C_3.N(NO_2).C_6H_2(NO_2)_2-C_6H_2(NO_2)_2.N(NO_2).$-$C_3H_7$; mw 538.39, N 20.82%; lt-yel pltlts (from concd nitric acid), mp – explodes at 213°. It was prepd by nitrating the above tetranitro deriv with nitric acid (d 1.49) (Refs 3 & 4)
Refs: 1) Beil, not found (Parent compd) 2) Beil **13**, [109] 3) Beil **13**, [110] 4) G. van Romburgh, Rec **41**, 42(1922) & CA **16**, 1238(1922) 5) Neither the parent compd nor its nitrated derivs were found in later refs thru 1966

Diisopropylbenzidine and Derivatives

N,N′-Diisopropylbenzidine, N,N′-Diisopropyldiphenylene or *Diisopropyldiaminobiphenyl*, $(H_3C)_2.HC.HN.C_6H_4.C_6H_4.NH.CH(CH_3)_2$; mw 268.39, N 10.44%. It may be considered as the parent compd (Ref 1) of its nitrated derivs, although not used to prep them: *Dinitrodiisopropylbenzidine*, $C_{18}H_{22}N_4O_4$, not found in the literature thru 1966

3,5,3′,5′-Tetranitro-N,N-diisopropylbenzidine, $(H_3C)_2HC.HN.C_6H_2(NO_2)_2-C_6H_2(NO_2)_2.NH.-CH(CH_3)_2$; mw 448.39, N 18.74%, dk-red ndls (from methyl salicylate), mp – dec ca 250°. It was prepd by heating isopropylamine with tetranitrobiphenol in alc in a sealed tube at 100° (Refs 2 & 4). It can be nitrated to a hexanitro deriv (qv)

N,N′-3,5,3′,5′-Hexanitro-N,N′-diisopropylbenzidine, $(H_3C)_2HC.N(NO_2).C_6H_2(NO_2)_2-C_6H_2(NO_2)_2.N(NO_2).CH(CH_3)_2$, mw 538.39, N 20.82%, nearly colorless crysts, mp – dec ca 209°. It was prepd by nitrating the tetranitro deriv with abs nitric acid (Refs 3 & 4)

Refs: 1) Beil, not found 2) Beil **13**, [109] 3) Beil **13**, [110] 4) G. van Romburgh, Rec **41**, 42(1922) & CA **16**, 1238(1922) 5) Neither the parent compd nor its nitrated derivs were found in the literature thru 1966

Dipropylbutyrate and Derivatives

Dipropylbutyrate or *Propylbutyrate*, $CH_3.CH_2.CH_2.O.CO.CH_2.CH_3.CH_3$, mw 130.18, liq, fr p – minus 95.2°, bp 142.7° at 764mm press; sp gr 0.8730 at 20°; n_D 1.4001 at 20°. It was prepd by reacting butyric acid with propyl alcohol in the presence of p-toluenesulfonic acid in hot benz

Refs: 1) Beil **2**, 271, (120), [246] & {596}

Azido, $C_7H_{13}N_3O_2$, and *Diazido*, $C_7H_{12}N_6O_2$, derivs, not found in the literature thru 1966

2-Nitropropyl-butyrate, $C_3H_7.COO.CH_2.CH(NO_2).CH_3$; mw 175.18, N 8.00%, liq, bp 226–28° (some decompn), sp gr 1.0860 at 20°, n_D 1.4321 at 20°; was prepd by heating 2-nitropropyl alc with butyric acid & concd sulfuric acid in benz (Refs 1 & 2)

Refs: 1) Beil **2**, {597} 2) J.B. Tindall, IEC **33**, 65(1941) & CA **35**, 2113(1941)

Dinitro, $C_7H_{12}N_2O_6$, *Trinitro*, $C_7H_{11}N_3O_8$, and *Tetranitro*, $C_7H_{10}N_4O_{10}$, derivs not found in the literature thru 1966

2,2-Dinitropropyl-4,4,4-trinitro-butyrate, (DNPTNBu)

$CH_3.C(NO_2)_2.CH_2.O.CO.CH_2.CH_2.C(NO_2)_3$; mw 355.12, N 19.72%, OB to CO_2 minus 29%; wh crysts, sp gr 1.68, mp 95–96°; exists in three crystalline modifications, of which Forms I & III are unstable, while Form II is stable and its mp is 95°. It was prepd by slowly mixing dinitropropanol, trinitrobutyryl chloride and Al chloride in carbon tetrachloride at 60°, followed by refluxing the mixture for 2 hrs. After the reaction:

$$CH_3.C(NO_2)_2.CH_2.\boxed{OH+Cl}OC.CH_2.CH_2.C(NO_2)_3 \rightarrow \text{DNPTNBu}$$

was completed, the mixture was cooled and the crystalline product separated and purified. Its mp was 95°

Note: Water in dinitropropanol was removed by azeotropic distillation before the acid chloride was added

Following are some properties of DNPTNBu:

Explosion Temperature – 300° in 5 secs

Rate of Detonation – 7600m/sec at sp gr 1.67

Vacuum Stability Test at 100° – 0.5cc/40 hrs

It can be stored dry and cast loaded. Upon solidification of molten material, Form II is always observed

Refs: 1) Beil, not found (DNPTNBu) 2) M.E. Hill, "Preparation and Properties of 2,2-Dinitropropanol Esters", NAVORD Rept **2497** (1952) 3) W.B. Hewson, "Hercules Report on High Explosives," Navy Contract, NOrd-11280, Task A (October 1954) 4) J.R. Holden & J. Wenograd, "Physical Properties of Experimental Castable Explosive 2,2-Dinitropropyl-4,4,4-trinitrobutyrate", NAVORD Rept **4427** (1956) 5) D.J. Glover, Tetrahedron **19**, Suppl 1, 219(1963) & CA **59**, 13358(1963) 6) A. Solomonovici & S. Blumberg, IsraelJChem **3**(2) 63(1965) (Engl) & CA **63**, 6851(1965) 7) M.E. Hill, USP 3223725(1965) & CA **64**, 6570(1966); USP 3230247(1966) & CA **64**, 9598(1966) 8) US Army Materiel Command Pamphlet, AMCP **706–177**, Washington, DC, 20315(1971), pp 113–15

Hexanitropropylbutyrate, $C_7H_8N_6O_{14}$, not found in the literature thru 1966

Di(propylenediamino)-copper (II) Diazide, $[Cu(CH_3CH.NH_2.CH_2.NH_2)_2](N_3)_2$; mw 295.84, N 47.35%; dk violet ndls; two modifications; mp 165–66°; bp – burns in flame with crepitations; dissolves in w imparting a violet coloration. It can be prepd from Cu azide & propylenediamine in alc soln (Ref 2)

Does not detonate by impact

Refs: 1) Beil, not found 2) M. Straumanis & A. Cirulis, ZAnorgChem **251**, 339–40(1943) & CA **37**, 6573(1943)

Dipropyleneglycol and Derivatives

Dipropyleneglycol or *1,1'-Oxydi-2-propanol,* $(CH_3.CHOH.CH_2)_2O$, mw 134.17, O 35.77%; colorless liq, sp gr 1.025 at 20/20°, fr p >–50°, bp 231.8°, fl p 280° F; viscosity 1.07 poise at 20°; sol in w & in toluene. It can be prepd by the condensation of propylene oxide & propylene glycol at 117–18°(Refs 1 & 2). It is twice as toxic as propylene glycol. Industrial prepn is described in Ref 3. It is a fairly good solvent for NC

Refs: 1) Beil. **1**, [537] & {2149} 2) P.A. Levine & A. Walti, JBiolChem **75**, 331(1927) 3) D.R. Keck, Petroleum Engr **24**, No 9, C39 (1952) & CA **46**, 11672(1952) 4) CondChemDict (1961), 409-R

Dipropyleneglycol Dinitrate (DPGDN), $CH_3.CH(ONO_2).CH_2.O.CH_2.CH(ONO_2).CH_3$, mw 224.17, N 12.50%; colorless liq, fr p not reported, bp 100° at 0.1mm press; sp gr 1.2310 at 21°, n_D 1.4460 at 15°(Ref 3). It can be prepd by nitration of dipropyleneglycol. Macy & Saffitz (Ref 2) prepd it in PicArsn Chem Lab by nitration of product supplied by Carbide & Carbon Corp using a nitrating mixt composed of acetic anhydride, acetic acid, and 93% nitric acid at 3–5°. DPGDN is volatile to the extent of 0.35mg/sq cm/hr at 60°C (Ref 2)

It is an expl with impact sensitivity of 9 inch drop of 2kg weight (vs 14 inches for TNT), using PicArsn Apparatus. In the 100°C Heat Test, it lost 5.3% the first 48 hrs & 6.7% in the second 48 hrs. A 1 gram sample gave 1.08cc gas in 40 hrs at 100°. Its % acidity was 0.123 in 5 days at 60° in Hydrolysis Test (Ref 2)

It is suitable for use as an ingredient of double-base proplnts.

Refs: 1) Beil, not found 2) P.F. Macy & A.A. Saffitz, PATR **1638**(1947), p6 & Tables 3) Anne-Marie Pujo & J. Boileau, CR **237**, 1422(1953) & CA **49**, 853(1955); MP **37**, 44 (1955) & CA **51**, 11244(1957) 4) Not found in later refs thru 1966

Dipropyl Ethers, $C_6H_{14}O$, mw 102.17, O 15.66%. The following isomers are known:

n-Dipropyl Ether, $(C_2H_5.CH_2)_2O$, colorless liq, fr p minus 122°, bp 90.1°; sl sol in w; sol in alc & ether (Ref 1)

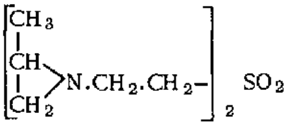
Di-(iso-Propyl) Ether, $[(CH_3)_2CH]_2O$, colorless liq, fr p minus 60°, bp 68.5–69°; sl sol in w; sol in alc & ether (Ref 2)

n-Propyl-iso-propyl Ether, $C_2H_5.CH_2.O.CH(CH_3)_2$, colorless liq, bp 82–83°; sl sol in w (Ref 3)

Morgan & Pickard (Ref 4) described two explns which took place in England during the distillation of ethers (mostly of Di-iso-propyl ether), which were stored for several years. These ethers gave tests for presence of peroxides. They may be removed either by treatment with Hg or K permanganate soln

Refs: 1) Beil **1**, 354, (178), [367] & {1415} 2) Beil **1**, 362, [381] & {1459} 3) Beil. **1**, 362, [381] & {1458} 4) G.T. Morgan & R.H. Pickard, Chem & Ind **55**, 421–22(1936) & CA **30**, 5416 (1936)

Di(or Bis)(2-propyleniminoethyl) sulfone, {called 1,1'-(Sulfonyldiethylen)bis[2-methylaziridine] in CA Formula Index **56–65**(1962–66), p115F},



$$\left[\begin{array}{l} CH_3 \\ | \\ CH \\ | \\ CH_2 \end{array} \!\!> N.CH_2.CH_2- \right]_2 SO_2$$

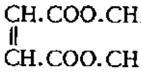
mw 237.35, N 11.81%; water-white to lt-amber liq, bp 145–48° at 0.8mm press, sp gr 1.0846 at 25°, n_D 1.4816 at 25° and viscosity of 70cp at 25°. It was produced by reaction of divinyl sulfone with propylenimine in a mole ratio of ~2.15 at 15–25°. The product is recovered by stripping off the excess imine under vacuum

It was proposed for use as a curing agent in COOH- contg polymers and used with such polymers in proplnts with Amm Perchlorate as the oxidizing agent

Refs: 1) Beil, not found 2) R.C. Christena & E. Broderick of ThiocolChemicalCorp, BritP 1003385(1965) & CA **63**, 12963(1965); USP 3235544(1966) & CA **64**, 14168(1966)

Dipropyl-fumarate and Derivatives

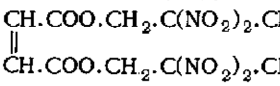
Di(or Bis)(propyl)-fumarate (called in CA *Dipropyl Ester of Fumaric Acid*),



$$\begin{array}{l} CH.COO.CH_2.CH_2.CH_3 \\ \| \\ CH.COO.CH_2.CH_2.CH_3 \end{array}$$

, mw 200.23, O 31.96%, liq, bp 110° at 5mm press, sp gr 1.0129 at 20°, n_D 1.440 at 20°. It was prepd by reacting Ag fumarate with propyl bromide in boiling benzene (Ref 1)

Bis(2,2-dinitropropyl)-fumarate,



$$\begin{array}{l} CH.COO.CH_2.C(NO_2)_2.CH_3 \\ \| \\ CH.COO.CH_2.C(NO_2)_2.CH_3 \end{array}$$

, mw 380.23, N 14.73%, transparent ndls (from chlf & hexane) mp 85.6–86.2°; sp gr 1.60, sol in CCl_4, CS_2, dichloro ethylene & nitrobenz. It was prepd in 80% yield by heating at 45° dinitropropyl alcohol with fumaryl chloride in chlf and $AlCl_3$ as a catalyst

Its impact sensitivity shows a 50% expln point above 320cm, thermal stability 0.59 cc/g/100°/48 hrs; and ignition temp at 50% point is 369°

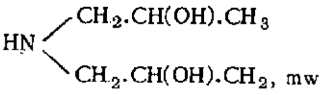
Because of its stability and waxy-like characteristics, this compd may be useful as a desensitizing coating for expls, or as an ingredient in expl compns (Refs 3 & 4)

Refs: 1) Beil **2**, 742, [639] & {1904}
2) Beil, not found (Dinitro) 3) M.E. Hill, "Preparation and Properties of 2,2-Dinitropropanol Esters", NAVORD **2497**(1952)
4) M.E. Hill, USP 3223725(1965) & CA **64**, 6570(1966)

Dipropylketone or **4-Heptanone**. See Butyrone in Vol 2, p B393-L

Di(propyl)-amine and Derivatives

Di(iso-propylol)-amine; N,N-Di(2-propylol)-amine or *Bis(β-hydroxypropyl)-amine,*

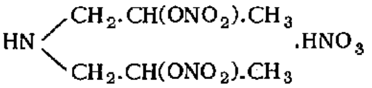


$$HN\begin{cases} CH_2.CH(OH).CH_3 \\ CH_2.CH(OH).CH_2 \end{cases}$$

, mw 133.19, N 10.52%, crysts, mp 44.5–45.5°, bp 249–50° at 745mm press, sp gr 1.003 at 25°; sol in w & alc; sl sol in eth. It can be prepd by reaction of propylene oxide & ammonia in aq soln. It forms cryst salts of Hydrochloride, Hexachloroplatinate & Picrate (Ref 1). It gives on nitration the compd listed below

Refs: 1) Beil **4**, [737] & {761} 2) Carbide & Carbon Chem Corp, USP 1988225(1932)
3) J.H. Jones, JAssocOfficialAgrChem **27**, 462–67(1944) & CA **38**, 6276(1944)

Di(2-nitroxy-iso-propyl)-amine Nitrate; N,N-Di(β-nitroxy-iso-propyl)-amine Nitrate or **N,N-Bis(β-nitroxy-iso-propyl)-amine Nitrate,**

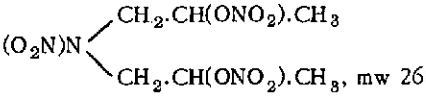


$$HN\begin{cases} CH_2.CH(ONO_2).CH_3 \\ CH_2.CH(ONO_2).CH_3 \end{cases}.HNO_3$$

mw 286.20, N 19.58%, OB to CO_2 minus 55.9%; crysts (from w), mp 119.6°(Ref 1) & 144° (Ref 2). It can be prepd by treating N,N-di-(β-propylol)-amine with 98% nitric acid at below 15°(Ref 1). It was used by Blomquist & Fiedorek for prepn of the compd listed below

Refs: Same as under next compd

Di(2-nitroxy-iso-propyl)-nitramine; N,N-Di-(β-nitroxy-iso-propyl)-nitramine or **N,N-Bis-(β-nitroxy-iso-propyl)-nitramine,**



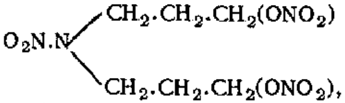
$$(O_2N)N\begin{cases} CH_2.CH(ONO_2).CH_3 \\ CH_2.CH(ONO_2).CH_3 \end{cases}$$

, mw 268.19, N 20.89%, OB to CO_2 minus 59.7%; crysts (from ether), mp 103–104°(Ref 2). It was prepd by Blomquist & Fiedorek by dehydration of N,N-di(β-nitroxypropyl)-amine nitrate on heating at 40–45° with acetic anhydride with small amt of HCl, followed by addition of chloride catalyst such as Zn chloride (Refs 2 & 3)

The **Nitrate** & **Nitramine** compds are expl. The Trinitrate of Brandner (Ref 3) was highly resistant to shock & friction, but was easily detonated by primary expls. It was recommended as a base chge in compd detonators

Refs: 1) Beil. **4**, {762} 2) A.T. Blomquist & F.T. Fiedorek, OSRD **4134** (PB **18867**) (1944), pp 48 & 127–28 3) J.D. Brandner, USP 2415001 (1944) & CA **41**, 2578 (1947)

N,N-Di(3-nitroxypropyl)-nitramine, called by Fishbein *Dihydroxypropyl Trinitrate*,



$$O_2N.N\begin{cases}CH_2.CH_2.CH_2(ONO_2)\\CH_2.CH_2.CH_2(ONO_2)\end{cases}$$

, mw 268.19, N 20.89%, OB to CO_2 minus 55.9%; crysts (from water), mp 143° exploding on heating at higher temp. It can be prepd by nitration of di(propylol)-amine with mixed nitric-sulfuric acid at a temp between 0° & 10°

It was patented in Europe for use as an explosive alone or in mixtures

Refs: 1) Beil. **4**, {750} 2) Dynamit AG, BritP 358157 (1930) & CA **26**, 6141 (1932) 3) M.S. Fishbein, Voyennaya Khimiya (Military Chemistry) (Russia) **1933**, No 6, pp 3–8; CA **29**, 7077 (1935) 4) Clift & Fedoroff **2** (1943), p D7 & **4** (1946), p26

Di(propylol)-ethylenediamine and Derivatives

N,N'-Di(propylol)-ethylenediamine, N,N'-Di(propanol)-ethylenediamine or *N,N'-Bis(β-hydroxypropyl)-ethylenediamine*, $HO.H_7C_3.HN.CH_2.CH_2.NH.C_3H_7.OH$, mw 178.27, N 15.71%. This compd may be considered as the parent compd of the nitrated derivatives listed below, although they were not prepd from it:

N,N'-Di(propylol)-ethylenedinitramine; N,N'-Di(propanol)-ethylenedinitramine or **N,N'-Bis(β-hydroxypropyl)-ethylenedinitramine,** $HO.H_7C_3.(O_2N)N.CH_2.CH_2.N(NO_2).C_3H_7\,OH$; mw 268.28, N 20.89%; sl colored glassy oil. It was obtd by stirring for several days a suspension of ethylenedinitramine and propyleneoxide in water contg some NaOH. The product did not crystallize

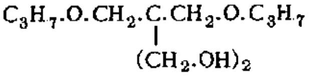
Nitration of this product with 98% nitric acid at 5–10° gave two crops of crysts. The first crop had, after recrystallization from ethanol, mp 94–6°, whereas the second crop had 78–81°. It was assumed that they are (probable) meso and racemic modifications of: **N,N'-Di(2-nitroxypropyl)-ethylenedinitramine,** $(O_2NO).H_7C_3.(O_2N)N.CH_2.CH_2.N(NO_2).C_3H_7(ONO_2)$, mw 358.28, N 23.46%; obtd in two crops: 1st crop (alc), mp 94–96°, 2nd crop (alc), mp 78–81°. The above oil was added, while stirring, to 98% nitric acid at 5–10° for 15 mins, and the reaction quenched in ice & water. The crude product was twice recrystallized.

No expl props of this compd are reported

Refs: 1) Beil, not found (Dipropylol-ethylenediamine) 2) Beil, not found (Nitrated derivs) 3) A.T. Blomquist, OSRD **5155** (1945), 175–76 4) Not found in later refs thru 1966

Dipropylpentaerythritol Ether and Derivatives

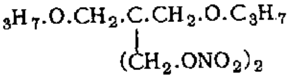
Dipropylpentaerythritol Ether,



$$C_3H_7.O.CH_2.\underset{\displaystyle (CH_2.OH)_2}{\underset{|}{C}}.CH_2.O.C_3H_7$$

mw 220.30, O 29.05%, liq, bp 115° at 1mm press, sp gr 0.993 at 20°, n_D 1.4461 at 20°. It was prepd by hydrogenating at RT & atmospheric press, using Pt oxide, a soln of diallyl pentaerythritol in 95% alc (Ref 3)

Dipropylpentaerythritol Dinitrate [called 2,2-Bis(propoxymethyl)-1,3-propanediol Dinitrate in CA],



$$C_3H_7.O.CH_2.\underset{\displaystyle (CH_2.ONO_2)_2}{\underset{|}{C}}.CH_2.O.C_3H_7$$

mw 310.30, N 9.03%, liq sp gr 1.144, n_D 1.4470 at 20°. It was prepd by nitrating the parent compd with nitric acid (Ref 3). It has possible use as a gelatinizer of NC

Refs: 1) Beil, not found (Parent) 2) Beil, not found (Dinitrate) 3) R. Evans & J.A. Gallaghan, JACS **75**, 1248–49 (1953) & CA **49**, 3811 (1955) 4) Not found in later refs thru 1966

NOTE: No higher nitrated derivs of Dipentaerythritol Ether were found in the literature thru 1966

Di-n-propyl-peroxide, $C_3H_7-OO-C_3H_7$, mw 118.17, O 27.08%; liquid, bp 51–53° at 8cm press. It can be prepd from a mixt of hydrogen peroxide (cooled to –20°), KOH in water & dipropyl sulfate. The non-aqueous upper layer was separated by distillation at reduced pressure (Ref 4). This peroxide exploded at sl lower pressures than Diethyl Peroxide (Refs 3, 4 & 5)

Refs: 1) Beil, not found 2) E.J. Harris, PrRoySoc **173A**, 126(1939) 3) R.H. Wiley, USP 2357298(1944) & CA **39**, 312(1945) 4) Tobolsky & Mesrobian (1954), 164 5) F. Welch et al, JACS **77**, 551(1955) & CA **50**, 780(1956) (IR absorption spectrum tabulated) 6) S.W. Benson, JChemPhys **40**(4), 1007(1964) & CA **60**, 6255(1964) (Kinetics of pyrolysis & bond dissocn energy)

Di(iso-propyl) -peroxide, $(CH_3)_2.CH-OO-CH(CH_3)_2$ mw 118.17, O 27.08%; liquid, bp ~94° at 760mm & 55–58° at 233mm press w/o expln; not exploded by impact; undergoes photo-initiated, auto-inhibited chain decompn at 26°(Ref 6)

The only detailed method of prepn, by distilling a mixt of isopropyl methanesulfonate, KOH in water & hydrogen peroxide at 51°, is that of Pryor (Ref 10). Its elemental analysis is dangerous. Pryor (Ref 8) reported an accidental expl of Di(iso-propyl) -peroxide in the gas phase at ca 200° in an O stream

Refs: 1) Beil, not found 2) W. Rockemüller & L. Pfeuffer, Ann **537**, 178(1939) & CA not found 3) E.K. Maĭzus & N.M. Emanuel, DoklAkadNauk **83**, 711(1952) & CA **46**, 6915(1952) (Homogeneous catalysis of oxidation of propane) 4) Tobolsky & Mesrobian (1954), 164 5) H. Langhans, GerP 951626(1956) & CA **53**, 4216(1959) [Pb Di-(iso-propyl)-peroxide] 6) G.R. McMillan, JACS **83**, 3018–23(1961) & CA **56**, 3053(1962); JACS **84**, 2514(1962) & CA **57**, 7091(1962) 7) G.I. Likhtenshtein, ZhFizKhim **36**, 1503(1962) & CA **57**, 11927 (1962) (Calculation of heat of formation) 8) W.A. Pryor, Tetrahedron Letters **1963**(18), 1201 & CA **59**, 15148(1963) (Decompn) 9) S.W. Benson, JChemPhys **40**(4), 1007 (1964) & CA **60**, 6255(1964) (Kinetics of pyrolysis & bond dissocn energy) 10) W.A. Pryor et al, JACS **86**(20), 4237(1964) & CA **61**, 14495(1964)

Dipropyl Succinate and Derivatives

Di(or Bis)(propyl)-succinate (called *Dipropyl Ester of Succinic Acid* in CA) (Called Bernsteinsäure-dipropylester, in Ger)

$$\begin{array}{l}CH_2.CO_2.CH_2.CH_2.CH_3\\ |\\ CH_2.CO_2.CH_2.CH_2.CH_3\end{array}$$

mw 202.24, O 31.64%, liq, fr p minus 5.9°, bp 101.5° at 3mm press, sp gr 1.0020 at 20°, n_D 1.4250 at 20°. It can be prepd by azeotropic distillation of succinic acid, propyl alcohol & benz (Ref 1)

Dinitrodipropyl Succinate,

$$\begin{array}{l}CH_2.CO_2.CH_2.CH(NO_2).CH_3\\ |\\ CH_2.CO_2.CH_2.CH(NO_2).CH_3\end{array}$$

mw 382.24, N 9.59%

The *Bis(2-nitropropyl) Succinate* (Ref 4) and the *Bis(3-nitropropyl) Succinate* (Ref 5) are listed in the literature, but no description of the compds is found in CA

2,2,2′,2′-Tetranitrodipropyl Succinate,

$$\begin{array}{l}CH_2.CO_2.CH_2.C(NO_2)_2.CH_3\\ |\\ CH_2.CO_2.CH_2.C(NO_2)_2.CH_3\end{array}$$

mw 382.24, N 14.68%, crysts (from MeOH), mp 85–85.6°, sp gr 1.51. It was prepd by the Al chloride catalyzed esterification of 2,2-dinitropropanol with succinyl chloride in methylene chloride soln (Refs 3 & 6). It is an insensitive compd (50% Impact Sensitivity, 2.5kg wt >320cm), with excellent thermal stability (Ignition Temp >400° & 0.10cc gas/g/48hrs at 100°) This compd is a castable expl

Refs: 1) Beil **2**, 611, (264), [551] & {1664} 2) Beil, not found (Nitro compds) 3) M.E. Hill, NAVORD Rept **2965**(1953), p4 4) E.B. Hodge & G.J. Lafferty, FrP 1364172(1964) & CA **62**, 7988(1965) 5) D.J. Beaver & P.J. Stoffel, BritP 1003467(1965) & CA **63**, 14711(1965) 6) M.E. Hill, USP 3223725 (1965) & CA **64**, 6570(1966)

Dipropyl-sulfone and Derivatives

Dipropyl-sulfone (Called Propylsulfone in CA),

$H_3C.CH_2.CH_2.SO_2.CH_2.CH_2.CH_3$, mw 150.24, O 21.30%; flakes, mp 29.5–30.5°, sp gr 1.0278 at 50°, n_D 1.4456 at 30°. It can be prepd by oxidation of dipropyl sulfide with Na hypochlorite soln or with a 3% aq permanganate soln (Ref 1)

Dinitro, $C_6H_{12}N_2O_6S$, and *Tetranitro*, $C_6H_{10}N_4O_{10}S$, derivs were not found in the literature thru 1966

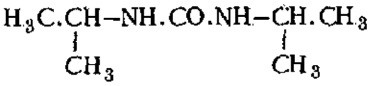
3,3,3,3′,3′,3′-Hexanitropropyl-sulfone or **Di(3,3,3-trinitropropyl)-sulfone,** $(O_2N)_3.C.CH_2.CH_2{-}SO_2{-}CH_2.CH_2.C(NO_2)_3$, mw 420.24, N 20.00%; wh crysts, mp 152–53°; burned with a flash in a flame. It was prepd by Tawney of US Rubber Co from divinyl sulfone & trinitromethane in MeOH for ½ hr (Refs 3 & 4). This compd is an expl and is detonated by a heavy hammer blow
Refs: 1) Beil **1**, 359, [373] & {1435} (Parent) 2) Beil **1**, not found (Dinitro-, Tetranitro- & Hexanitro- derivs) 3) US Rubber Co, Quart Progress Rept **No 4**, July 31, 1948 to Sept 30, 1948, NOrd 10129, p9 4) P.O. Tawney, USP 3146270(1964) & CA **61**, 13195(1964)

Dipropylureas and Derivatives

N,N′(or sym)-n-Dipropylurea, $H_5C_2.CH_2{-}NH.CO.NH{-}CH_2.C_2H_5$, mw 144.21, N 19.43%, ndls, mp 105°, bp 255°; sl sol in cold w; v sol in alc & eth. It can be prepd from N,N -n-dipropylthiourea & moist mercuric oxide, or by heating propylisocyanate with water at 100° (Ref 1)

N,N(or unsym)-n-Dipropylurea, $(H_5C_2.CH_2)_2N.CO.NH_2$, mw 144.21, N 19.43%, ndls (from petr eth), mp 76°; v sol in w. It can be prepd from reaction of dipropylamine & nitrourea in water. It forms cryst salts, such as Nitrate, Oxalate & Picrate (Ref 2)

N,N′-Di-iso-propylurea,



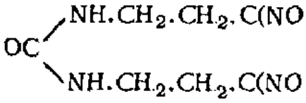
$$H_3C.\underset{\underset{CH_3}{|}}{C}H{-}NH.CO.NH{-}\underset{\underset{CH_3}{|}}{C}H.CH_3$$

mw 144.21, N 19.43%, crysts (from aq alc), mp 192°. It can be prepd by heating isopropylisocyanate with water, or by heating an aq soln of the K or Na salt of isobutyric hydroxamylbenzoate (Ref 3)

N,N(or unsym)-Di-iso-propylurea, $[(H_3C)_2CH]_2N.CO.NH_2$; mw 144.21, N 19.43%; crysts (from eth), mp 103°. It can be prepd by treating diisopropylamine sulfate with KCNO. It forms salts of Nitrate, Picrate & Oxalate, $2C_7H_{16}N_2O + C_2H_2O_4$, ndls (from alc), dec at 111° (Ref 4)
Refs: 1) Beil **4**, 142, [626] & {261} 2) Beil **4**, 143, (366), [626] & {262} 3) Beil **4**, 155 & [631] & {277} 4) Beil **4**, 155

Dinitro, $C_7H_{14}N_4O_5$, and *Tetranitro*, $C_7H_{12}N_6O_9$ derivs of Dipropylurea were not found in the literature thru 1966

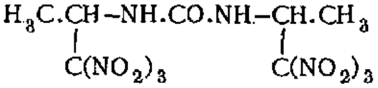
3,3,3,3′,3′,3′-Hexanitro-N,N′-n-dipropylurea or **1,3-Bis(3,3,3-trinitropropyl)-urea,**



$$OC\begin{matrix}\diagup NH.CH_2.CH_2.C(NO_2)_3 \\ \diagdown NH.CH_2.CH_2.C(NO_2)_3\end{matrix}$$

; mw 414.21, N 27.05%, wh crysts (from chlf), mp 170–71°. It was prepd by refluxing the hexanitrodipropylisocyanate with a soln of acetone & water until crysts pptd. Prepn of starting material is described (Ref 3). This compd is expl

1,1,1,1′,1′,1′-Hexanitro-di-iso-propyl-urea or **Bis (1,1,1-trinitro-iso-propyl) -urea,**

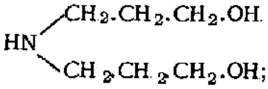


$$H_3C.\underset{\underset{C(NO_2)_3}{|}}{C}H{-}NH.CO.NH{-}\underset{\underset{C(NO_2)_3}{|}}{C}H.CH_3$$

mw 414.21, N 27.05%; wh crysts mp 145° (decomp explosively). It was prepd by reaction of nitrofom (0.03 mole), acetaldehyde (0.03 mole) & urea (0.015 mole) in 25ml acetic acid heated at 90–95° for one hr. It was cooled, and the ppt crystallized from chlf & Skellysolve B. The product flashes on a hot plate & detonates with a hammer blow (Ref 2)
Refs: 1) Beil, not found 2) USRubberCo, QuartProgressRept **No 14**, NOrd 10129, Feb 1 to May 1, 1951, p5 3) M.H. Gold et al, JOrgChem **27**, 334(1962) & CA **56**, 15341(1962)

Di(propylol)-amine and Derivatives

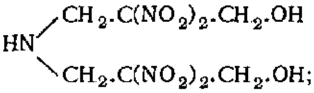
N,N-Di(3-propylol)-amine; N,N-Bis(propanol)-amine; N,N-Di(3-hydroxypropyl)-amine or *Amino-dipropylol,*



$HN\begin{matrix}\diagup CH_2.CH_2.CH_2.OH\\ \diagdown CH_2.CH_2.CH_2.OH\end{matrix}$; mw 133.19, N 10.52%, no props given. It was obtd in a mixt of mono-, di- & tris(hydroxypropyl)amine when 3-hydroxyproprionitrile was treated with H in an autoclave in the presence of Ni-kieselguhr catalyst. The mixt can be separated by distillation (Refs 1 & 2). It forms a *Hexachloroplatinate*, $2C_6H_{15}NO_2.H_2PtCl_6$, dec 211–12°

Refs: 1) Beil 4, {746} 2) O. Schlichting, GerP 573983(1933) & CA **27**, 4241(1933)

N,N-Di(2,2-dinitro-3-propylol)-amine; N,N-Di(3-hydroxy-2,2-dinitropropyl) -amine or N,N-Bis(2,2-dinitro-3-propanol)-amine,



$HN\begin{matrix}\diagup CH_2.C(NO_2)_2.CH_2.OH\\ \diagdown CH_2.C(NO_2)_2.CH_2.OH\end{matrix}$; mw 313.55, N 22.45%. No description of compd is found in the abstracts of patents (Ref 2). It can be prepd by treating 2 mols of 2,2-dinitropropanediol with 1 mol of ammonia:

$$2HO.CH_2.C(NO_2)_2.CH_2.OH + NH_3 \rightarrow$$
$$2H_2O + HN\,[CH_2.C(NO_2)_2CH_2OH]_2$$

It was patented in Sweden and other countries for use as an ingredient of some expl and proplnt compns

Refs: 1) Beil, not found 2) F.R. Schenk & G.A. Wetterholm, SwedP 148217(1954); BritP 729469(1955); USP 2731460(1956) and CA **50**, 1893 & 7125(1956)

Dipyridine Diazido-Metal Complexes

Dipyridinecadmium (II) Diazide, $[Cd(C_5H_5N)_2](N_3)_2$; mw 354.65, N 31.59%; solid; mp-on slow heating melts, evolving pyridine; then it assumes yellow color and detonates with a loud report; sol in hot water. It can be prepd by reaction of pyridine Cd chloride in w with a soln of NaN_3 in pyridine (Ref 2, p213)

Dipyridinecopper (II) Diazide or Dipyridinodiazidocopper (II), $[Cu(C_5H_5N)_2](N_3)_2$; mw 305.78, N 36.62%; exists in two modifications: I - dark green ndls melting at 138°, but starting to decomp above 100° loosing pyridine to give $Cu(N_3)_2$; explodes somewhat violently at 205° (on preheated metallic block); very difficultly sol in cold w; decompd by hot w; expl by a sharp blow of a hammer; II - brown crysts, mp 138°, bp – expl at 203–205°; very diffc sol in w; impact sensitivity 20cm with 1 kg weight.. Both modifications give the same X-ray diagrams (Ref 2, pp210–11 & Ref 3)

Dipyridinenickel (II) Diazide, $[Ni(C_5H_5N)_2](N_3)_2$; mw 300.93, N 37.23%; lt green crysts; mp – not reported. It can be prepd from dipyridine Ni chloride & a NaN_3 soln (Ref 2, p217–18)

Dipyridinezinc (II) Diazide, $Zn(C_5H_5N)_2](N_3)_2$; mw 307.62, N 36.43%; wh crysts, mp – not reported. It can be prepd from Zn dipyridine chloride, pyridine & an aq NaN_3 soln (Ref 2, p212)

Refs: 1) Beil, not found 2) W. Strecker & E. Schwinn, JPraktChem **152**, 210–13 & 217–18(1939) & CA **33**, 5314(1939) 3) M. Straumanis & A. Cirulis, ZAnorgChem **251**, 349–50(1943) & CA **37**, 6574(1943)

Dipyridylamine and Derivatives

Dipyridylamine, $(NC_5H_4)_2NH$; mw 171.20, N 24.55%. It exists as Di-α-(or Di-2-pyridylamine, crysts (from w), mp 95° & ndls (from eth), mp 84°; and Di-γ-(or Di-4-pyridyl amine, lt-yel ndls (from benz), mp 138°. Both form cryst salts (Ref 1)

Dinitrodipyridylamine, $[NC_5H_3(NO_2)_2]_2NH$; mw 261.20, N 26.81%:

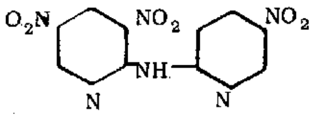
3,3'-Dinitro-(di-a-pyridylamine), yel pltlts (from 80% acet ac), mp 179–80°

5,5'-Dinitro-(di-a-pyridylamine), lt-yel ndls (from 80% acet ac), mp 219–20° or lt-brn ndls (from pyridine), mp 224°

X,X-Dinitro-(di-a-pyridylamine), yel-brn ndls (from toluene), mp 195–96°

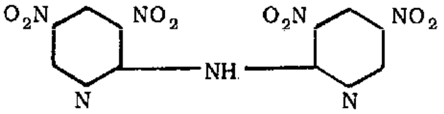
Other props & methods of prepn are found in the Refs (Refs 2 & 5)

3,5,5'-Trinitro-(di-α-pyridylamine),



mw 306.20, N 27.45%, yel crysts (from 80% acet ac), mp 197°; sol in mineral acids & in cold alkalies; sl sol in toluene, ethyl acet & chlf; insol in alc, eth & water. It was prepd by heating the 5-nitro or 5,5'-dinitro deriv with mixed nitric-sulfuric acids at 90° (Refs 3 & 5)

3,5,3',5'-Tetranitri(di-α-pyridylamine),



mw 347.19, N 28.24%, lt-yel ndls (from 3% acet ac), mp 140–41°; sol in mineral acids & cold alkalies; mod sol in hot eth acet & acet ac; sl sol in w, alc & chlf; insol in water. It was prepd by nitrating Di-α-pyridylamine or its nitro derivs with an excess of mixed nitric-sulfuric acids on a steam bath. The compd evolves ammonia on heating (Refs 4 & 5)

Refs: 1) Beil **22**, 630 & [331, 341] 2) Beil **22**, [336, 337, 339] 3) Beil **22**, [339] (Trinitro) 4) Beil **22**, [339] (Tetranitro) 5) A.E. Chichibabin & V.A. Preobrazhenskii, Ber **61**, 202–06(1928) & CA **22**, 1357(1928)

Di(*iso*-quinolino)-diazido Copper (II) Complex or Di(*iso*-quinolino)-copper(II) Diazide,

$[Cu(C_9H_6N)_2](N_3)_2$; mw 403.88, N 27.75%, yel-green crysts; mp – explodes at 197–200°; burns in flame; sol in ethylenediamine with violet color freeing some *iso*-quinoline; darkens in warm acid. It is insensitive to impact. It was prepd by dissolving Cu azide in an *iso*-quinoline amine soln in alc, by the addn of NaN_3 to an amine soln of the Cu^{++} salt in MeOH, or by rearrangement of the cuprate amine (Ref 2)

Refs: 1) Beil, not found 2) A. Cirulis & M. Straumanis, JPraktChem **162**, 313(1943) & CA **38**, 1969(1944)

Directed Explosives. One of the terms for Shaped Charges, described as *Munroe-Neumann Effect* in Vol 4, under DETONATION, p D442-R

Directed Missiles. Same as Guided Missiles

Direct Photography. Photography by reflected light to distinguish it from Shadow, Schlieren and other methods which are described in Vol 2, pp C13 to C19 under CAMERAS, HIGH-SPEED PHOTOGRAPHIC

Diresorcinol or Tetrahydroxybiphenyl. See Biresorcinol in Vol 2, p B126-R. Hexanitrobiresorcinol is described on p B127-L

Direzione Artiglieria. Ital for Ordnance Department

Diripsite. A chlorate expl of which two samples were examined in 1889 by the British authorities. It was rejected on account of its great sensitivity fo friction and insufficient chemical stability

Ref: Daniel (1902), 206

Disalicylic Acid and Derivatives

Disalicylic Acid; Displosal or Salysal, $HO.C_6H_4.CO_2.C_6H_4.CO_2H$; mw 258.22; colorless crysts, mp 148–49°, bp decomp; insol in w; sl sol in benz; sol in alc, eth, acet & CCl_4. It can be prepd by heating α-disalicylide,

, with glacial acet acid and by other methods (Ref 1)

Its nitration is discussed in Ref 3. Substitution takes place only in position 5 on the nucleus bearing the free OH. Thus, nitration gives:

5-Nitrosalicyl-salicylic Acid, $HO.C_6H_3(NO_2).CO_2.C_6H_4.COOH$; mw 303.22, N 4.62%, yel ndls (from chlf-abs alc), mp 196° (Ref 3). No other props given

NOTE: No *Azido,* $C_{14}H_9N_3O_5$, *Diazido,* $C_{14}H_8N_6O_5$, or higher nitro derivs were found in the literature thru 1966

Refs: 1) Beil **10**, 84, (40), [54] & {147} 2) Beil **10**, not found (Nitro derivs) 3) C.

Dufour et al, BullFr **1951**, 104–06 & CA **45**, 8478(1951)

Di-Salz. German designation of Dimethylammonium Nitrate, described in this Vol, p D1313-L

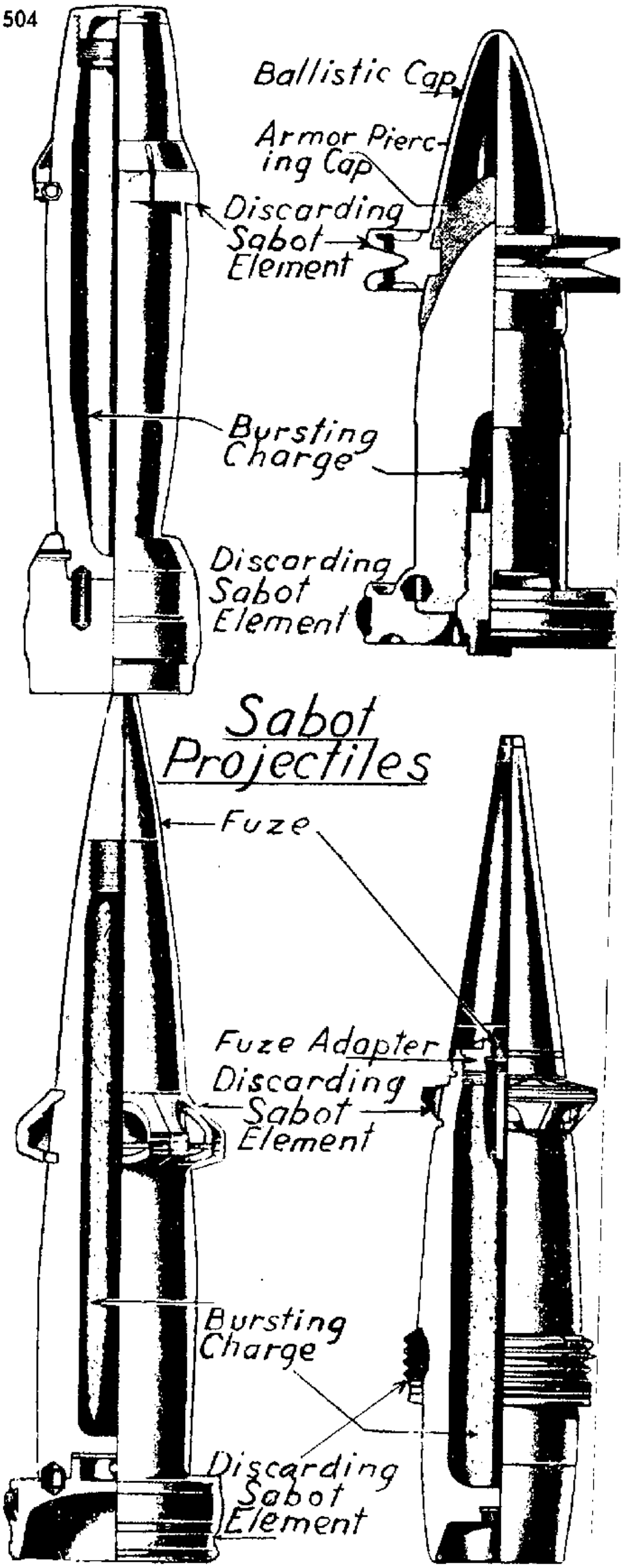
Disappearing Carriage. Type of fixed mount in which a balancing mechanism lifts the major caliber cannon to a position above its protective parapet for the act of firing, after which the cannon again "disappears" behind the parapet
Ref: Glossary of Ord (1959), 98-L

Disappearing Target. Target that is fully exposed to the firer's view for a short time. A practice disappearing target which is pulled down, or turned so that only its edge faces the firer, during the time that the target is not fully exposed is called a *Bobbing Target* (Ref, p 40-L)
Ref: Glossary of Ord (1959), 98-L

Disarm. To remove the detonating device or fuze of a bomb, mine, shell, etc or otherwise render it incapable of exploding in its usual manner (Ref, p98-L)

Compare it with *deactivate*, which means to render an explosive inert or harmless (Ref, p89-L) and with *defuze*, which means to remove the fuze from a munition (Ref, p91-L)
Ref: Glossary of Ord (1959), 89, 91 & 98

Discarding and Nondiscarding Sabot Projectiles. *Sabot* is a French term for a wooden shoe made of one piece, worn by peasants in Belgium, France and Holland. In Ordnance, "sabot" means a lightweight carrier, (usually made of brittle metal or plastic) in which a subcaliber projectile (core) is encircled. The sabot diameter is about twice as large as that of the core and it fills the bore of the gun. The principle of "sabot projectile" is to allow its firing from large caliber weapon in order to have a larger surface exposed to pressure



of propelling gases and then to have the surface reduced (after breaking and discarding sabot) so that the air resistance becomes small. A sabot which drops from the core of a projectile a short distance after leaving the muzzle of the gun is called *discarding sabot.* Several types of German projectiles of WWII using such sabots are shown in Figs from p Ger 171 of Ref 2

American "hypervelocity armor-piercing discarding sabot" (HVAPDS) projectile consists of a tungsten carbide core and a discarding sabot (Ref 3)

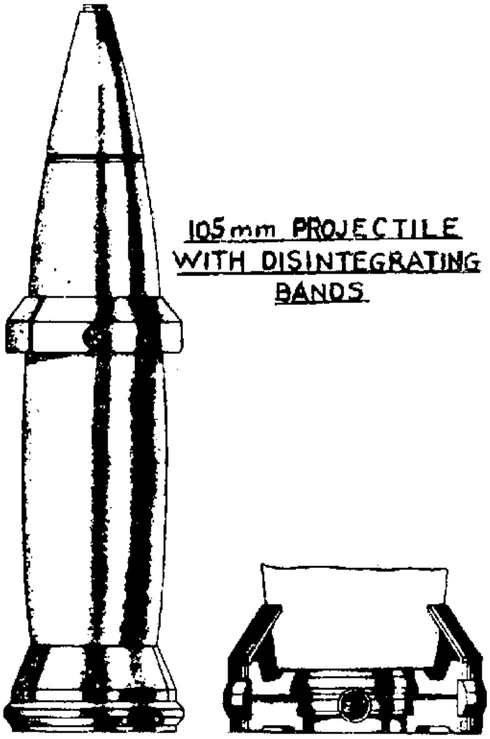
There are also lightweight sabots which do not discard on leaving the gun but remain on the core. The so-called "hypervelocity armor-piercing" (HVAP) projectiles are provided with such sabots. Because of the low total weight of such projectiles, "hypervelocity" (muzzle velocity of 3500 feet per second or more) is obtainable within allowable pressure of the gun tube. The velocity is rapidly lost, but at short ranges the projectile is effective against armor (Ref 3)

One type of German sabot projectile, used during WWII, consisted of a sintered tungsten carbide core and the softer sabot which did not discard until the projectile just began to penetrate inside the target (such as armor of the tank). After this the whole projectile disintegrated, spraying many deadly fragments inside the target (Ref 2)

Some of the German sabot projectiles were provided with one or two discarding bands, each in one piece. They were fired from normal rifled guns. Some of these projectiles are briefly described here as "Disintegrating Rotating Band Projectiles" (Ref 2)

Refs: 1) Armament Engineering (1954), 209 2) PATR **2510**(1958), p Ger 170 (Treibspiegelgeschoss) 3) Glossary of Ord (1959), 153 (Hypervelocity armor-piercing discarding sabot projectile); 250-L (Sabot)

Disintegrating Rotating Band Projectiles. Germans used during WWII the "sabot" type projectiles of 105mm & 150mm diam, which contained at the shoulder a detachable guide band, that was almost completely trisected by cuts, spaced 120 degrees apart. This band served as a bourrelet. The soft iron rotating band and its holder consisting of three detachable segments) were located at the base of the proj, which was keyed to receive them



105mm PROJECTILE WITH DISINTEGRATING BANDS

It was believed that after leaving the gun, the bourrelet and the driving band holder each split into three separate segments which were thrown off together with the pieces of metal which initially held them on the proj. The stripped proj not only had a better aerodynamic shape than conventional proj but also was 30% lighter (See Fig) (Refs 1–4)

Refs: 1) E. Englesburg, Ordn Sergeant, May 1944, p 308 2) L.E. Simon, "German Research in WWII", Wiley, NY (1947), 189 3) Anon, Dept of the Army Tech Manual, **TM 9-1985-3** (1953), 363–71 4) Fedoroff et al, PATR **2510**(1958), p Ger 37-R

Disk Test using a lead disk is one of the output tests measuring brisance of a stab detonator. It is briefly described, with Fig of apparatus, in Vol 4 of Encycl, p D1084. A more detailed description & evaluation of this test is given by V.J. Menichelli, Ordn **48**, 665–66(1964) who concluded there was non-correlation of this test with ability to cause detonation and was inadequate in some applications

Dislite (Called Dyslyt by Baup). A compd obtd together with *Eulite* by the action of nitric acid on citraconic acid. See Vol 3, p C324-L

Disodium-5-nitraminotetrazole. See under Aminotetrazole in Vol 1, p A259-R

Disol. Ger and Swiss name for 2,4-Dinitroanisole, described under Anisole in Vol 1, p A448-L

Dispenser M106, (Amer), known as **Mity Mite.** It evolved from a need to flush Viet Cong from tunnels. Employed as a blower unit, it forces a tear agent or smoke cloud through underground hiding places. An adaptation of a commercial sprayer-duster, it is portable and is powered by a 3.5-horsepower gasoline engine. The blower can displace 450 cubic feet of air a minute. It was manufd during Vietnam War by the Edgewood Arsenal, Maryland
Ref: Anon, Ordnance, July–August 1968, p 30-L

Dispersed System; Disperse Phase; Dispersing Agents and Dispersion Medium. *Dispersed system* is an apparently homogeneous system which consists of a microscopically heterogeneous mixture of two or more finely divided phases, eg: liquid-liquid (emulsions such as milk); liquid-solid (suspension, such as clay in water); gas-liquid (aerosols, such as fogs, clouds, mists) and gas-solid (such as smoke, dusts)

The phase which is distributed in a discontinuous manner in the larger amount of continuous phase, is called the *disperse phase*, while the continuous phase is called *dispersion medium*. In the case of milk, oil is the "disperse phase", while water is the "dispersion medium"

When the "dispersion medium" is a liquid, then the agent which helps the dispersion *(dispersing agent)* belongs to a class of substances called "surface active agents"

Dispersing agents which help the formation of emulsions are called "emulsifiers" and those which help in removing dirt are called "detergents"
Ref: Hackh's Dict (1944), 282 and the books on "Colloids", "Surface Active Agents", "Surface Chemistry and Physics, etc"
Note: In case of colloided smokeless proplnts, NC is "the dispersed phase"; solvent for NC – "the dispersion medium", which may also be called "the dispersing agent", and the propellant itself "the dispersed system"

Dispersion in Detonation (and Explosion). See under Detonation (and Explosion), Lateral Expansion (Dispersion) and Lateral Loss, in Vol 4, pp D421-R to D423-L

Dispersion in Shooting. In artillery it is the scattering of shots fired on a target by the same gun or group of guns. In aerial bombing it is the scattering of bombs dropped under approximately the same conditions upon a target

A **Dispersion Diagram** is made by superimposing the dispersion ladder (qv) for direction on the dispersion ladder for range and indicating in each rectangle the percentage of shots expected to fall therein

Dispersion Error is the chance variation in a series of shots even though firing conditions are kept as constant as possible. For practical purposes, it is considered the distance from point of impact or burst to the center of impact or burst

Dispersion Ladder is a table that shows the probable distribution of a succession of shots made with the same firing data. It consists of 8 zones, in each one of which is shown the percentage of shots expected to fall within it. It may be made for direction or for range

Dispersion Pattern is the distribution of a series of shots obtd under conditions as nearly identical as possible

Refs: 1) E. Shaitanov, MAF **14**, 731–86 (1935) (Sur les lois de dispersion) 2) T. Felsztyn, Wyadomosci Techniczne Uzbrojenia **1935**, No 29, p245; MAF **18**, 706–67 (1937) (Relation entre la dispersion d'une arme et les conditions à imposer pour le tir avec cette arme) 3) J.Dupuis, MAF **18**, 899–958(1939) (Notions sur la dispersion) 4) Glossary of Ord (1959), 98-R (Dispersion) 5) OrdTechTerm **ST 9-159** (1962), 102

Dispiro [fluorene-9,3'-s-tetroxane-6,9-fluorene]. The name given in CA's for the compd listed by us as 9,9'-Bis(fluorenyl)-diperoxide in Vol 2, p B143-L

Displacement Process (or Thomson's Method) of Manufacture of Nitrocellulose. See under CORDITE in Vol 3, p C538-L

Displacement Pumps operate by forcing the liquid that is to be moved from one point to another by means of pressure exerted by a gas or vapor. The "acid egg" (Vol 1, p A88-L) is a device of this type frequently encountered in chemical industry

Ref: Perry (1950), 1439

Display Rocket. It is a rocket used for pyrotechnic display. It may be constructed using a cardboard case with a chamber in the forward end separated by a plug of clay or other material thru which a piece of quick match passes. The chamber is filled with black powder and colored light compositions shaped in the form of stars, primed with mealed powder. The tail end of the rocket contains a propellant and a guiding stick. When the rocket is shot into the air and reaches its maximum height, flame from the propellant is conveyed by quick match to the forward section, igniting the charge of black powder and the stars. This bursts the walls of the chamber and scatters brightly burning stars

Refs: 1) Marshall **2**(1918), 608–09 2) Davis (1943), 74–8

Disposal of Bombs. See "Bombs, Disposal of" in Vol 2, p B234-L and also "Bombs, Destruction of", p B233-L

Disposal of Captured German Warfare Materials. The disposition of more than 12000 tons of munitions and toxic chemicals, including mustard gas, arsenicals & chloroacetophenone, is described by Loucks & Elliot (Ref)

Ref: C. E. Loucks & R.B. Elliot, ArmedForcesChemJ **3**, No 4, 14–15(1949) & CA **43**, 3947(1949)

Disposal of Defective or Obsolete Projectiles in Russia. Accdg to Shilling (Ref), the following methods can be used:

Method 1, used when it is not desired to save either casing of projectile or explosive

After removing the fuzes, the projs are placed in piles on the bottom of a specially constructed trench. One of the projs is provided with an electric detonator and a booster. After covering the trench with earth, the proj provided with a detonator is exploded. This causes the detonation of other projs, although they have no detonators. After this the metal is collected as scrap and the trench reloaded

Method 2, used when it is desired to save the casing, but to destroy the expl

The defuzed projs are placed nearly horizontally in piles in an open fire burning in a pit. The openings of the projs should face the fire in such a manner that the flame touches only the expl and not the casing of proj. This is in order to prevent detonation and to have the expl burn while pouring out

Method 3, used if it is desired to save the expl & casing. If an expl melts at a temp below 100° (such as TNT, Pentolite, Cyclonite, etc), the procedure is as follows:

The defuzed projs are placed vertically with open ends downward inside a long rectangular tank provided with a perforated false bottom. The ogival part of each proj should rest in one of the holes of false bottom. Then the tank is partly filled with water and live, low pressure steam is injected thru a perforated pipe located below the projs. This causes the water to boil and the heat melts the expl, thus emptying the projs. The molten material is accumulated at the bottom of the tank and then drained. The expl thus collected is usually suitable for industrial uses

If the proj is charged with an expl which does not melt at the temp of boiling water (such as PA, Tetryl, etc), the removal from projs is accomplished by means of live, superheated steam. In this method, a long jet of steam is directed against the expl thru an opening in the proj. The latter is usually on an inclined rack in such a manner that, as part of the expl is removed, the proj slides in the direction of the jet so that the tip of the jet is always touching the surface of the expl

The superheated steam method is more dangerous than that using low pressure steam, expecially when projs were stored for a long time in hot climate. However, it permits the recovery of high melting expls which are either insol or only partly sol in water

If an expl compn is a blend of sol expl (such as AN) and insol low melting expl (such as TNT), low pressure steam may be used, but if the insol component is high melting, the superheated steam may be preferred. The soluble expls are usually not recovered
Ref: Shilling (1946), 231–34

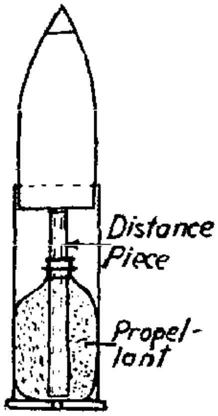
Disposal of Waste Ammunition, Explosives, Propellants and Pyrotechnic Compositions. See Under Decomposition, Destruction and Disposal of Ammunition, Explosives, etc in Vol 3, pp D23-L to D25-R
See also Destruction and Disposal of Explosives, pp D25-R to D35-R

Disposal Slide Rule. This is a handy, ready reference slide rule for Explosive Ordnance Reconnaissance & Explosive Ordnance Disposal personnel for use during identification and disposal of CBR (Chemical, Biological & Radiological) agents which might be encountered in expl ordn incidents or accidents. The methods of disposal outlined in the slide rule are for the guidance of trained personnel and not intended for use by unskilled persons unfamiliar with the items or procedures covered
Ref: Lt. R.R. Vennell, "CBR Munitions and Reconnaissance and Disposal Slide Rule", PATR **3307** (Sept 1965)

Dissociation Pressure. The sum of the partial pressures on breaking apart of a molecule by physical means
Ref: Hackh's Dict (1944), 283-R

Dissolving Tank Explosions – Practical Step Towards Eliminating. A device is described for controlling dissolving tank explns in a sulfate mill by shattering the smelt stream with a mech fan-like device above the liquor level in the tank
Ref: C.P.R. Cash, PaperTradeJ **140**, No 15, 28, 30(1956); Pulp & Paper Mag Can **57**, No 3, 320(1956) & CA **50**, 17459(1956)

Distance Piece or Cross Tube (Kreuzrohr, in Ger). This device was used in Germany when a proplnt charge of semi-fixed artillery ammunition was shorter than the cartridge case. The piece consisted of one or several tubular sticks of



double-base proplnt, which were inserted into the proplnt bag and tied tightly at its neck. The upper end of the sticks extended as far as the bottom surface of a closing cup (or the base of the proj), while the lower end held the bag against the primer. With this arrangement the proplnt chge was not loose and, being held close to the primer flash hole, the proplnt was readily ignited
Ref: Fedoroff et al, PATR **2510**(1958), p Ger 38-L

Distance Piece and Distance Wadding. When the capacity of a cartridge case in fixed ammo is greater than the volume of the proplnt chge, some inert material (such as a rectangular cardboard sheet folded into a triangular shape), is placed to occupy the excess volume and to keep the proplnt back around the primer. In case of separate-loaded ammo the cardboard serves to maintain the proper distance betw the mouth plug and the wad which covers the proplnt chge
Ref: Glossary of Ord (1959), 99-L

Distances for Storage of Explosives, American Table of. See Effect of Explosions and Table of Distances for Storage of Explosives

DISTILLATION

(Batch Distillation; Evaporation and Condensation; Continuous Distillation; Fractionation; Rectification; Reflux Distillation; Vacuum Distillation; Steam Distillation; Azeotropic & Extractive Distillation; Destructive Distillation; Molecular Distillation; Distillation by Compression; and Sublimation)

Distillation is a unit operation designed to separate a slurry or a solution into volatile and non-volatile components, using either heat or other methods (such as blowing an inert gas thru a liquid or application of a very reduced pressure)

The simplest example is the *batch distillation*, conducted in an apparatus such as used in the laboratories. It consists of a flask (or retort), connected to a condenser (cooled by water or air), and a receiving vessel. This method is also known as *evaporation* (Refs 1, 4, 7 & 17). It is followed by *condensation* and the process can be conducted under atmospheric pressure or under reduced pressure (known as *vacuum distillation)* if it is required to conduct the operation at lower temperature in order to avoid decomposition (Refs 8 & 15). Some distillations are conducted under excess pressure (Refs 5a &15)

A similar apparatus but of a larger size can be used for "industrial batch distillation". Examples of "batch stills" are shown in Figs 290, 291 & 292, pp 384–86 of Riegel's book (Ref 1)

When a mixture of several volatile substances has to be separated the batch method is replaced by *continuous* method using *fractionating column* and the process is known as *fractionation.* This is defined by Riegel as a "multiple distillation". Fractionating columns can be either "plate towers" (using bubble caps or sieve plates) or "packed towers" (using Raschig rings). Various types of towers are shown in Figs 293, 294, 295 & 296, pp 387–89 of Riegel's book (Ref 1) (See also Refs 9, 10, 14 & 15)

Liquids which contain 3, 4 or more components cannot be separated into the different pure components in one operation even in a plate column. Nevertheless, they can be separated into mixtures boiling within narrower, carefully defined limits. The plate column is tapped at various levels and "cuts" are drawn off, which are generally commercial products without further treatment. When it is required to obtain purer distillates (as for example in production of grain alcohol), the process known as *rectification* is used. This is defined by Perry (Ref 13, p 563) as a distillation carried out in such a way that the vapor rising from a still comes in contact with a condensed portion of vapor previously evolved from the same still. A transfer of material and an interchange of heat result from this contact, thereby securing a greater enrichment of the vapor in the more volatile components than could be secured with a single distillation operation using the same amount of heat. The condensed vapors, returning to accomplish this object are termed *reflux.* The generally used devices in which vapors from a still on their way to condenser can flow counter-currently to a portion of the condensate return as reflux, are called *rectifying columns* or *towers*. This method of purification may also be called *reflux distillation* (Ref 1, p387 & Refs 6, 10 & 15)

Azeotropic distillation was briefly described in Vol 1 of Encycl, p 318, under "Azeotropy" (Ref 20)

Perry (Ref 13, p 634) and Weissberger (Ref 15, pp 317 to 387) describe azeotropic distillation together with *extractive distillation*. Perry says that these distillations are *fractionations* that are facilitated by deliberately adding a new component to the system. This component, being judiciously chosen to shift the vapor-liquid equilibrium in a favorable direction, may be referred to as a "separating agent". The operation is called an *azeotropic distillation* if the separating agent forms one or more azeotropes with components in the system (usually minimum-boiling azeotropes) and by virtue of this fact is present in appreciable concentration on most of the plates in the rectifying column

The operation is called an *extractive distillation* if the separating agent is relatively non-volatile compared with the components to be separated and, therefore, is charged continuously near the top of the distilling column in order to maintain an appreciable concentration of separating agent in the liquid on most of the plates of the column. The basis for extractive distillation is the change of volatility caused by introducing the solvent. The change is not the same for each component, and the resulting difference in volatility permits fractionating the feed components, usually with fewer plates and at a low "reflux ratio" (See also Refs 12, 14 & 18)

Some difficultly volatilized substances (such as those with boiling points above 100°) can be distilled under reduced pressure (or in *vacuum*), as is mentioned above. In some cases, however, the temperature of vaporization may be lowered if "live" steam is introduced thru a pipe near the bottom of the still. As a result of this operation, one (or several) substances desired to be removed are carried over by steam to a receiver where they are collected, condensed, and form, after standing, a layer separated from water layer. Steam used in this method, known as *steam distillation*, is termed "open steam" and it can be superheated for removing substances requiring high temperatures for volatilization (See Refs 6, 12, 13 & 15)

Destructive distillation includes those operations in which the material under process, heated out of contact with air, first undergoes thermal decomposition and the volatile products formed are then withdrawn as vapors for recovery. The method is designed to decompose complex organic substances into a number of split products (Ref 2a, p283 & Ref 13, p563)

For example, destructive distillation of wood gives methanol (wood alcohol), wood tar or pitch and charcoal. Destructive distillation of bituminous coal gives coal gas, coal tar, coal tar pitch and coke. These products are described in Vol 3 of Encycl, pC379

Molecular distillation is designed for separating compounds of extremely high molecular weights and for separating liquids of boiling points in the range of less than 0.01mm Hg. Description of the method and its uses are given in Refs 2, 7, 12 & 18)

Under the title *distillation by compression* is mentioned in Army Ordn (Ref 5a) a method of prepn of fresh water from sea water developed by R.V. Kleinschmidt of the US Navy during WWII. The method involves distillation by means of mechanical energy instead of externally applied heat

Sublimation. Accdg to definition given in Perry (Ref 13, p660), it covers the physical changes encountered by a substance in passing from a solid phase to a gas and back to a solid phase. It is characterized by the absence of the liquid phase and is used for purification of volatile substances like iodine, camphor, etc. Detailed description of the process is given in Refs 12, 13, 15 & 18). A brief definition is given in Ref 2a, p813

Refs for Distillation:

1) E.R. Riegel, "Chemical Machinery", Reinhold, NY (1944), 382–95 (Distilling and fractionating equipment) 1a) Ibid, 2nd edition (1953), 481–509 2) K.C. Hickman,

ChemRevs **34**, 51–106(1944) (Molecular distillation) (110 refs) 2a) Hackh's Dictionary (1944), pp 283 & 813 3) K.C. Hickman et al, IEC **38**, 28–9(Jan 1946) (Molecular distillation), and in succeeding years in the January numbers, under "Unit Operations") 4) W.L. Badger & R.A. Lindsey, IEC **38**, 4–5 & 30–31(1946) (Evaporation), and in succeeding years under "Unit Operations") 5) T.J. Walsh, IEC **38**, 8–9 & 31–32(1946) (High temperature distillation), and in succeeding years under "Unit Operations" 5a) Anon, Army Ordn, **31**, 18 (July–August 1946) (Distillation by compression) 6) H. Stage & G.R. Schultze, "Theory, Apparatus and Processes of Distillation and Rectification", translated from the German edition of 1944 and published by Hobart Publishing Co, Washington, DC (1947) (Contains bibliography 1920–1944) 7) E.S. Perry, "Methods of Laboratory Molecular Distillation", Distillation Products Inc, Rochester, NY and AnalChem **20**, 392(1948) 8) K.C. Hickman, IEC **40**, 16–18(1948 (High-vacuum distillation), and succeeding years in January numbers under "Unit Operations") 9) T.P. Carney, "Laboratory Fractional Distillation", Macmillan, NY(1949) 10) A. Rose, AnalChem **21**, 81–4(1949) (Analytical distillations, including simple laboratory distillation and all forms that involve rectification and fractionation) (154 refs) 11) G.G. Brown, Edit, "Unit Operations", Wiley, NY (1950), pp 322 & 388 (Distillation) 12) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **5**, pp 156–187 (Distillation, including molecular distillation); 927–948 (Evaporation) 13) J.H. Perry, Edit, "Chemical Engineers' Handbook'', McGraw-Hill, NY(1950), pp 561–660 (Distillation and sublimation) 14) C.S. Robinson & E.R. Gilliland, "Elements of Fractional Distillation", McGraw-Hill, NY(1950) 15) A. Weissberger, Edit, "Physical Methods of Organic Chemistry", Interscience, NY, Vol **4** (1951), pp 1 to 174 (Theory of distillation); 175 to 316 (Ordinary fractional distillation); 317 to 387 (Extractive and azeotropic distillation); 389 to 461 (Distillation of liquefied gases and low-boiling liquids); 463 to 494 (Distillation under moderate vacuum); 495 to 602 (Distillation under high pressure) 16) C.H. Nielson, "Distillation in Practice", Reinhold, NY(1956) 17) E.J. Zuiderweg, "Laboratory Manual of Batch Distillation", Interscience, NY(1957) 18) P.H. Groggins, Edit, "Unit Processes in Organic Synthesis", McGraw-Hill, NY(1958), pp—not found (Distillation) 19) M.G. Larian, Edit, "Fundamentals of Chemical Engineering Operations", Prentice-Hall, Englewood Cliffs, New Jersey (1958), pp 343-424 (Distillation) 20) Encyclopedia of Explosives, PATR **2700**, Vol **1** (1960), p A518(Azeotropy) 21) Perry, 4th Edit (1963), pp 13-2 to 13-55 22) Kirk & Othmer, 2nd edit **7** (1965), pp 204–48 (Distillation)

Distler, Blecher & López Explosives were patented safety expls consisting of nitroxylenes as chief constituents. The nitro compds had varying amts of cumol, mesitylene & xylene nitro constituents mixed with AN, other nitrates or $KClO_3$ or metal oxides. For example, if 12 parts nitrated solvent naphtha are intimately mixed with 88 parts of AN, a very insensitive expl is produced, which is unaffected by either shock or percussion; which does not explode at 200° or in an open flame, but which has an extremely powerful shattering effect when suitably detonated
Ref: Colver (1918), p259

Distress Signals. When in distress, one can use pyrotechnic devices which produce in daytime smokes, while in night time lights (of various colors), or stars are produced

Distress signals can be subdivided into flares, smoke grenades & stars of distinctive colors. Marine signals are used by aircraft personnel when forced down in water. Some are hand-held, cartridge-type projected by a Very Signal Pistol, others are floating type emitting orange smoke, and smoke & illumination types

They are described in the following Refs:
Refs: 1) Ohart (1946), 320 2) Weingart (1947), 72 3) TM **9–1981** (1951), 130–38 4) J.J. Berliner, "Fireworks", ResRept **No 3834**, NY(1953), p45 5) TM **9–1370–200**

(1958), p98 & others; TM **9–1370–200**(1966), pp 4–33ff 6) Ellern (1961), 276 & (1968) pp151–59

Distribution of Charge in a Bore Hole. This important factor is, accdg to Berliner (Ref), often overlooked. For example, in pulling a heavy burden, such as in cut holes in driving tunnels, it is important that sufficient explosives' energy be concentrated in the back of the hole. It is equally important that the explosive does not extend to near the collar of the hole

On the other hand in *slabbing* or *rib shots*, best results are usually obtd when the expl charge is well distributed thru the bore-hole. In the case of slabbing, a dense, *high bulk strength* explosive is required, while in the rib shots a *low bulk strength* explosive is advised

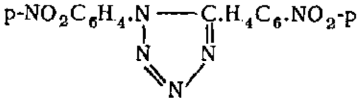
Ref: J.J. Berliner, Expls (1953), p5

Note: *Slabbing* or *Rib Shots* are cutting of a skip (omission or skip over) from the side of a face of coal or stone

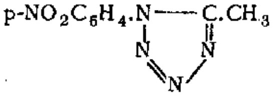
Distyryl-benzene. See Bis(styryl)-benzene in Vol 2, p B156-L

1,5-Disubstituted Tetrazoles. The prepn of 1,5-disubstituted tetrazoles by the coupling of diazotized p-nitroaniline with 1-acetyl-2-para-substituted benzoyl hydrazines and cyclization of the tetrazenes formed as intermediates has been investigated. From the ratio of the two tetrazoles isolated:

1,5-Di(4'-nitrophenyl)tetrazole,



1-(4'-nitrophenyl)-5-methyltetrazole,



it appears that preferential coupling occurs at the nitrogen adjacent to the para-substituted benzoyl residue (Refs)

The prepn of a variety of 3,5-diaryl-1-(5'-tetrazolyl)(IH) tetrazolium betaines by introducing electronegative substituents in the benzene residue of 1-(5'-tetrazolyl)-3-phenyltetrazene, together with the use of substituted benzaldehydes is reported (Ref 3)

Refs: 1) J.P. Horwitz & V.A. Grakauskas, JOrgChem **19**, 194–201(1954) & CA **49**, 3173 (1955) 2) Ibid, JACS **77**, 6711(1955) & CA **50**, 12036(1956)

Disuccinic Acid Peroxide; Disuccinyl Peroxide or Bis(3-carboxypropionyl)-peroxide. It is described as Bis(succinyl) -peroxide in Vol 2, p B156-R

The commercial product manufd by the Lucidol Div, Novadel-Agene Corp (Ref) was 90.0% (minimum) pure with active oxygen 6.15% (min) and mp 125° (with decompn)

Ref: Lucidol Division, Novadel-Agene Corp, Buffalo, NY, "Organic Peroxides Pamphlet", Data Sheet No 14 (1947)

Ditane. One of the names for Diphenylmethane, described in this Vol

Ditch Blasting for Pipe Line Construction is described in Blasters' Hdb (1952), pp 420–22; Ibid (1958), pp 423–22 and Ibid (1966), pp 394–98

Ditching, Agricultural. See "*Digging Ditches*", item f in Vol 1, p A113-R under "Agriculture and Forestry Uses of Explosives" and also in Blasters' Hdb (1952), pp 429–39; Ibid(1958), pp 379–88 and Ibid(1966), p367–78

Di(5-tetrazole)-hydrazine or 5,5'-Hydrazo-bis(tetrazole). See Bis(5-tetrazole)-hydrazine in Vol 2, p B157-L

Ditetrazolyl-(5,5'). A Ger name for 5,5'-Ditetrazolyl, described as 5,5'-Bis(1,2,3,4-tetrazole) in Vol 2, p B157-L

Di(tetrazolyl-5)-dihydro-sym-tetrazine. See 3,6-Bis(2H-tetrazolyl-5)-dihydro-1,2,4,5-tetrazine in Vol 2, p B157-R

Di[tetrazolyl-(5)]-diimide. Ger name for Azotetrazole described in Vol 1, pp A659 & A660-L

N^1,N^6-(Ditetrazolyl-5)-hexazadiene. See [N^1,N^6-Bis(*a*-tetrazolyl-5)]-hexazadiene under 5-AMINO-*a*-TETRAZOLE in Vol 1, p A260-R

(Ditetrazolyl-5)-hydrazine; Di[(4-hydrotetrazolyl)-5]-azine; Azino-di[4-hydrotetrazole)-5] or Hydrazo-di(tetrazolyl-5). See Bis(5-tetrazole)-hydrazine in Vol 2, p B157-R and in F.R. Benson, ChemRevs **41**, 8(1947)

Di(tetrazolyl-5)-sym-tetrazine. See 3,6-Bis(2H-tetrazolyl-5)-1,2,4,5-tetrazine in Vol 2, p B158-L

I,III-Di(tetrazolyl-5)-triazene. See 1,3-Bis(1H-tetrazolyl-5)-triazene in Vol 2, p B158-L.

Its *Monotriethyl Lead Salt* is on p B158-R and **Ditriethyl Lead Salt** is on the same page

Ditetryl or Bitetryl (US), **Octyl** (GtBrit). See 1,2-Bis(2',4',6'-trinitro-nitranilino)-ethane in Vol 2, p B131-R
Addn Refs: A) Ye.Yu. Orlova, "Khimia i Tekhnologya Brizantnykh Vzryvchatykh Veshchestv", GosIzdatOboronProm, Moskva (1960), Engl translation, "Chemistry and Technology of High Explosives", by Technical Documentation Liaison Office, MCLTD, WP-AFB, Ohio, MCL-844/1+2, June 1961, p 472 (compd called Ditetryl) B) R. Simkins, "Ditetryl", ERDE (Explosives Research & Development Establishment), TechMemo **No 21/M/64**, Sept 1964 C) T. Urbański, "Chemistry and Technology of Explosives", Translated from Polish, Pergamon Press, NY, Vol **3** (1967), p 65 (Compd called Ditetryl)

Dithekite. A Sprengel type expl consisting of NB (Nitrobenzene) and nitric acid *Dithekite 13.* It is prepd by mixing, with cooling, 78 parts of benzene with 42.5 parts of 88.9% nitric acid; small amt of phosphoric acid is added to prevent corrosion of container. The resulting soln of NB in nitric acid had d 1.37 & bp 118°; fumes off at 128°. It is not sensitive to impact or to rifle bullet; Power by Trauzl Test 116% PA & by Ballistic Mortar Test 124% PA; Heat of Detonation 1187cal/g; Detonation Rate above 6000m/sec; Stability – fairly stable up to 100°, but fumes off at 128°(Refs 1 & 2)

Cook (Ref 3) gives its compn as: NB 24, nitric acid 63 & water 13%
Refs: 1) A.H. Blatt, OSRD Rept **2014**(1944) 2) ArmamentEngrg(1954), 71 3) Cook(1958), 190–94

Dithiocarbamate of Ammonium, $H_2N.C.S.S.NH_4$; mw 110.20, N 25.42%; lt-yel unstable crysts; mp – decomp; sol in w and in soln the product can be preserved for several weeks; the dry product can be preserved for several days at 0° with little decompn. Its lab prepn from ammonia and carbon disulfide is described in Ref 2

Detailed listings of its props & reactions are found in Ref 1
Refs: 1) Beil **3**, 216, (86), [155] & {353} 2) OrgSynth **3**(1950), 48–50 (7 refs); OrgSynthCollective Vol **3**(1955), 763–65

Di(thiocarbamyl)-hydrazine. See N,N'-Bis-(thiocarbamyl)-hydrazine in Vol 2 of Encycl, pp B158-R & B159-L

Dithiocarbonyl Azide. See Azidodithiocarbonic Acid and Derivatives, Vol 1, p A632-L

Dithiocarbonyldiazide Disulfide or **Azidocarbondisulfide,** $(N_3.CS)_2S_2$, mw 236.33, N 35.56%. See Diazidodithioformyl Disulfide or Diazidodithiocarbonyl Disulfide in this Vol

Dithio-hydrazo-di carbonamide. One of the names for N,N'-Bis(thiocarbamyl)-hydrazine, described in Vol 2, p B158-R

1,2-Dithiol-ethyl-phosphorous Acid, $(CH_2S)_2PO.C_2H_5$, mw 128.24; liq with unpleasant odor; sp gr 1.2629 at 20/0°, n_D 1.5922 at 20°; bp 98–99° at 5mm. It can be prepd by adding $C_2H_5OPCl_2$ dropwise to ice-cooled $(CH_2SH)_2$ with pyridine in ether. It ignites with flame on contact with concd nitric acid (Ref 2). It yields adducts with CuCl & CuI which rapidly decomp in air

1,2-Dithiol-methyl-phosphorous Acid, $(CH_2S)_2PO.CH_3$, mw 154.20; liq with unpleasant odor; bp 97–98.5° at 7.5mm press, sp gr 1.3426 at 20°, n_D 1.6200 at 20°. It can be prepd by adding CH_3OPCl_2 dropwise to ice-cooled $(CH_2SH)_2$ with pyridine in ether

It ignites with flame on contact with concd nitric acid

Heating of methyl derivative with CH_3I in a sealed tube produced a dark liquid which exploded with loss of the product

Refs: 1) Beil, not found 2) A.E. Arbuzov & V.M. Zoroastrova, IzvestAkadNauk(OtdelKhimNauk) **1952**, 453–58 & CA **47**, 4833(1953)

Dithizonates or **Dithiozonates.** These are salts of dithizone or diphenylthiocarbazone, $C_6H_5N:N.CS.NH.NH.C_6H_5$. Dithizonates of silver, lead, zinc & copper were prepd in two ways: 1) pptn by addn of the cation soln to an ammoniacal soln of dithizone and 2) treatment of a chlf soln of dithizone with an aq soln of the cation. All showed rapid, almost expl decompn at the following temps: *Silver* 186°, *Lead* 215°, *Zinc* 225° & *Copper* 150°. Above the expl decompn, slow addnl decompn proceeds with the final formation of Ag, PbS, PbO, ZnO & CuO, respectively

Ref: J.C. Pariaud & P. Archinard, Rec **71**, 634–38(1952) (in French) & CA **46**, 11043 (1952)

Di(toluidino)-propane. See Bis(methylphenylamino)-propane in Vol 2, p B151-L

Ditolyl or **Bis-tolyl.** See Bitolyl in Vol 2, p B163-L

Ditolyl-amine or **Dimethyl-diphenylamine and Derivatives**

Ditolyl-amine, $(CH_3.C_6H_4)_2NH$, mw 197.27, N 7.10%. The following isomers exist:

Di-o-tolyl-amine(2,2'), liq, bp 312° at 727.8mm (Ref 1)

Di-m-tolyl-amine(3,3'), liq, bp 320–324° (Ref 2)

Di-p-tolyl-amine(4,4'), ndls, mp 79°, bp 330.5° (Ref 3)

Di-o,m-tolyl-amine(2,3'), lt-yel oil, fr p <–15°, bp 187° at 22mm press (Ref 4)

Di-o,p-tolyl-amine(2,4'), lt-yel liq, fr p – does not solidify on cooling, bp 183° at 19mm press (Ref 5)

Di-m,p-tolyl-amine(3,4'), liq, bp >300° (Ref 6)

All six are very sl sol in w

Snelling & Wyler (Ref 12) proposed Ditolylamine (isomer not specified) as a sensitizer for AN

Azido, $C_{14}H_{14}N_4$, and *Diazido,* $C_{14}H_{13}N_7$ derivs of Ditolyl-amine were not found in the literature thru 1966

Dinitro-ditolylamine or **Dinitro-dimethyldiphenylamine,** $C_{14}H_{13}N_3O_4$, mw 287.27, N 16.43%. The following isomers are found in Beil:

4,6-Dinitro-2,2'-ditolylamine, $CH_3.C_6H_2(NO_2)_2.NH.C_6H_4.CH_3$, red crysts (from alc), mp 109° (Ref 7, p 462)

2,6-Dinitro-3,3'-ditolylamine, brick-red crysts (from alc), mp 110° (Ref 7, p479)

4,6-Dinitro-3,3'-ditolylamine, orn-colored

crysts (from alc), mp 135°(Ref 7, p481)
2,6-Dinitro-4,4'-ditolylamine, yel crysts (at 60°) & orn-colored (<60°), mp 161°(Ref 7, p443)
2,2'-Dinitro-4,4'-ditolylamine, $CH_3.C_6H_3(NO_2).NH.(NO_2).C_6H_3.CH_3$, red ndls (from benz), mp 192–93°(dec) or yel-brn pltlts (from alc), mp 191°(Ref 7, pp1002 & 536)
4',6'-Dinitro-2,3'-ditolylamine, $CH_3.C_6H_2(NO_2)_2.NH.C_6H_4.CH_3$, brn-red crysts (from alc), mp 99°(Ref 7, p481)
2',6'-Dinitro-2,4'-ditolylamine, brick-red crysts, mp 124°(Ref 7, p443)
2,6-Dinitro-3,4'-ditolylamine, $CH_3.C_6H_4.NH.(NO_2)_2C_6H_2.CH_3$, red crysts, mp 131° or softens at 147° & melts at 152° (Ref 7, p494)
4,6-Dinitro-3,4'-ditolylamine, orn-colored crysts, mp 154° to a red liq; sol in benz & pyridine (Ref 7, p494)
2',6'-Dinitro-3,4'-ditolylamine, $CH_3.C_6H_2(NO_2)_2.NH.C_6H_4.CH_3$, dk-red crysts, mp 127°(Ref 7, p443)

Other props & methods of prepn are given in the Refs

Tetranitro-ditolylamine, $C_{14}H_{11}N_5O_8$, not found in the literature thru 1966

Hexanitrodi-o-tolyl-amine or **Bis(2-methyl-trinitro-phenyl)-amine,** $(O_2N)_3C_6H(CH_3)–NH–(CH_3)C_6H(NO_2)_3$; mw 467.27, N 20.98%, not found in the literature thru 1966

2,4,6,2',4',6'-Hexanitro-m-ditolyl-amine or **Bis(2,4,6-trinitro-m-methyl-phenyl)-amine,** $(O_2N)_3C_6H(CH_3)–NH–(CH_3)C_6H(NO_2)_3$, mw 467.27, N 20.98%. It was described in Vol 1 of Encycl, p A443-R as 2,4,6,2',4',6'-Hexanitro-3,3'-dimethyl-diphenyl-amine or Bis(3-methyl-2,4,6-trinitro-phenyl)-amine. All these names are correct, but the compd was erroneously placed under ANILINOXYLENE AND DERIVATIVES (Refs 8 & 11)

2,3,6,2',3',6'-Hexanitrodi-p-tolyl-amine. See 2,3,6,2',3',6'-Hexanitro-4,4'-dimethyl-diphenyl-amine, erroneously described in Vol 1, p A443-R under AMINOXYLENE AND DERIVATIVES (Refs 9 & 10)

Refs: 1) Beil **12**, 78, (377) & [437]
2) Beil **12**, 858 & [467] 3) Beil **12**, 907 (415) & [494] 4) Beil **12**, [467]
5) Beil **12**, [494] 6) Beil **12**, (414)
7) Beil **12**, 1002, (443) & [462, 479, 481, 494, 536] 8) Beil **12**, [482] (Hexanitro-m-ditolyl) 9) Beil **12**, 1013 (Hexanitro-p-ditolyl) 10) A. Lehne, Ber **13**, 1545(1880)
11) L. Brady et al, JCS **125**, 2404(1924) & CA **19**, 475(1925) 12) W.O. Snelling & J.A. Wyler, USP 1827675(1932) & CA **26**, 601(1932)

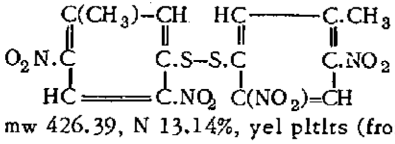
Di(tolyl) -disulfides or Dimethyl-diphenyl-disulfide and Derivatives
Di(or Bis)(tolyl)-disulfide, $H_3C.C_6H_4–S.S–C_6H_4.CH_3$, mw 246.37; exists as three isomers:
Di-o-tolyl-disulfide, pltlts (from alc), mp 38–39°(Ref 1)
Di-m-tolyl-disulfide, liq, fr p <–22°, bp – decomp (Ref 2)
Di-p-tolyl-disulfide, ndls, mp 47°, bp 210–15° at 20mm (Ref 3)

Other props & methods of prepn are found in the Refs

Dinitro-ditolyl-disulfide or **Dinitro-dimethyl-diphenyl-disulfide,** $C_{14}H_{12}N_2O_4S_2$; mw 336.40, N 8.33%. Several isomers are described in the literature:
4,4'-Dinitro-2,2'-ditolyl-disulfide or *Bis(4-Nitro-2-methyl-phenyl)-disulfide*, $H_3C.C_6H_3(NO_2)–S.S–(NO_2).C_6H_3.CH_3$, lt-yel ndls (from glac acet ac), mp 188° (Ref 4, p 1285)
5,5'-Dinitro-2,2'-ditolyl-disulfide, ndls (from alc), mp 147.3–48°; sol in eth & benz; mod sol in hot glac acet ac; sl sol in warm alc (Ref 4, p 1284)
6,6'-Dinitro-2,2'-ditolyl-disulfide, yel prisms (from benz), mp 150° or crysts (from aq alc), mp 145°(Ref 4, pp 343, 1284)
4,4'-Dinitro-3,3'-ditolyl-disulfide, tablets (from glac acet ac), mp 163°; sol in hot aq Na_2S soln (Ref 4, p 1339)
6,6'-Dinitro-3,3'-ditolyl-disulfide, yel-grn crysts (from glac acet ac or benz), mp 152° (Ref 4, p 1338)

2,2′-Dinitro-4,4′-ditolyl-disulfide, yel ndls (from glac acet ac) or prisms (from benz + benzine), mp 175–76°; sl sol in chlf, benz & hot glac acet ac; v sl sol in alc (Ref 4, pp 214, 401, 1439)

4,6,4′,6′-Tetranitro-3,3′-ditolyl-disulfide; 4,6,4′,6′-Tetranitro-3,3′-dimethyl-diphenyl-disulfide; or **Bis(4,6-Dinitro-m-tolyl)-Disulfide,**



mw 426.39, N 13.14%, yel pltlts (from xylene) or lt-yel ndls (from Nitrobenz+alc or Nitrobenz + solvent naphtha), mp 163–65°(dec), explodes on heating rapidly; darkens on exposure to light; v sl sol in most org solvs except xylene. It was first prepd by reaction of 5-chloro-2,4-dinitro-1-methylbenzene with Na_2S_2 in alc (Refs 5 & 6). It was prepd later by Kiprianov & Zhumrova (Ref 7) using somewhat the same procedure

Refs: 1) Beil **6**, 372 & [342] (ortho) 2) Beil **6**, 389 (meta) 3) Beil **6**, 425, (212), [400] & {1432} (para) 4) Beil **6**, (214), [343, 401] & {1284, 1285, 1338, 1339} 5) Beil **6**, 389 & [367] 6) G. Schultz & H. Beyschlag, Ber **42**, 748 (1909) & CA **3**, 1176 (1909) 7) A.I. Kiprianov & I.N. Zhumrova, DoklAkadNauk **85**, 789 (1952) & CA **47**, 9979 (1953); ZhurObshchKhim **23**, 493 (1953) & CA **48**, 3963 (1954)

Hexanitroditolyl-disulfide, $C_{14}H_8N_6O_{12}S_2$, not found in the literature thru 1966

Di(tolyl)-ethylenediamine. See Bis(methyl-phenyl-amino)-ethane in Vol 2, p B150-R

N,N′-Di(tolyl)-guanidine and Derivatives

Di(or Bis)(tolyl)-guanidine, $(H_3C.C_6H_4NH)_2C{:}NH$, mw 239.31, N 17.56%, exists in four isomers:

N,N′-Di-o-tolyl-guanidine, crysts, sp gr 1.10 at 20/4°, mp 178–79° (Refs 1 & 7)

N,N′-Di-m-tolyl-guanidine, ndls (from alc), mp 116–17° & 108–09° (Ref 2)

N,N′-Di-p-tolyl-guanidine, pltlts (from alc), mp 168–70° & 166.5–167.5° (Ref 3)

N-o-Tolyl-N′-p-tolyl-guanidine, ndls (from w), mp 120–21° (Ref 4)

Other props & methods of prepn are found in Refs

N,N′-Di-(2(?)-nitrotolyl)-guanidine or **N,N′-Bis(2(?)-nitro-4-methyl-phenyl)-guanidine,** $[H_3C.C_6H_3(NO_2).NH]_2C{:}NH$; mw 329.31, N 21.27%; orn-red prisms (from alc), mp 197° (dec); sol in acids; sl sol in cold alkalies; insol in w. It was prepd by treating N,N′-Di-p-tolyl-guanidine with nitric acid (d1.5) and avoiding any rise in temp during nitration (Refs 5 & 6)

NOTE: No higher nitrated derivs of Ditolyl-guanidine were found in the literature thru 1966

Refs: 1) Beil **12**, 803 & [445] (ortho) 2) Beil **12**, (401) & [470] (meta) 3) Beil **12**, 943 & [513] (para) 4) Beil **12**, 943 5) Beil **12**, 1005 6) Perkin, JChemSoc **37**, 697 (1880) 7) CondChemDict (1961), 413-R (ortho)

Di(tolyl)-hydrazine, Hydrazotoluene or Dimethylhydrazobenzene and Derivatives

N,N′-Di(or Bis)(tolyl)-hydrazines or Hydrazotoluenes, $H_3C.C_6H_4{-}NH.NH{-}C_6H_4.CH_3$, mw 212.28, N 13.20%; four isomers are known:

N,N′-Di-o-tolyl-hydrazine, lflts, mp 165°; sol in eth & benz (Ref 1)

N,N′-Di-m-tolyl-hydrazine, colorless oil, fr p – not reported; v sl sol in alc (Ref 2)

N,N′-p-tolyl-hydrazine, crysts, mp 133–34°, bp – decomp (Ref 3)

N-m-Tolyl-N′-p-tolyl-hydrazine, pltlts (from petr eth), mp 74° (Ref 3)

Other props & methods of prepn are found in Refs

Dinitro-N,N′-ditolyl-hydrazine or **Dinitro-dimethyl-hydrazobenzene,** $C_{14}H_{14}N_4O_4$; mw 302.28, N 18.54%. Three isomers are known:

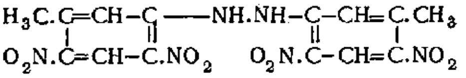
4,6-Dinitro-2,3′-dimethyl-hydrazobenzene, $H_3C.C_6H_2(NO_2)_2.NH.NH.C_6H_4.CH_3$, orn-red pltlts (from alc), mp 151–52° (dec); sol in alc, eth, benz, chlf & acet; sl sol in petr

eth; insol in w (Ref 4, p 153 & Ref 6)
4,6-Dinitro-3,3′-dimethyl-hydrazobenzene, orn-yel ndls (from alc), mp 165–66° (dec); sol in chlf, alc, acet & benz; sl sol in petr eth (Ref 4, p 153 & Ref 6)
4,6-Dinitro-3,4′-dimethyl-hydrazobenzene, orn-yel pltlts (from alc), mp 159–60° (dec); sol in chlf, alc, eth, acet & benz; sl sol in petr eth (Ref 4, p 234 & Ref 5)

Other props & methods of prepn are found in the Refs

Refs: 1) Beil **15**, 497, (147) & [223] 2) Beil **15**, 506 3) Beil **15**, 511 4) Beil **15**, (153) & [234] 5) M. Giua & A. Angelleti, Gazz **51** I, 324(1921) & CA **16**, 66(1922) 6) M. Giua & M. Giua, Gazz **53**, 171–72 (1923) & CA **17**, 2286(1923) 7) Dinitro derivs not found in later refs thru 1966

4,6,4′,6′-Tetranitro-di-(m-tolyl)-hydrazine; 1,2-Bis(4,6-dinitro-m-tolyl)-hydrazine (CA nomenclature); or **5,5′-Dimethyl-2,4,2′,4′-tetranitrohydrazobenzene** (Authors' name),



$$\begin{array}{l} H_3C.C{=}CH{-}C{-}{-}NH.NH{-}C{-}CH{=}C.CH_3 \\ O_2N.C{=}CH{-}C.NO_2 \qquad O_2N.C{-}CH{=}C.NO_2 \end{array}$$

mw 482.28, N 23.24%, orn-yel crysts (from acet ac), mp 244°; gives an intense purple color when dissolved in a soln of KOH (1%) in alc. It was obtd by heating 5-methyl-2,4-dinitrophenylhydrazine, 5-methyl-2,4-dinitrofluorobenzene & $CaCO_3$ in n-butanol for 6 hours (Ref 2)

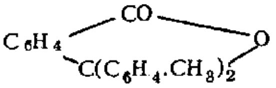
No expl props are reported

Refs: 1) Beil, not found 2) A.S. Bailey et al, Tetrahedron **22**(3), 996, 999(1966) & CA **64**, 17454(1966)

Hexanitro-ditolyl-hydrazine, $C_{14}H_{10}N_8O_{12}$, not found in the literature thru 1966

Di(tolyl)-phthalide and Derivatives

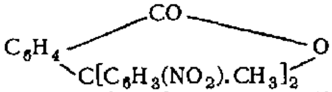
Di(or Bis)(tolyl-phthalide,



mw 314.36; exists as two isomers:
3,3-Di-o-tolyl-phthalide, crysts (from glac acet ac), mp 128–31° (Ref 1)
3,3-Di-p-tolyl-phthalide, crysts (from alc), mp 116.5–118° (Ref 2)

Other props & methods of prepn are found in Refs

3,3-Bis-(3-nitro-4-methylphenyl)-phthalide,



mw 404.36, N 6.93%, crysts (from alc), mp 157–58°; was prepd by nitrating 3,3-Di-p-tolyl-phthalide with nitric acid at –10° (Refs 3 & 6)

Tetranitro, $C_{22}H_{14}N_4O_{10}$, *and Hexanitro,* $C_{22}H_{12}N_6O_{14}$, *derivs not found in the literature thru 1966*

Octanitro-3,3-di-p-tolylphthalide, $C_{22}H_{10}O_2(NO_2)_8$, mw 674.36, N 15.87%; ndls, mp 289°; sol in warm Nitrobenz & acet; insol in alc, eth & benz. It was prepd by nitrating 3,3-Di-p-tolylphthalide with mixed nitric & sulfuric acids (Refs 4 & 5). No expl props are reported

Refs: 1) Beil **17**, [419] (ortho) 2) Beil **17**, 394 & [419] (para) 3) Beil **17**, 394 4) Beil **17**, 395 5) Limpricht, Ann **299**, 293(1898) 6) A. Beyer, Ann **354**, 184(1907) & CA **1**, 2698(1907) 7) Not found in later refs thru 1966

Di(tolyl)-propionic Acid and Derivatives

Di(or Bis)(tolyl)-propionic Acid, $C_{17}H_{18}O_2$; mw 254.31. Three isomers are known:
2,2-Di-p-tolylpropionic Acid, $(H_3C.C_6H_4)_2C(CH_3).COOH$, cubic crysts (from eth or alc) or monoclinic prisms, mp 151–52°, volatilizes w/o decompn; sol in eth, benz, chlf, carbon disulfide, glac acet acid & hot alc (Ref 1)
3,3-Di-p-tolylpropionic Acid, $(HC.C_6H_4)_2CH.CH_2.COOH$, crysts (from alc), mp 187–89° (Ref 2)
3,3-Di-o-tolylpropionic Acid, crysts (from benz), mp 133-34°; mod sol in alc & benz sl sol in petr eth; almost insol in w (Ref 2)

x,x-Dinitro-2,2-di-p-tolylpropionic Acid or *2,2-Bis(x-nitro-4-methylphenyl)-propionic Acid,* $[H_3C.C_6H_4(NO_2)]_2C(CH_3).COOH$, mw 344.31, N 8.14%, yel crysts (from glac acet ac), mp 129°(dec); sol in alc, eth & glac acet ac. It was prepd by nitrating the parent compd with aq nitric acid below 0°(Refs 3 & 6)

x,x,x,x-Tetranitro-2,2-di-p-tolylpropionic Acid, $[H_3C.C_6H_4(NO_2)_2]_2C(CH_3).COOH$, mw 434.31, N 12.90%, hydrated yel crysts (from alc) which decomp in the light, mp 223–25°(dec), mod sol in alc, eth, acet, chlf, carbon disulfide & glac acet ac. It was prepd by nitrating the parent compd with a mixt of 2 parts fuming nitric acid & 1 part concd sulfuric acid at 15° (Refs 4 & 5)

No expl props are reported

Refs: 1) Beil **9**, 687 & {3384} 2) Beil **9**, {3384} 3) Beil **9**, 688 & {3384} 4) Beil **9**, 688 5) Haiss, Ber **15**, 1478(1882) 6) R.B. Holmes & A.J. Hill, USP 2423025(1942) & CA **41**, 6285(1947) 7) Not found in later refs thru 1966

Hexanitro-ditolyl-propionic Acid, $C_{17}H_{12}N_6O_4$, not found in the literature thru 1966

Di(tolyl)-sulfide or Dimethyl-diphenylsulfide and Derivatives

Di(tolyl)-sulfide, $(H_3C.C_6H_4)_2S$, mw 214.32. Several isomers are known:
Di-o-tolyl-sulfide, tablets (from alc), mp 64°, bp 285°(Ref 1)
Di-m-tolyl-sulfide, yel oil, bp 175° at 15mm & 290° at 760mm (Ref 2)
Di-p-tolyl-sulfide, ndls (from alc), mp 56.6–57°, bp 175° at 16mm (Ref 3)
Di-o,m-tolyl-sulfide, odorless oil, bp 170° at 11mm (Ref 4)
Di-o,p-tolyl-sulfide, liq, bp 173° at 11mm, sp gr 1.0774 at 15/4°(Ref 5)
Di-m,p-tolyl-sulfide, liq, fr p 28°(Ref 6)

Other props & methods of prepn are found in the Refs

Dinitro-ditolyl-sulfide; Dinitrodimethyl-diphenylsulfide or **Bis(nitromethylphenyl)-sulfide,** $[H_3C.C_6H_3(NO_2)]_2S$; mw 304.33, N 9.21%. Several isomers are known:
6,6'-Dinitro-2,2'-dimethyl-diphenyl-sulfide, yel prisms (from benz), mp 150° or yel crysts (from alc), mp 152°(Ref 7, pp 343 & 1284)
4,4'-Dinitro-3,3'-dimethyl-diphenyl-sulfide, pale-yel crysts (from glac acet ac), mp 164°; insol in hot aq soln of Na_2S or Na_2S_2 (Ref 7, p 1339)
2,2'-Dinitro-4,4'-dimethyl-diphenyl-sulfide, dk-red prisms (from acet), mp 125–26°(Ref 7, p401)
6,2'-Dinitro-3,4'-dimethyl-diphenyl-sulfide, pale yel pltlts (from aq acet ac), mp 131–32° (Ref 7, p1438)

Other props & methods of prepn are found in the Refs

Trinitro-ditolylsulfide – not found in the literature thru 1966

Tetranitro-ditolyl-sulfide, Dinitrodimethyl-diphenylsulfide or **Bis(dinitromethylphenyl)-sulfide,** $H_3C.C_6H_2(NO_2)_2S.C_6H_2(NO_2)_2CH_3$; mw 394.32, N 14.21%. Several isomers exist:
Bis(4,6-dinitro-2-methyl-phenyl) -sulfide, yel crysts, mp 210°; unchanged by warming in nitric acid; was prepd by reaction of 3,5-dinitro-2-bromotoluene with Na_2S (Ref 8, p373)
2,6,2',6'-Tetranitro-3,3'-dimethyl-diphenyl-sulfide, yel crysts (from glac acet ac), mp 222°; mod sol in chlf, benz, acet & glac acet ac; almost insol in alc, eth, carbon disulfide & petr eth. It can be prepd from equimolar mixt of 2,3,4-trinitrotoluene & thiosemicarbazide in hot alc (Ref 8, p367) & Ref 10)
4,6,4',6'-Tetranitro-3,3'-dimethyl-diphenyl-sulfide, lt-yel prisms (from xylene), mp 189–90°; sol in hot alc, acet & xylene; diffc sol in other org solvs. It was obtd with other products by reaction of 2,4,5-trinitrotoluene & thiourea or allylthiourea in hot alc (Ref 8, p367); Refs 9 & 12
Bis(2,6-dinitro-4-tolyl)-sulfide, yel crysts (from chlf + petr eth), mp 274–75°; prepd from reaction of a mixt of N-(4-methyl-2,6-dinitrophenyl) -pyridinium toluene-p-sulfonate, thioacetic acid, dry chlf & dry pyridine refluxed for 2 hrs (Ref 11)

No expl props of the tetranitro derivs were reported

Hexanitro-ditolylsulfide or **Bis(2,4,6-trinitro-3-tolyl)-sulfide,** $H_3C.C_6H(NO_2)_3S.C_6H(NO_2)_3.CH_3$; mw 484.32, N 17.35%; crysts (from acet ac), mp 170°;

was prepd by refluxing for 1 hr 1-chloro-2,4,6-trinitro-3-toluene with thiourea in 95% alc (Ref 12). No expl props were reported

Refs: 1) Beil **6**, 371, [342] & {1280} 2) Beil **6**, 388 & [366] 3) Beil **6**, 419, (209), [395] & {1405} 4) Beil **6**, 388 5) Beil **6**, 418 6) Beil **6**, 418 & {1404} 7) Beil **6**, [343, 401] & {1284, 1339, 1438} 8) Beil **6**, 373 & [367] 9) M. Giua & A. Ruggeri, Gazz **53**, 294(1923) & CA **17**, 3865 (1923 10) M. Giua & R. Petronio, Gazz **55**, 671(1925) & CA **20**, 1062(1926) 11) J.H. Barnes et al, JChemSoc **1953**, 1455 & CA **48**, 5151(1954) 12) S.S. Joshi & T.C. Mathur, JIndianChemSoc **40**, (11), 939(1963) & CA **60**, 5376(1964)

Di(tolyl)-triazenes and Derivatives

Di(or Bis)(tolyl)-triazenes

$H_3C.C_6H_4-NH.N:N-C_6H_4.CH_3$, mw 225.28, N 18.65%, exists in six isomers:

1,3-Di-(o-tolyl)-triazene, orn-yel crysts, mp 51°. It was first prepd in 1887 by Fischer & Wimmer (Ref 1, p703 & Ref 2) by reaction of 2 moles o-toluidine, 3 moles aq HCl & 1 mole $NaNO_2$ soln in the presence of Na acetate and cooling. ADL (Ref 9) describes its method of prepn and gives heat of combstn Q_c=1914kcal/mol at C_v & water liq

1,3-Di-(m-tolyl)-triazene, yel ndls (from petr eth), mp 50–52°; mod sol in org solvs. It was prepd by treating m-toluenediazonium nitrate with a cooled soln of m-toluidine in pyridine (Ref 1, pp 705, 407)

1,3-Di-(p-tolyl)-triazene, lt-yel ndls (from petr eth) or lt-yel, almost colorless prisms (from alc amm sulfide), mp 115–116.5°; can be prepd by introducing N gas into an alc-eth soln of p-toluidine and by other methods (Ref 1, pp709, 407, 355). See also Refs 3, 4, 5, 6 & 7

Gave on treatment with nitrogen oxide an explosive product, sensitive to impact & heat (Ref 8)

2,3′-Di-(o,m′-tolyl)-triazene, pure-yel crysts (from petr eth), mp 74°; stable in light; was obtd with other products on treating o-toluenediazonium nitrate with a cooled soln of m-toluidine in pyridine (Ref 1, p705)

2,4′-Di-(o,p′-tolyl)-triazene, lt-yel ndls (from petr eth), mp 119–20°; was prepd by reaction of o-toluenediazoniumchloride & p-toluidine hydrochloride in the presence of Na acetate (Ref 1, p708)

3,4′-Di-(m,p′-tolyl)-triazene, lt-yel ndls (from petr eth), mp 96–97°; mod sol in hot org solvs; less sol in cold; was prepd from m-toluenediazoniumchloride & p-toluidine hydrochloride in the presence of Na acetate or Na dicarbonate (Ref 1, p708)

Refs: 1) Beil **16**, 703, 705, 708, 709, (407) & [355] 2) B. Fischer & H. Wimmer, Ber **20**, 1582(1887) 3) G.T. Morgan & L.P. Walls, JChemSoc **1930**, 1502 & CA **24**, 4280(1930) 4) F.P.J. Dwyer et al, JProcRoySoc, N.S.Wales **66**, 315(1932) & CA **27**, 1331(1933) 5) L. Hunter, JChemSoc **1937**, 320 & CA **31**, 3019(1937) 6) F.P.J. Dwyer, JSCI **56**, 70(1937) & CA **31**, 3460(1937) 7) R.J.W. Le Fèvre & H. Vine, JChemSoc **1937**, 1805 & CA **32**, 1532(1938) (Dipole moment) 8) T.W. Campbell, JACS **73**, 4019(1951) & CA **46**, 7574(1952) 9) ADL, Synth HE's, 3rd Rept (1953), 237, 422

Dinitro-ditolyl-triazene or **Dinitro-dimethyl-diazoaminobenzene**,

$H_3C.C_6H_3(NO_2)-NH.N:N-C_6H_3(NO_2).CH_3$; mw 315.28, N 22.22%. Four isomers are found in Beil:

5,5′-Dinitro-2,2′-dimethyl-diazoaminobenzene, yel ndls (from alc or glac acet ac), mp 212° (dec); sol in chlf, acet & benz; sl sol in alc; insol in w. It was prepd by treating a soln of 4-nitro-2-aminotoluene in glac acet ac at 0° with a soln of Na nitrite (Ref 1, p703 & Ref 3)

4,4′-Dinitro-2,2′-dimethyl-diazoaminobenzene, golden-yel crysts (from acet), mp ca 237°; prepd by treating 5-nitro-2-aminotoluene in glac acet ac with a Na nitrite soln (Ref 1, p704 & Ref 5)

3,3′-Dinitro-2,2′-dimethyl-diazoaminobenzene, yel ndls, mp 191°; prepd similarly as above compd (Ref 1, p704 & Ref 4)

2,2′-Dinitro-4,4′-dimethyl-diazoaminobenzene, dk-red-brn ndls (from alc), mp 163° (dec); v sl sol in hot alc; sol in amyl alc, eth, carbon disulfide, chlf, acet & benz. It was prepd by treating 3-nitro-4-aminotoluene in

alc with nitric acid (d 1.52) & K nitrite soln (Ref 1, p711 & Ref 2)
Refs: 1) Beil **16**, 703, 704,711 2) Niementowski, Ber **22**, 2565(1889) 3) Witt et al, Ber **25**, 3155(1892) 4) Noelting, Ber **37**, 2583(1904) 5) Meunier, BullFr [3] **31**, 641(1904) 6) Not found in later Refs thru 1966

NOTE: No higher nitrated derivs were found in the literature thru 1966

Di(tolyl)-trimethylene-diamine. See Bis-(methylphenylamino)-propane in Vol 2, p B151-L

N,N′-Di(tolyl) -ureas and Derivatives
Di(or Bis)(tolyl)-ureas,
$CH_3.C_6H_4-NH.CO.NH-C_6H_4.CH_3$, mw 240.29, N 11.66%; exists in four isomers:
N,N′-Di-o-tolyl-urea, ndls, mp 255–56°; insol in w; sl sol in hot alc (Ref 1)
N,N′-Di-m-tolyl-urea, ndls, mp 223–25°; insol in w; sol in hot alc (Ref 2)
N,N′-Di-p-tolyl-urea, crysts, mp 266–68°; insol in w; sl sol in alc & eth (Ref 3)

Accdg to Davis (Ref 5), 40 parts of para isomer dissolved in benz are required for complete gelatinization of 100 parts of Pyrocellulose
N-o-Tolyl-N′-p-tolyl-urea, ndls (from alc), mp 263–64°; v sl sol in hot alc (Ref 4)

Other props & methods of prepn are found in the Refs
Refs: 1) Beil **12**, 801, (382) & [444] 2) Beil **12**, 863 & [470] 3) Beil **12**, 941, (425) & [512] 4) Beil **12**, 941 5) Davis (1943), 322

Dinitro-ditolyl-urea; Bis(nitrotolyl)-urea; Bis(nitromethylphenyl)-urea or **Dinitro-dimethyldiphenyl-urea,**
$CH_3.C_6H_3(NO_2).NH.CO.NH.C_6H_3(NO_2).CH_3$; mw 330.29, N 16.96%. Several isomers exist:
N,N′-Bis(5-nitro-2-methylphenyl)-urea, ndls, (from glac acet ac), mp 300–05° (sublimation); insol in benz, eth & chlf; sl sol in alc; sol in glac acet ac. It was prepd by heating 4-nitro-2-aminotoluene & phosgene in toluene in a tube at 130° (Ref 1, p846 & Ref 2, p662)
N,N′-Bis(4-nitro-2-methylphenyl)-urea, ndls (from glac acet ac), mp 305–10° (sublimation); insol in w, eth, chlf & benz; sl sol in alc; sol in glac acet ac. It was prepd by heating 5-nitro-2-aminotoluene & phosgene in toluene in a tube at 130° (Ref 1, p847–48 & Ref 2, p659
N,N′-Bis(3-nitro-4-methylphenyl)-urea, yel prisms (from ethyl acet), mp 251–52°; sl sol in eth & benz; sol in hot concd acetic acid & hot alc. It was prepd by heating 2-nitro-4-aminotoluene with urethane at 210–20° (Ref 1, p999 & Ref 2, p663)
N,N′-Bis(2-nitro-4-methylphenyl)-urea, yel ndls (from glac acet ac), mp 244–45°; easily sublimes; sol in alc & benz; insol in w. It was prepd by heating 3-nitro-4-aminotoluene with carbonyl chloride in toluene at 130° (Ref 1, p 1004–05 & Ref 2, p661)
Refs: 1) Beil **12**, 846, 847, 999, 1004 2) Vittenet, BullFr [3], **21**, 659, 661, 662, 663(1899) 3) Not found in later Refs thru 1966

Tetranitro, $C_{15}H_{12}N_6O_9$, and *Hexanitro,* $C_{15}H_{10}N_8O_{13}$, derivs of Di(tolyl)-urea were not found in the literature thru 1966

Di(triazacyclohexylmethyl)-ether. See Bis(1,3,5-triaza-3,5-dinitro-cyclohexylmethyl)-ether in Vol 2, pB159-L

Ditriazole [called Di[1.2.4-triazolyl]-(3.3′), in Ger]. See Bis(β-sym or β-1,2,4-triazole) in Vol 2, p B159-R

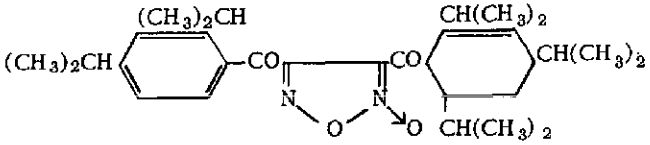
3,3′-Ditriazolyl-formamidine. See N,N′-Bis(s-triazol-3-yl)-formamidine in Vol 2, p B159-R

Ditriazo-mesidine. See 2-Amino-4,6-diazidomesitylene in Vol 1, p A224-L, under AMINOMESITYLENES AND DERIVATIVES

Di-triethyl Lead Azoaminotetrazole or **Bis-triethyl Lead Azoaminotetrazole.** It is described in Vol 2 of Encycl on p B158-R as 1,3-Bis(1H-tetrazolyl-5)-triazene, Ditriethyl Lead Salt, $C_{14}H_{31}N_{11}Pb_2$, mw 767.90, N 20.07%
Note: Its mw is shown erroneously on p B158-R as 767.81 and N as 20.06%

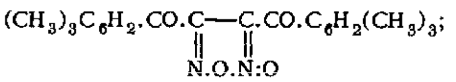
Di(triethyl) Lead Styphnate. See Bis-(triethyl) Lead Styphnate in Vol 2, p B160-L

Di(2,4,6-triisopropylbenzoyl)-furoxan.
Note: It was listed in Vol 2, p B160-L as Bis(2,4,6-triisopropylbenzoyl)-furoxan, but its properties and prepn were not described because the ADL reports listed here as Refs 2 & 3 were classified at that time. They are now declassified



mw 546.72, N 5.12%; wh crysts (from eth), mp 128° (dec); sol in eth & acet; insol in w. It was prepd by treating 2,4,6-tri-isopropylacetophenone in glac acet ac with concd nitric acid & aq Na nitrite soln at 60° (Refs 2 & 3)
Refs: 1) Beil, not found 2) ADL Synthesis HE's, 3rd Rept (1953), p384 3) Ibid, 4th Rept (1956), p57

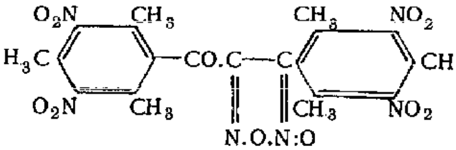
Di(trimethylbenzoyl)-furoxan and Derivatives
Di(2,4,6-trimethylbenzoyl)-furoxan.
Note: It was listed in Vol 2, p B160-L as Bis(2,4,6-trimethylbenzoyl)-furoxan, but its props and prepn were not described because the ADL rept listed here as Ref 2, was classfied



$$(CH_3)_3C_6H_2.CO.\underset{\|}{C}—\underset{\|}{C}.CO.C_6H_2(CH_3)_3;\quad N.O.N{:}O$$

mw 378.41, N 7.40%; yel ndls (from eth), mp 141–45°; prepd by warming 2,4,6-trimethylacetophenone with concd nitric acid (Refs 1 & 2)

Di(3,5-dinitro-2,4,6-trimethylbenzoyl)-furoxan or **Bis(3,5-dinitro-2,4,6-trimethylbenzoyl)-furoxan,**



mw 558.41, N 15.05%; wh crysts (from chlf); mp 235°; sol in Nitrobenz, acet & 2-nitropropane; mod sol in chlf; sl sol in alc & MeOH; v sl sol in eth, glac acet ac & concd nitric acid; insol in water. Its heat of combstn is 2655kcal/mol at const vol with water liq. The parent compd was nitrated with fuming nitric acid (d 1.52) below 0° & sulfuric acid-water soln added dropwise at –5°, and the reaction mixt poured into ice water. The yield was 81%
Refs: 1) Beil **27**, 686 2) ADL Synthesis HE's, 3rd Rept (1953), pp376, 378 & 421

Di(or Bis) (2,4,6-trimethylpyridino)-diazido-copper (II), $[Cu(C_8H_{11}N)_2](N_3)_2$, mw 389.94, N 28.74%; greenish-brown crysts, mp – explodes on heating to 198–202° or when dropped on metallic block preheated to 220–25°; does not explode from impact. It can be prepd by dissolving Cu azide in trimethylpyridine soln, or by addg NaN_3 to the Cu^{++} salt of trimethylpyridine (Ref 2)
Refs: 1) Beil, not found 2) A. Cirulis & M. Straumanis, JPraktChem **162**, 312(1943) & CA **38**, 1969(1944)

Di(or Bis) (trinitrobutyl)-urea. See under Di-(butyl)-urea in this Vol. This and the next compd were listed in Vol 2, p B160-R but not described because they were considered classified

Di(trinitro-*iso*-butyl)-urea. See under Di-(iso-butyl)-urea in this Vol

Di(or Bis) (2,2,2-trinitroethyl)-amine. See under Diethyl-amine in this Vol

1,2-Di(or Bis)(trinitroethylamino)-benzene. See under 1,2-Di(ethylamino)-benzene in this Vol

1,4-Di(or Bis)[N(2′,2′,2′-trinitroethyl)-carboxamide]-piperazine. See under Diethyl-carboxamide-piperazine in this Vol. It was also listed in Vol 2, p B160-R, but its formula was wrong because it contained the expression $-\ CO(NO_2)_3$ instead of $-\ C(NO_2)_3$

Di(or Bis)(2,2,2-trinitroethyl)-hydroxylamine. See under Di(ethyl)-hydroxylamine in this Vol

Di(or Bis)(2,2,2-trinitroethyl)-nitramine, (Code named **HOX**). See under Diethylamine in this Vol

Di(or Bis)(2,2,2-trinitroethyl)-urea. See under Diethyl-urea in this Vol

N,N′-Di(or Bis)(2,4,6-trinitrophenyl)-ethylene-dinitramine or **N,N′-Di(or Bis)-(2,4,6-trinitrophenyl-nitramino)-ethane** (Code named **Bitetryl, Ditetryl or Octyl**). See under Bis(anilino)-ethane in Vol 2, p B131-R

1,3-Di(or Bis)(2′,4′,6′-trinitrophenyl-propylene)-dinitramine; 1,3-Di(or Bis)-2′,4′,6′-trinitrophenyl-nitramino)-propane or **N,N′-(Hexanitrodiphenyl)-propylene-1,3-dinitramine**(Code named **Methylene Ditetryl**). See 1,3-Bis(2′,4′,6′-trinitro-N-nitranilino)-propane in Vol 2, p B133-L

Di(trinitrophenyl)-urea. See N,N′-Bis-(2,4,6-trinitrophenyl)-urea or Dipicryl-urea in Vol 2 of Encycl, p B156-L

Dittman Dynamites (Brit). Blasting expls patented in England in 1867 which consisted of finely pulverized charcoal (previously treated with concd soln of K or Na nitrate and dried) serving, together with sawdust (or its partly nitrated product), as absorbents for NG
Ref: Daniel (1902), 207

Dittmar, C. F. (1837–1883). German officer and expls specialist who worked for a while with A. Nobel in Sweden and then immigrated to the USA, where he established the first Dynamite plant on the Amer continent at Neponset, Massachusetts. He invented, before coming to the US, *Dualine* and some other expls, listed under next item

After DittmarJs death, his wife, Maria, successfully continued to manage the business until about 1903
Ref: Van Gelder & Schlatter (1927), 415 & 621

Dittmar's Dynamites. While working for A. Nobel in Sweden, Dittmar patented, beginning in 1867, the following Explosives:
Dualin (See in this Vol) ;
Glucodines: a) NG 33.2, NSu 3.2, sugar 8.4, Na nitrate 31.2, NC 23.4 & unaccounted 0.6%. b) NG 30.2, NSu 4.0, sugar 8.9, Na nitrate 37.9, NC with charcoal 19.3 & unaccounted 0.7% (Ref, p345)
Titan. An expl prepd by impregnating vegetable fibers with a soln of sugar and mannite in amylene, C_5H_{10}. Amylene is a colorless transparent liquid obtd by dehydration of amylic alcohol (Ref, p768) and
Xyloglodine. An expl prepd by nitrating a mixture of glycerin, starch, cellulose and mannite (Ref, p812)
Ref: Daniel (1902), 208, 345, 768 & 812

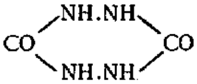
Di(tri-fluoromethylthio)-fluorobutane or **Bis(tri-fluoromethylthio)-fluorobutane,** $CF_3SCF_2{-}CF_2SCF_3$; mw 302.17; liq, bp 80–81°; was obtd when bis(trifluoromethyl)-disulfide was refluxed while tetrafluoroethylene gas was concurrently passed into the reaction zone, and the mixt irradiated with UV light yielding a main fraction of 39.3%

This compd & other fluorobutanes are useful as thermally stable dielectric coolants & insecticidal fumigants
Refs: 1) Beil, not found 2) E.E. Gilbert & R.E.A. Dear (Ciba-Geigy Corp), USP 3646221(1972)

Di(uranate) Potassium, $K_2U_2O_7$, mw 666.34, or $K_2U_2O_7.3H_2O$; yel salt; insol in water. It can be prepd as a trihydrate salt by precipitating a uranyl salt with KOH and drying the product at 100°

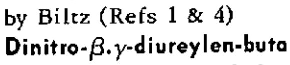
It was proposed for use in smoke compositions. Eg: K diuranate 4, K ClO_4 4, dextrin 5 & Amm hydrogen fluoride (NH_4HF_2) 4 parts yielded a heavy grayish smoke
Ref: J. DeMent, USP 2995526(1961), p22 & CA 55, 25100(1961)

Diurea (Dihamstoff, in Ger) or **p-Urazine,**



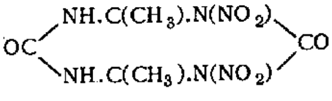
; mw 116.08, N 48.27%. This structure was assigned by Curtius & Heidenreich to the compd described in Vol 1, p A472-L as **4-Amino-urazole**
Refs: Same as given in Vol 1, p A472-L

β,γ-Diureylen-butan. German name for the compd listed in foreign literature as *2.2′-Dioxo-4.5-dimethyl-hexahydro-[imidazolo-4′.5-imidazol]* β.γ and *7.8-Dimethyl-acetylendiurein,*



- mw 170.17, N 32.93%; ndls or pltlts (from water); mp — dec 350–55° with frothing; sol in hot w; insol in eth, chlf & benz and very diffc sol in boiling alc. It was prepd by Franchimont & Klobbie (Refs 1 & 3) by allowing to stand diacetyl and urea in water. It was also prepd by Biltz (Refs 1 & 4)

Dinitro-β.γ-diureylen-butan, called in Beil *1.3-Dinitro-7.8-dimethyl-acetylendiurein,*



mw 260.17, N 32.31%; colorless crysts, mp — darkens at 230°; diffic sol in w & alc; nearly insol in eth, chlf & benz. It was prepd by Franchimont & Klobbie (Refs 2 & 3) by allowing to stand 1 part of β.γ-diureylen-butan with 5 parts abs nitric acid

Its expl props were not reported
Refs: 1) Beil **26**, 445 & (131) (Parent) 2) Beil **26**, 445 (Dinitro) 3) A.P.N. Franchimont & (?) Klobbie, Rec **7**, 251(1888)(Parent) and p253 (Dinitro) 4) H. Biltz, Ber **40**, 4811(1907) (Parent) and **41**, 1882(1908) (Parent)

Dive Bombing. This type of bombing, which preceded *skip bombing* (See below), is an effort to improve its accuracy. Aiming his plane at the target, the pilot plunges downward at a steep angle (not over 70°), opens his airbrakes as the bomb is released, and pulls out of the dive upward immediately. The velocity of the bomb is exactly the same as that of the plane at the instant of release. Dive bombing is particularly effective against slow moving targets but such planes may be knocked out of the air by fast fighters and massed AA fire (Refs 1 & 2). *Skip bombing* is by releasing one or more bombs from a plane flying at a low altitude, so that the bomb or bombs glance off the surface of the ground or water and strike the target (Ref 3, p 265-L)

Other methods of bombing are:
Glide bombing. The action of bombing a target from a plane descending at a normal or nearly normal angle of attack with little or no thrust (Ref 3, p136-L)
Laser-guided bombing. A guidance concept devised & developed by MICOM (US Army Missile Command, Redstone Arsenal, Huntsville, Alabama) scientists applicable to bombs, rockets, missiles & artillery shells. The Army technology & experimental hardware were subsequently used by the Air Force to develop their laser-guided "smart bomb" Army & Navy laser-guided missiles have demonstrated unprecedented accuracy in dozens of applications (Ref 3)
Low-angle bombing. The action of bombing at a slight dive angle. It is actually a species

of dive bombing (Ref 2, p176-L)
Toss bombing. A bombing action that involves the use of centrifugal force to carry the bomb dropped away from the plane (Ref 2, p297-L)
Refs: 1) Newman (1943), 318 2) Ohart (1946), 199–200 3) Glossary of Ord (1959), 99, 136, 176 & 297 4) US Army R&D Magazine, Vol **13**, No 6, pp 5, 32–33 (Sept 1972)

Dive Machine-Gunning. The action of firing machine-guns or automatic cannons against personnel or low ground targets from an aircraft rapidly descending steeply nose-downward and then, when only a few feet above the ground, continuing the flight, firing, nearly parallel to the ground

This technique, for example, was successfully used by US Mustangs during the destruction of Dresden in February 1945 when about 135 thousand were killed and many more wounded
Ref: D. Irving, "Destruction of Dresden", Ballantine, NY (1965)

Diver. US Navy guided air-to-surface missile, jet-powered and based on a 1000lb torpedo. It is a member of the "Kingfisher" family which are essentially radar-controlled winged torpedoes
Ref: F. Ordway & R. Wakefield, "International Missile and Spacecraft Guide", McGraw-Hill, NY (1960), 118

Diver's Liquid or Diver's Solution. It is a soln of AN in liquid ammonia, the ammonia being recovered and reused. Davis et al (Ref 2) disclosed the formulation of AN-fuel mixts w/wo expl sensitizers by effecting a very intimate mixt in Diver's soln. The Davis process produces mixts which will propagate satisfactorily in small-diam chges. They are soft & pliable and also propagate in regular dynamite cartridges. However, the mixts have no moisture resistance and are not easily waterproofed. Hence, they require watertight containers
Refs: 1) BritP 15852(1905) 2) C.O. Davis, USP 2168562(1939) & CA **33**, 9648 (1939) 3) J. Whetston & J. Taylor, BritP 597717(1945) & BrAbstracts BI(1948), p 348 4) USP 2393594(1946) 5) Cook (1958), 13 6) J. Taylor, "Solid Propellant and Exothermic Compositions", Interscience, NY (1959), p77

Dividing Wall in Ordnance Storage Magazines. Safe distances between the magazines, distances from inhabited buildings, passenger railroads and public highways are calculated depending on the amount of explosives stored in each magazine, employing "American Table of Distances for Storage of Explosives", such as given in DuPont's Blasters' Hdb (1966), p458. For example, for barricaded one-room magazines, each holding 4500lbs of explosives, the separation should be not less than 61ft. In order to be able to place more magazines on a limited space, this distance can be shortened if magazine will be subdivided into two compartments by the so-called "dividing wall". This wall must be of concrete, at least 12 inches thick, reinforced at both sides by iron rods, at least 1/2 inch in diameter and placed in parallel rows, at least 12 inches apart, both horizontally and vertically. Safe distances between such subdivided magazines are not based on the total amount of explosives in each magazine, but just on the largest amount stored in one of the sections

For example, if the magazine is subdivided into a section holding 2500lbs and another one holding 2000lbs, separation of magazines will be based on the amount of 2500lbs and this is 52 ft, accdg to the table

Such arrangement is possible because it is assumed that if one of the sections will detonate, the dividing wall will protect the other section from sympathetic detonation
Refs: Anon, "Ammunition Inspection Guide", WarDeptTechManual, **TM 9-1904**(1944), 800-01 2) Anon, "Safety Manual", **AMCR 385-100** (1970), 2–11 & 5–3, US Army Materiel Command, Washington, DC 20315

Divine's Explosives. American chemist, S.R. Divine, patented in GtBritain in 1881, 1882 & 1883 powerful expls consisting of K chlorate with NB (Nitrobenzene), or with other combustible oils. Of these expls, the best known was **Rack-a-rock** because it was used for blasting out Hell Gate Channel in New York Harbor. On Oct 10, 1885, 240399lbs of Rack-a-rock, along with 42331lbs of Dynamite, were exploded for this purpose in a single blast. Divine's expl was prepd just before its use by adding 21 parts of NB to 79 parts of K chlorate contained in water-tight copper cartridges (Ref 6, p354)

Accdg to Pérez Ara (Ref 7), explosivo de Divine, "Rack-a-rock" o "Break-hills" was in the form of a paste consisting of K chlorate 23.5 and NB 76.5%. It was prepd just before use and loaded into paraffined paper cartridges

Other Divine expls listed in Ref 7 are:
a) K chlorate 83.4, NB 8.3 & petroleum oil 8.3%
b) K chlorate 87.5 & kerosene 12.5%
c) K chlorate 89.5 & turpentine 10.7%

Sulfur was incorporated in Divine expls when it was desired to diminish their brisance, while PA (Picric Acid) was added to increase their brisance

Divine expls were extensively used at the end of the 19th century, not only in the US but also in some foreign countries, such as China where they were used in construction of railraods. In Cuba, an expl known as "rompe-roca" consisted of K chlorate, NB with a small amt of red lead and red dye (Ref 7, pp231–33)

An explosive similar to the above is listed by Giua (Ref 8, p398-L) as "esplosivo della Rendrock Powder Co" (1904): K chlorate 80, resin 18, asphalt 1 & nitrated wood pulp 1%

Gody (Ref 3, p576) listed as "explosif Divine" (1895) a mixture of DNB, DNT & Pb nitrate, w/o giving their percentages. He also listed (p265) under "Divine Rack-a-rocks" several different compns, such as: a) K chlorate 75–80 & NB 25–20%; b) K chlorate 78 and a mixture of tar oil with CCl_4 22% and c) K chlorate 80.5, NB 18.0 & sulfur 1.5%

Compositions of Divine Explosives were also listed in Refs 1, 2, 4 & 5

Divine explosives are sometimes called "Sprengel Type Explosives" because they are based on K chlorate, one of the chlorates patented in 1871 by H. Sprengel for use in his expls (Ref 6, p353)

A fairly complete list of various chlorate expls is given in Vol 2 of Encycl, pp C202 to C207

The most important group of chlorate expls is listed under "Cheddites" or "Street Explosives". They are described in Vol 2, pp C155 to C160

Refs: 1) Cundill (1889). French translation in MP **6**(1892), p76 2) Daniel (1902), p207 3) Gody (1907), 265 & 476 4) Marshall **1** (1917), 46 5) Colver (1918), 135 (Rack-a-Rock) 6) Davis (1943), 354 6a) Clift & Fedoroff **2** (1943), p R1 7) Pérez Ara (1945), 231–33 8) Giua, Trattato **VI** (1) (1959), 393

Divine's Explosive Rack-a-Rock. See in previous item

Divinyl or Bivinyl. See 1,3-Butadiene in Vol 2 of Encycl, p B365-R

Divinylacetylene, DVA or **1,5-Hexadien-3-yne,** $H_2C{:}CH.C\vdots C.CH{:}CH_2$; mw 78.11; oily liq, sp gr 0.785, bp – decomp at 83.5°. It can be prepd by passing acetylene into ammoniacal cuprous chloride. It is a basis for synthetic butyl rubber (Refs 1 & 4)

Accdg to Nieuwland et al (Ref 2), DVA rapidly absorbs oxygen from the air, forming a highly expl peroxide (ozonide) which has been found to detonate spontaneously with violence upon standing. In the pure state, DVA may be stabilized for storage by the addn of "antioxidants", such as hydroquinone, catechol, dibutylamine, phenyl-α-naphthylamine, etc

Accdg to Cupery & Carothers (Ref 3), when DVA is allowed to stand with a little air, it changes to an oxygen-containing jelly which is dangerously explosive

Accdg to Schildknecht (Ref 5), when

DVA is heated in absence of air, it polymerizes to give liquid low polymers which harden in air to cross-linked resins. Heating of DVA in presence of air usually causes explosions

Note: Must not be confused with *Vinyl Acetylene*, $H_2C:CH.C\vdots CH$, described in Ref 6, p 1210-L

Refs: 1) Beil **1**, [247] & {1058} 2) J.A. Nieuwland et al, JACS **53**, 4197 & 4200 (1931) 3) M.E. Cupery & W.H. Carothers, JACS **56**, 1167(1934) 4) Hackh's (1944), 285-R 5) C.E. Schildknecht, "Vinyl and Related Polymers", Wiley, NY (1952), 702 6) CondChemDict (1961), 414-L (States that DVA is prepd by passing acetylene into a hydrochloric acid soln contg metallic catalysts)

Divinylbenzene (DVB) or **Vinylstyrene,**

$H_2C:CH.C_6H_4.CH:CH_2$; mw 130.18; exists as o-,m- and p-isomers. The commercial form contains the 3 isomers together with ethylvinylbenzene and diethylbenzene

Divinyl-m-benzene, wh liq, easily polymerized; sp gr 0.9289 at 20°; fr p minus 66.9°, bp 199.5°. It can be prepd (with other products) by heating 1,3-diethylbenzene or 3-ethyl-1-vinylbenzene & superheated steam passed over a bed of activated charcoal at 700° (Ref 1, p 1367 & Ref 2) and by other methods

It is a highly reactive compd, resembling in some props DVA. Polymerization, once started, may proceed with violence

Used as a polymerization monomer for some synthetic rubbers

Note: Must not be confused with *Vinylbenzene*, described as *styrene* in Ref 3, p 1083-L

Refs: 1) Beil **5**, 518, [414] & {1366–67} 2) R.R. Dreisbach, USP 2385696(1945) & CA **40**, 602(1946) 3) CondChemDict (1961), 414-L

Divinyl Ether, Divinyl Oxide (DVO), Vinyl Ether or **Ethenoxyethene,**

$H_2C:CH.O.CH:CH_2$; mw 70.09; colorless liquid, sp gr 0.773 at 20/20°, bp 28.3°; fl p below minus 22°; n_D 1.3989 at 20°; highly volatile, giving off at comparatively low temps vapors which form flammable, explosive mixtures with air or oxygen; v sl sol in w; sol in alc and v sol in eth. It can be prepd by interaction of β,β'-dichlorodiethyl-ether with caustic alkali in the presence of ammonia. DVO must be protected from light. Its explosive props were investigated by Jones & Beattie (Ref 3)

Investigation at the US Rubber Co lab showed that DVO reacted with nitroform in methanol to give a liq, bp 50° at 0.8mm, n_D 1.4417 at 20°, which burned vigorously and could be detonated by a hammer blow (Ref 4)

DVO is used as an anesthetic (Ref 5)

Refs: 1) Beil **1**, 433, [473] & {1864} 2) W. Ruigh & R. Major, JACS **53**, 2662(1931) 3) G. Jones & B. Beattie, IEC **26**, 557(1934) 4) USRubberCo Rept **No 5** (1948–1949), p14 5) CondChemDict (1961), 1211-L (Vinyl ether – a misnomer)

Dixylyl or **Tetramethyl-biphenyl.** See Bixylyl in Vol 2, p B164-R

Dixylylamine and Derivatives

sym-Dixylyl-amine or *Tetramethyl-diphenyl-amine*, $(CH_3)_2C_6H_3.NH.C_6H_3(CH_3)_2$; mw 225.32, N 6.22%. Two isomers are found in Beil:

2,4,2',4'-Tetramethyl-diphenylamine, prisms, (from alc), mp 58–58.5°; insol in w, alkalies & acids; was prepd by treating 4-azido-m-xylene with alcoholic sulfuric acid in the cold (Ref 1)

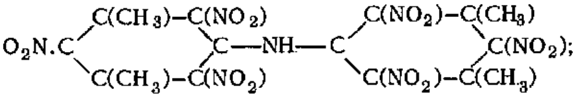
3,4,3',4'-Tetramethyl-diphenylamine, thick oil, bp – partially decomp 340–45°; prepd from asym o-xylene, bromo-Zn-ammonia & Amm bromide heated in a tube at 300–310° (Ref 2)

Azido, $C_{16}H_{17}N_4$, *Diazido*, $C_{16}H_{17}N_7$, and *Dinitro*, $C_{16}H_{17}N_3O_4$, derivs of Dixylamine were not found in the literature thru 1966

2,4,6-Trinitro-3,5,3',5'-tetramethyl-diphenylamine, $(CH_3)_2C_6(NO_2)_3.NH.C_6H_3(CH_3)_2$; mw 360.32. N 15.55%; yel crysts, mp 209°; sl sol in alc; prepd by heating 5-bromo-2,4,6-trinitro-m-xylene with m-xylidine in alc at 150–60° (Refs 3 & 4)

Tetranitrodixylamine, $C_{16}H_{15}N_5O_8$, not found in the literature thru 1966

2,4,6,2',4',6'-sym-Hexanitroxylylamine or **3,5,3',5'-Tetramethyl-2,4,6,2',4',6'-hexanitrodiphenylamine,**



$$O_2N.C\left\langle\begin{array}{l}C(CH_3)-C(NO_2)\\C(CH_3)-C(NO_2)\end{array}\right\rangle C-NH-C\left\langle\begin{array}{l}C(NO_2)-C(CH_3)\\C(NO_2)-C(CH_3)\end{array}\right\rangle C(NO_2);$$

mw 495.32, N 19.80%; yel crysts (from glac acet ac); mp 222° or brn-yel (from alc), mp 231°(dec) (Ref 5). It can be prepd by treating 2,4,6-trinitro-3,5,3',5'-tetramethyldiphenylamine with nitric & sulfuric acids (Refs 3, 4 & 5). It forms cryst salts with NH_4, K, Rb & Cs (Ref 5)

This compd is expl, although no expl props are reported

Refs: 1) Beil **12**, 1116 & [608] (Parent) 2) Beil **12**, 1104 3) Beil **12**, 1113(Trinitro & Hexanitro) 4) J.J. Blanksma, Rec **25**, 374–75(1906) 5) C.E. Moore et al, Talanta **13** (2), 171–74(1966) & CA **64**, 6533(1966)

DM. Chemical Warfare Service (CWS) symbol for Diphenylaminechloroarsine (Adamsite). See Vol 2 of this Encycl, p C167-R

DMW-Pulver. German Fast-burning NC propellant used in 7.65mm standard cartridges for pistols and revolvers. It was in the form of small greenish cylinders 0.4mm diam and 0.4mm high, which were not graphited.
Refs: 1) Stettbacher (1948), p45 2) PATR **2510**(1958), p Ger 38-L

DN or **Dn.** Fr abbr for Dinitronaphthalene

DN. A designation given on p 4312 of the 5thChemAbstractsDecennialIndex, Subject for 2-Cyclohexylnitrophenol

DNA. Abbr for Dinitroaniline. See Vol 1, p A408-R

DNAns. Our abbr for Dinitroanisole. See Vol 1, p448-L

DNB. Abbr for Dinitrobenzene. See Vol 2, p B46-L

DNDACP. Abbr for 1,3-Dinitro-1,3-diazocyclopentane, known also as 1,3-Dinitroimidazolidine. Described under Imidazolidine

DNEB. Abbr for Dinitroethylbenzene

DNEU. Abbr for Dinitroethyleneurea

DNG. Abbr for Dinitroglycerin

DNN. Abbr for Dinitronaphthalene

DNPA. Abbr for 4,4-Dinitropimelic Acid, $HOOC.CH_2.CH_2.C(NO_2)_2.CH_2.CH_2.COOH$. Its description is given in:
Ref: C.F. McDonough et al, "DNPA Summary Report", Nov 1961–Nov 1963, American Cyanamid Co, Contr NOw 60–07450C (Conf)

DNPF. Designation for Bisdinitrophenylformate

DNPN. Designation given in NavOrdRept **3020** for 1,3-Bis(nitroguanidino)-propanol-2-nitrate

DNPS. Designation for Bisdinitropropylsuccinate

DNPT or **DPT.** Abbr for Dinitropentamethyleneteramine; also called 3,7-Dinitro(bicyclo)-2,4,6,8,9-pentamethylene-1,3,5,7-tetramine, and as 1,5-Endomethylene-3,7-dinitro-1,3,5,7-tetraza-2,4,6,8-cyclooöctane; called in CA 3,7-Dinitro-1,3,5,7-tetraazabicyclo[3.3.1]-nonane. It is described under 1,5-Endomethylene-1,3,5,7-tetraza-2,4,6,8-cyclooöctane and Derivatives

DNPTB. Abbr for Dinitropropyltrinitrobutyrate

DNsoPT. Abbr for Dinitrosopentamethylenetetramine, described as 1,5-Endomethylene-3,7-dinitroso-1,3,5,7-tetraza-2,4,6,8-cyclooöctane

DNT. Abbr for Dinitrotoluene

DNT/HV$_2$. A Fr expl mixt which will be described as HV$_2$ (Explosif)

Dobgerät. A Ger device used to launch the "Taifun" biliquid rocket. It is described in detail in Ref 1

Refs: 1) TM9–1985–2(1953), p 223
2) PATR **2510**(1958), p Ger 38-L

Dodeconitrocellulose. See Vol 2, p C100-R under Cellulose Nitrates and p C108-R under High Nitrogen Nitrocelluloses

"Do-It-Yourself" Ammonium Nitrate – Fuel Oil (ANFO) Explosives. Under this name are known expls prepd at the site of blasting operations, either by pouring prilled AN (such as DuPont's SP–2 Prills which have an apparent density of 0.80 to 0.85g/cc) into the dry borehole and adding the oil later, or by adding the oil to the bag contg prills immediately before pouring into the borehole. Another similar method has been recently devised by which the oil is introduced into the unopened prill bag by means of an oil probe thru which the oil is metered. The unoiled prills can also be mixed with fuel oil mechanically at special plants and then distributed to sites of blasting (Ref 8, pp 62–3)

Some expls concerns, for example, DuPont Co, supply ready-made expls, such as "**Nilite 303**", which consists of prilled AN and of essentially non-volatile fuel oil. The density of this mixt is betw 0.75 & 0.85g/cc and the product is nearly odorless and shows very little tendency to segregate. It is applicable to nearly all field situations except for use in wet holes, but its major use has been in underground mining where fumes are a problem. However, it has been used also as a top load in quarry and construction work (Ref 8, p61)

Another expl consisting of prilled AN & fuel oil and supplied by the DuPont Co is the **ANFO-P**, which is a free-flowing product of density 0.75 to 0.85g/cc. As the fuel (or diesel) oil of this expl is volatile and of sharp odor, the mixture is not recommended for underground work, but its major application is in large quarry blasting (Ref 8, p62)

The third product of DuPont Co is the **ANFO**, which contains a blend of coarse- and fine-grained AN with inexpensive fuel (or diesel) oil. It is a free-running (but not blowable) product of density 0.90 to 1.00 g/cc. The best performance has been in larger drill holes, but small holes, down to 2 inches in diam have been shot successfully; not applicable for use in wet holes (Ref 8, p 62)

According to Urbański (Ref 9, p 482), the ratio of AN to fuel oil giving a zero oxygen balance is 95/5, and the rate of deton is highest (3300m/sec in a 100mm diam cartridge in a steel tube) when the fuel oil content is 5–6%. The most easily initiated mixt contains 2% fuel oil, while with a 10% fuel oil content the ease of deton is considerably impaired. Wetterholm (cited in Ref 9, p 482) has reported the expl props of ANFO mixts and the min chge diam for stable deton w/wo water in the mixt. It is advisable to use AN contg as little as possible of added inert material to prevent its caking. Wetterholm suggests that the quantity of inert material should not exceed 0.4% of the AN(FGAN contains 2–5% of added matter). See Vol **1**, p A364-L

Various methods used for the manuf of ANFO mixts and illustrations of some special equipment used in more elaborate methods are presented by Urbański (Ref 9, p 508). Safety recommendations have been made by the USBurMines (Refs 4 & 7)

Although invented in the USA (ca 1957), ANFO mixts are now used in opencast mining in Canada, Japan, Sweden & Russia (under the name *Igdanit*). Such expls are about twice as cheap as conventional AN rock expls and three times cheaper than Dynamites (Ref 16, p 483)

See also Refs 8, 9, 10, 11a, 12, 14 & 15

NOTE: Compare with Ammonium Nitrate Blasting Explosives in Vol 1 of this Encycl, pp A341ff

Refs: 1) Cook (1958), 14, 57 1a) S.R. Brinkley Jr & W.E. Gordon, ComptRendCongrInternChimInd 31^e, Liège, 1958 (Publ in IndChimBelge (Suppl), 231–39(1959) (Expl props of AN-fuel oil system. Max deton was obtd with 6% oil content, 1300ft/sec) 2) W.E. Tournay et al, "Some Studies in Ammonium Nitrate-Fuel Oil Compositions", Univ of Missouri SchMines&MetBullTechSer **97**, 164–74(1959) 3) R.W. van Dolah et al, "Further Studies on Ammonium Nitrate-Fuel Oil Compositions", Univ of Missouri SchMines &MetBullTechSer **98**, 90–101(1960) (Deton products & production of oxides of N) 4) Staff, USBurMinesInfCirc **7988**(1960), 12pp (Tentative safety recommendations for field-mixed AN blasting agents) 5) R.W. van Dolah et al, "Underground Blasting Accident Involving an Air-Placed Ammonium Nitrate-Fuel Oil Mixture", USBurMines RI **5988**(1962), 13pp 6) R.W. van Dolah & J.S. Malesky, "Fire and Explosion in a Blasting Agent Mix Building, Norton, Va", USBurMines RI **6015**(1962), 12pp 6a) T. Tsuchiya, JapP 9593(1963) & CA **60**, 6696(1964) (ANFO) 7) Staff, USBurMinesInfCirc **8179** (1963) (Safety recommendations for sensitized AN blasting agents) 7a) R.A. Cooley, USP 3113060(1963) & CA **60**, 6696(1964) (ANFO) 7b) K. Hino et al, USP 3111437(1963) & CA **60**, 6696(1964) (ANFO, cap sensitive) 7c) H.S. Berczely, USP 3129128(1964) & CA **61**, 4144(1964) (ANFO of low toxicity) 8) T. Seguiti, IndMineraria(Roma), **16**(6), 289–98(1965) & CA **64**, 7960(1966) (General uses and props of AN-combustible oil expls) 9) Ibid, **16**(8), 413–22(1965) & CA **64**, 7960 (1966) (Underground uses for AN-combustible oil expls) 10) H. Dabrowski, Cement-Wapno-Gips **20**(8–9), 267–74(1965) & CA **64**, 12453(1966) (A Polish expl called **Iponit** consisting of AN(granulated, porous) 94 & diesel fuel oil 6%. Methods of prepn and uses in limestone & gypsum quarries are described) 11) Blasters' Hdb (1966), 61–63 11a) H. Eckhard; R. Amberg, ZErzbergbau-Metallhuettenw **19**(4), 163–66, 169–72 (1966) & CA **61**, 563(1966) (ANFO in German iron ore mining) 12) J. Dusek & R. Mecir, Rudy (Prague) **14**(3), 72–74(1966) & CA **65**, 5292(1966) (First experience with ANFO for blasting in Czechoslovakia) 13) R.W. van Dolah et al, USBurMinesRept **6903** (1966), 32pp & CA **66**, 6374(1967) (Sympathetic detonation of ANFO) 14) Y. Wakazono, Kogyo-KayakuKyokaishi **27**(5), 288–94(1966) & CA **66**, 10931(1967) (Blasting perfomance of ANFO) 15) J. Kaemmler & J. Ruhmann-seder, Rudy (Prague) **14**, 139–41(1966) & CA **67**, 4302(1967) (Development of ANFO expls Dekamon 1 & Dekamon 2 at potash mines in East Germany) 16) Urbański **3** (1967), 470, 482, 483, 487 & 508

Dolgov Bomb. A closed bomb, similar to Bichel Bomb, but vertical, was mentioned in Vol 3 of Encycl, p C331-R. Accdg to its description given in the book of Yaremenko & Svetlov, its usual capacity is 15 to 50 liters, but the most commonly known bomb is of 50 liters. A sample of up to 100g can be exploded in a 50-liter bomb. Cylindrical body of the bomb is of heavy steel and it is closed, hermetically, with a horizontal, heavy steel cover, by means of bolts and nuts. The cover is provided with pressure gage, vents for removing the air before the expln, for removing the gases after the expln, and thermometer for measuring the temp inside the bomb before and after expln. A sample, provided with an electric detonator, is placed inside the bomb and its two electric wires go thru perforations in the cover to an electric switch. The cover of Dolgov Bomb is convenient to handle because the body of the bomb is in vertical position and not in horizontal as in Bichel Bomb

Procedure. After placing the sample inside the bomb, the cover, which is suspended over the bomb by means of a pulley, is lowered and fastened carefully on open top of cylinder by means of bolts and nuts. The air is evacuated from the bomb and residual pressure is measured (in mm Hg) as well as the temperature inside the bomb. After exploding the sample, the pressure and temperature of gases are measured and then the bomb is allowed to cool to RT. As result of this cooling, the water vapor will condense to liquid. If required to detn the compn of gases, they are conducted to a gas analyzer, such as of Orsat, Burrell, etc

The volume of dry gases can be detd from the formula:

$$Vg = \frac{Vb(P-w) \times 273 \times 1000}{760 \times T \times a} \text{ liters/kg}$$

where: Vg = Volume of gases formed on expln minus vol of water vapor, calcd to atm press and temp 0°C

Vb = Volume of bomb in liters

P = abs press after expln (in mm) minus residual press before the expln

w = tension of water vapor saturating the atmos at absol temp T°

T = absol temp of bomb's body at the moment of expln

a = weight of expl sample (100g)

To the above vol of dry gases, it is necessary to add the vol of water vapor, which is detd by evacuating the bomb by blowing thru it dry air and conducting the moist air thru tared drying tubes contg $CaCl_2$. If the wt of of absorbed water is b, wt of sample a, the vol of water vapor at atm pressure and 0°C can be calcd from the formula:

$$V_{H_2O} = \frac{22.4 \times b \times 100}{18 \times a} \text{ liters/kg}$$

and total specific volume:

$$V = Vg + V_{H_2O} \text{ liters/kg}$$

Maximum pressure formed on expln can be measured by any of the devices mentioned in Vol 3 of Encycl, p C330-R, or it can be calcd from the formula given on p C330-L. In the latter case, the vol of gases per unit wt must be previously known. If it is not known, it can be approximately calcd if the compn of the sample is known (as shown on p C330-L

Yaremenko & Svetlov give the following Table listing specific volumes as detd in Dolgov Bomb

Table

Explosive	Density g/cc	Specific Volume of Gases in l/kg	
		H_2O liq	H_2O gas
Trotil (TNT)	1.5	610	750
Tetryl	1.55	620	740
Gheksoghen (RDX)	1.5	700	890
TEN (PETN)	1.65	550	790
Ammonal 80/20 (80/20 Amatol)	0.7	300	720

Ref: N.E. Yaremenko & B.Ya. Svetlov, "Theory and Technology of Industrial Explosives", Promstroyizdat, Moscow (1957), pp 75–7

Domergue Explosive (1889) (Fr). A mixture of coarsely ground K chlorate and sulfur
Refs: 1) Daniel (1902), 207 2) Giua, Trattato **6**(I) (1959), 395

Donar (1901). A Sprengel-type expl patented in Russia by Fielder. It consisted of 80 parts of pulverized oxidizer (mixt of K chlorate 70 & K permanganate 30%) and 20 pts of a fuel oil (mixt of MNB 80 & turpentine 20%). As its solid and liquid components are not expl, by themselves, they could be transported safely to the place of work where they were mixed any time before blasting operations
Note: This expl may be considered as be-

longing to the type of "Do-It-Yourself" Explosives
Refs: 1) Daniel (1902), 300 (under Fielder) 2) Gody (1907), 266 3) Giua, Trattato **6**(I) (1959), 397 (under Esplosivo Fielder)

Donarit. A type of mining expl manufd in Germany for many years. It is known that at least one of the Donarits was used during WWII for military purposes (loading of some hand grenades) under the name of Füllung Nr 56 (Filler No 56). Its compn was: AN 67–80, TNT 25–12, NG 3.8, collodion cotton 0.2 & vegetable oil 4%. It was a yel, semi-plastic substance possessing nearly the same expl props as 80/20 Amatol, except that it was slightly more sensitive in impact- and rifle bullet tests (Ref 2, p 90 & Ref 4, p Ger 47-R)

The commercial WWII Donarits included:
Donarit 1 (Powdery type): AN 81.5, TNT 14, DNT (liq) 2.0, woodmeal 2 & dye (Caput mortum) 0.5%. It was manufd at the Krümmel Fabrik of DAG (Ref 4, p 38-L)
Donarit 1 (Gelatin type) AN 55.0, NGc 22.0, collodion cotton 1.0, TNT 5.0, DNT (liq) 6.0, woodmeal 0.8, Na nitrate 10.0 & dye (Caput mortum) 0.2%. It was manufd at the Krümmel Fabrik of DAG (Ref 4, p 38-L)
Donarit 2 (Powdery type): AN 84.0, NG 4.0, aromatic nitrocompds 3.0 & woodmeal 9.0% (Ref 1, p94 and Ref 4, p Ger 38-L)

Accdg to Weichelt (Ref 3, pp 37–8 & 375) there were used in Germany at the time of publication of his book (1953) three Donarits which had the approximate composition of: AN 86, Sprengöl (NG with NGc) 4–6 & TNT with Al powder 8–10%. Their props were reported as: cartridge density (including the paper) 0.87 to 0.98g/cc, Trauzl test values 355 to 435cc, deton velocity 3800 to 4850m/sec, impact sensitivity with 2kg wt 60 to 70cm, temperature developed on expln 2580 to 3345°C, volume of gases on expln at NPT 832 to 924 liters/kg and specific pressure 9900 to 10270kg/sq cm

Giua (Ref 5) gives for one of the German Donarits: AN 80, TNT 14, NG 4 & sawdust 2%; its detonation velocity is 4850m/sec
Refs: 1) Beyling & Drekopf (1936), 94 2) All & EnExpls (1946), p 90 2a) H. Gronemann, ZentrGewerbehygUnfallverhüt **30**, 97(1943) & CA **40**, 4523(1946) (Freezing of Donarit) 3) Weichelt (1953), 37–8 & 375 4) PATR **2510**(1958), pp Ger 38 & Ger 47) 5) Giua, Trattato **6**(I)(1959), 373 6) Urbański **3**(1967), 263 (Mining expl used in ammo during WWI)

Donarita. A Spanish, non-permissible coal mining expl listed in Vol 3 of Encycl, p C422-L under COMMERCIAL OR INDUSTRIAL EXPLOSIVES

Donarite. Engl for Donarit. Marshall **3** (1932), p175 gave: AN 67–80, NG 3.8, coll cotton 0.2, TNT 25–12 & rye flour 4%

Dope, Balanced. See next item

Dope (Base or Absorbent). In the prepn of expls such as Dynamites, which contain liquids (such as NG or NGc, known as "explosive oils") as their principal expl ingredients, it is essential to incorporate substances which are capable of absorbing these liquids, thus preventing exudation (sweating) during subsequent transporation and storage. The dopes may be subdivided into:
1) *Inactive*, such as kieselguhr, clay, NaCl, etc. They do not take part in any expl reaction, but serve only as absorbents. The expl consisting of 75% NG and 25% kieselguhr, known as *Guhrdynamite* was invented in 1867 by A. Nobel. The manuf of inactive-base Dynamites has been discontinued in the USA since 1907, but they are still used in Europe and other countries
2) *Active*, such as mixts of combustible and oxidizing materials. As combustibles may be used materials such as sawdust, woodpulp, woodmeal, dextrin, starch, various flours,

cellulose, collodion cotton, powdered cork, coal, charcoal, sulfur, ivory nut meal, etc; and as oxidizers AN, Na or Ba nitrate, K chlorate, K perchlorate, K bichromate, etc. These ingredients actually participate in the reaction of expln

For expls contg only a small amt of NG (or NG + NGc), such as *Permissible* which contain a maximum of 10% NG + NGc, the dope must be of low absorptive power to prevent lowering the sensitivity of the expl mixts. Expls contg NG (or NG + NGc) and powdery or coarsely ground dopes are known as *Powdery (or Straight) Dynamites*

If a small amt of soluble (low N content) NC (such as collodion cotton) is added to Dynamites, the NG (or NG + NGc) gelatinizes it and the resulting mixt is a more or less viscous gel. Such expls are known as *Gelatin Dynamites.* With larger amts of NC (7–10%), the resulting gel is so tough that no exudation can take place even without addition of absorbing dopes. Such expls are very powerful and are known as *Blasting Gelatins* (See Vol 2 of Encycl, p B211-R). With Dynamites contg less than 7% NC and high percentage of NG (or NG + NGc) some absorbent must be added but in much smaller quantity than is required in NG expls contg no NC

The term "dope" also includes the anti-acidic compds, such as Ca or Mg carbonates and Mg or Zn oxides. These are added in small quantities to neutralize the acidity which usually develops on prolonged storage of Dynamites

There are also "dopes" which were patented by various investigators or plants. For example, Daniel (Ref 1, p32) listed a product patented by the Atlantic Dynamite Co. The dope was prepd by heating a mixt of sulfur & rosin until the foaming ceased and then pulverizing the resulting solid after its cooling

It should be noted that in selecting dopes, one must remember that not only the compn of a material is of importance, but also its physical state. When it is desired to obtain a Dynamite of low density (as in the case of permissible expls), very coarsely ground dopes are used. For example, it was proposed some time ago to incorporate in permissible expls coarsely ground peanut hulls or uncooked corn flakes

The term *Balanced Dope* is applied to any mixture consisting of a combustible material and an oxidizer in proportion corresponding as closely as possible to zero oxygen balance to CO_2 (Ref 6, p 9)

Refs: 1) Daniel (1902), 32 2) Naoúm, NG (1928), 324–35, 349–50, 362–65, 367–68, 371, 373, 398–99, 401–02, 407, 409, 411, 414–15, 418–20 & 423–45 3) Davis (1943), 275, 334, 339–42, 345–46 & 351–53 4) Bebie (1943), 73 5) Kast-Metz (1944), 456–57 (Zumischpulver) 6) Cook (1958), 9 7) W.H. Rinkenbach in Kirk & Othmer **8**, 2nd edit (1965), 639ff

Doppelzünder (Double Igniter). A Ger double igniter for acoustic mines, developed during WWII at Troisdorf Fabrik DA-G. These mines consisted of two delay detonators (crimped into a sleeve) and mounted coaxially with their bases pointing away from each other, and with their fuseheads connected in series for simultaneous firing. The fuseheads had one direct connecting wire between them, while the other connecting wire from each of them made contact with a metal ring on the outside of the assembly. This arrangement permitted the fuseheads to be fired by applying an appropriate voltage to these two rings

Ref: W. Taylor et al, BIOS Final Rept 644 (1945), p 17

Doppler Effect (or Principle) and Its Application for Military Purposes. The Doppler (or more correctly Döppler) effect, first observed by C.J. Döppler of Prague in 1842, involves the apparent change in the frequency of light, sound, or electromagnetic waves observed while the source and the observation point are in motion relative to each other. The effect is commonly observed in the change of pitch of a train whistle as the train approaches and then continues on by

Because electromagnetic waves possess many of the characteristics of light, particularly as their frequencies approach that of light, the Döppler principle is applied now to electromagnetic radiations in connection with radic and *radar* equipment for determining velocity and distance of moving targets such as *guided missiles* or planes

In one system which can be used to track a missile, the transmitter operates at a constant and accurately known frequency (f). The beam from the transmitter triggers a beacon transmitter in the missile, and the transmitter, in turn, operates at twice the original frequency (2f). The latter signal is picked up by ground receiving stations and, at the same time, these stations receive the ground transmitter's signal and doubles its frequency internally thru a frequency-multiplying stage. The two doubled frequencies are then mixed and the resulting difference frequency is proportional to the velocity of the missile. This is because the motion of the missile effectively changes the transmitted frequency by an amount proportional to the velocity of the missile. The velocity vector betw the missile and any point on the ground can be accurately measured by means of a *döppler radio* system. By integrating the velocity values from the instant of takeoff, position-vectors can be detd with great accuracy. If two or more stations are used, the position of the missile with respect to a fixed coordinate system can be readily established (Ref 2, pp 165–66) (See also Ref 4, p 100)

If instead of transmitting a continuous wave, the Döppler system transmits a saw-tooth frequency-modulated (FM) wave, both the velocity and the instantaneous distance betw the missile and the ground station can be detd simultaneously. This principle is explained in Ref 2, p 166 and illustrated in the graph. The FM radar system is capable of measuring accurately only short distances (Ref 2, p 166)

Döppler principle is also used in radar employed in *homing guidance systems*. A missile homing (terminal) guidance system is one which can "see" the target by some means and then institute commands to the control system so that the missile can fly to the target. The homing of a missile can be active, semiactive and passive. If *active*, the missile is both the originator and receiver of radar signals, and as such is subject both to detection and to jamming by window or tinsel. If *semiactive*, the missile uses a radar receiver to pick up the target from echoes of pulses sent out by a cooperating ground facility; in which case the missile's guidance system is subject to jamming but not readily to detection. If *passive*, the missile depends only on radiation from the target, such as heat (infra red rays), electrostatic discharge, radio sound or light; in which case the system (except when using light) is more or less resistant to detection (Ref 4, p 150) (See also Ref 2, pp 449–51). Radar cannot be used in passive system (Ref 2, p 449)

The *Döppler Homing Systems*, which belong to the active group, may be subdivided into two systems:

a) *FM-CW Döppler System* is provided with the circuitry, receiving antenna and equipment contained in the signal converter block, permitting it to convert the frequency of echo signal into indication of the velocity of the target with respect to the missile. The difference between the transmitted signal and the frequency of the returned signal is due to the Döppler shift (Ref 2, pp 451–52)

b) *Pulse Döppler System*, performs the functions of an FM-CW system, but it can also select a target by its range. In this system, the transmitter generates short pulses at a repetition frequency which can be continuously varied. The stabilized local oscillator (stalo) furnishes low-intensity priming power to the transmitter to effect phase coherence betw successive pulses. A duplexer provides low-impedance paths and effectively reduces the transfer of energy in all but the desired direction. The stalo also provides a local oscillator signal, suitably shifted in frequency, which is mixed with the receiver signals to generate a receiver intermediate frequency. The Döppler receiver, with its associated velocity gate, filters the signal so as to reject the undesired Döppler frequencies (Ref 2, pp 451–52)

The so-called *Velocity-Damping Döppler Radar* used for velocity damping of an inertial system is somewhat different from the homing type of Döppler. Its brief description is given in Ref 2, p 464

The Döppler principle is also employed in a radar telemetering system to accurately determine instantaneous values of range, altitude and velocity. Under title *Pulsed-Döppler-Modulation* (Ref 2, p 529) is discussed the Döppler radar system designed for use in a missile. It detects the change in frequency between a transmitted signal and the echo. This frequency change is converted into voltage proportional to ground speed

The device known as **DOVAP** (Döppler Velocity and Position), used for determination of flight of a missile, is described in Ref 3. This instrumentation system is so organized that several receiving stations feed data simultaneously from outlying locations to a centrally located recorder station. A remote control system permits an operator at the central recording station to check out and operate all receiver stations associated with the recording station

There are many modifications of the basic Döppler technique resulting in systems such as DORAN, EXTRADOP, TRIDOP, UDOP & others (Ref 5)

Refs: 1) Burgess (1957), 118–20 (Döppler radar) 2) Anon, "Guided Missiles Fundamentals", Air Force Manual, **AFM 52-31** (1958), 165–70, 434, 451–52, 464 & 529 3) Anon, "Development of DOVAP Remote Operation Equipment", Land-Air, Inc., Holloman, New Mexico, Final Rept (March 1958); Contract DA-29-040-ORD-1302 4) Glossary of Ord (1959), 100 (Döppler effect); 150 (Homing) 5) Capt G. Merrill, "Principles of Guided Missile Design", Van Nostrand, Princeton, NJ (1959), 199 (Döppler Effects, Döppler Radar, Döppler Shift), 200 (DOVAP) 6) OrdTechTerm (1962), 103-R

Dora or Sevastopol Gun, known also as **Gustav Geschütz.** A Ger mortar caliber 80cm (reported also as 82cm) used during WWII at the siege of Sevastopol, Russia. It fired an 8 ton shell with muzzle velocity of 2200–2400ft/sec and maximum range of 29 miles. Wt of expl was 2000 lbs, wt of proplnt 2500 lbs, wt of mortar 1375 tons and length of barrel 105 ft. One of the shells is on display at Aberdeen Proving Ground Museum, Maryland

Refs: 1) PBRept **925**(1943), p 18 2) PATR **2510**(1958), p Ger 176-L (Sevastopol Gun) 3) PATR **2700**, Vol 2(1962), p B114-L

Dorée, Charles (1875– ?). A Brit scientist specializing in Cellulose and its derivatives. Author of the books "The Methods of Cellulose Chemistry", Chapman & Hall, London, 2nd ed (1947), 543pp and "Les Méthodes de la Chimie de la Cellulose Comprenant des *Méthodes de Recherches sur les Celluloses* Composées", Dunod, Paris (1949), 578pp and many other publications

NOTE: No biographical info was in the literature thru Dec 1966

Doremus, R. O. (1824–1899). An Amer scientist who invented the so-called "Doremus Cartridges" (See below), which were used to some extent in the War between the States (1861–1865). These cartridges replaced those using brass, which was in short supply. He also invented some expls which were used for blasting the *Mont Cenis* tunnel betw Italy & France

Ref: Van Gelder & Schlatter (1927), 25

Doremus Cartridge. For its prepn a chge of BkPdr, intended to serve as a proplnt, was compressed in a brass mold having the shape of a sewing thimble. The compressed chge was removed and its flat end was glued to the base of a lead bullet. After waterproofing the cartridge by dipping it into collodion, it was dried and the bullet greased. The finished cartridges were packed together with primers and then transported to the troops

Their use was discontinued after the US Civil War
Ref: Van Gelder & Schlatter (1927), 25

Dorfit. A Favier-type expl, formerly used in Germany for coal mining. Two compns are listed in Ref 1 and one in Ref 2:
Dorfit I – AN 65, K nitrate 5, TNT 6, meal 4 & NaCl 20%; permissible at charge smaller than 530g (Ref 1)
Dorfit II – AN 61, K nitrate 5, TNT 15, meal (rye flour) 4 & NaCl 15%; Trauzl test value 219cc; permissible at chge smaller than 300g (Refs 1 & 2)
Refs: 1) Anon, SS **3**, 337 (1908) 2) Marshall **1** (1917), 391 3) Clift & Fedoroff **2** (1943), p N68

Dormant Missile. A Rus device known as **Golem** can be left unattended in concealed locations and then fired at will by remote control
Ref: Anon, Ordn **48**, 488 (1964)

Doron. A light, body armor material developed in the USA during WWII. It is a glass fabric base plastic laminate, having a density of 1.75g/cc and greater ballistic efficiency than Hadfield steel (used for making helmets) in stopping fragments from small shells (up to 40mm HE), or in stopping bullets from 45mm pistol or from Tommy gun. It is not suitable, however, for stopping rifle or machine gun bullets at close range, or in stopping fragments from large shells

The high tensile strength of "Doron" depends on the fact that it contains a multitude of glass fibers, each of them possessing a tensile strength capable of resisting pressures up to 300000psi. It might be stated that Doron stops fragments or bullets by forcing them to expend their energies in breaking a great number of fibers in succession
Ref: L. Gilman, "Plastics Developments", Tech Div Lecture, Picatinny Arsenal (31 Jan 1947), 3–4

Dortmund-Derne Versuchsstrecke (Testing Gallery at Dortmund-Derne). German facility used for the following official tests of permitted explosives (Wettersprengstoffe):
a) Trauzl Block Test. The maximum expansion allowable for permissible explosives was 240cc for a 10g sample initiated by a No 8 cap
b) Gap Test (Detonationsübertragung). The minimum acceptable gap was 20mm when testing cartridges of 35mm diameter were initiated by No 8 caps. Nearly all permissible explosives had much higher gap values than 20mm and the sheathed explosives usually gave a value of 300mm due to the sensitivity of the sheathing which contained about 15% of NG
c) Ability to Transmit Detonation (Detonationsfähigkeit) was determined by the so-called "Four Cartridge Test" (qv)
d) Power of Detonators was formerly determined in a type of ballistic pendulum. Only No 8 detonators were allowed to be used in coal mines. The usual filling for such detonators was: 0.75g Tetryl and 0.5g MF or 0.3g LA/LSt mixture
e) Gallery Tests were conducted with methane-air mixtures and with coal dust
Refs: 1) BIOS Final Rept 1266 (1947), pp 1–3 2) PATR **2510** (1958), p Ger 215-R (Versuchsstrecke)

DOUBLE-BASE AND TRIPLE-BASE PROPELLANTS. A description of *double-base proplnts* containing NC + NG was given in this Encycl under: BALLISTITE (Vol 2, pp B8-L to B10-L), under CANNON PROPELLANT (Vol 2, pp C33-L to C37-L) and under CORDITE (Vol 3, pp C531-L to C541-R). Some Ball Powders, described in Vol 2, pp B11-R to B16-L can be made double-base. The description did not include the German proplnts which were described previously in Refs 1, 2 & 3 and in Ref 4, pp Ger 142 (Table 43) & Ger 146 (Table 46), under PROPELLANT (Treibmittel)

The *triple-base proplnts* contg NC + NG + NGu are described in Vol 2 of Encyl, pp C34 (Table V) and p C37-L

We are not giving here the description of Ger double-base proplnts contg NG because there was nothing unusual about them and many were similar to US proplnts

The description given below will be devoted to the problem of gun barrel erosion caused by NC-NG proplnts and to the development in Germany, before WWII, of a new class of proplnts which reduced the erosion to such an extent that the serviceable life of a barrel increased 4–5 times in comparison with NC-NG proplnts

It has been known for a long time that *erosion* is the greatest factor in the wearing of the rifling of a gun and in shortening its serviceable life. Even before the gun becomes unusable, the firings begin to be less effective because of the escape of gases between the walls of the barrel and of the shell. This escape not only reduces the chamber pressure (causing reduction in muzzle velocity of the shell with consequent reduction of range and penetration), but also causes excessive muzzle flash

Before WWII it was detd in Germany that the serviceable barrel life of rapid firing AA gun was only 1700 firings when NC-NG proplnt of calorific value of 950 kcal/kg was used, and the muzzle velocity was 3300ft/sec. When the NG content was reduced in order to obtain the calorific value of 820kcal/kg, the number of serviceable firings could be increased to 3500. This meant that reduction of 130kcal/kg in calorific value, doubled (approximately) the life of the barrel. No further improvement in life of AA guns could be obtd when using NC-NG proplnts (Refs 1 & 2 and Ref 4, p Ger 43, under Erosion of the Bore)

As in the period before and during WWII, Germany suffered great shortage of steel hardening metals (such as Cr, Ni, Mn, Mo, etc) required for making modern gun barrels and also due to the shortage of labor and, to some extent, of ordinary steel, the replacement of eroded guns became quite a problem. Mainly for these reasons a series of proplnts of low calorific values was developed in Germany beginning in 1934, which finally permitted prolonging barrel life of rapid firing guns to as much as 17000 firings. This value was higher than anticipated (See below)

When General Uto Gallwitz (killed at the Russian Front near the end of WWII) developed the proplnt known as **"G" Pulver** ("G" stands for the 1st letter of the name Gallwitz) by replacing in double-base proplnts NG with DEGDN (Diethyleneglycoldinitrate), it was assumed that it should prolong the barrel life to 2 x 3500 = 7000 firings. This value was calcd on the assumption that each 130kcal/kg reduction of cal value should double the life of the barrel, as it was doubled in the case of reduction from 950 to 820kcal/kg. This theory worked in the case of NG proplnts, but did not seem to be applicable when NG was replaced with DEGDN, since 17000 firings were obtd instead of 7000 anticipated (Refs 1 & 2 and Ref 4, p Ger 43)

One of the earliest "G" proplnts consisted of: NC (blend of sol & insol NC's giving an average N content of 12.2%) 61.53, commercial DEGDN 26.37, EtCentr 7.50, vaseline 1.60, phthalate (not specified) 0.65, Mg oxide 0.25, graphite 0.10 & K sulfate 2.00%. Its calorific value was betw 670 & 690kcal/kg. This proplnt was used in tubular shape during WWII in 100mm Army Gun designated as K18 (Kanone 1918) (Ref 4, pp Ger 71 & Ger 145)

A *flaked* proplnt for use in various howitzers contained NC (13.0% N) 54.40, DEGDN 44.50, Acardite 0.50, graphite 0.05, MgO 0.05 & K sulfate 0.50% (Ref 4, p Ger 145). A *tubular* proplnt for use in 88mm Army AA Gun contained: NC (12.0% N) 64.08, DEGDN 27.47, EtCentr 5.35, phthalate 0.90, vaseline 1.85, graphite 0.10 & MgO 0.25%; calorific value 730kcal/kg (Ref 4, p Ger 145). A tubular proplnt for various Army guns: NC (12.0% N) 60.55, DEGDN 25.95, EtCentr 3.75, hydrocellulose 3.00, DNT 4.00, a-MNN 2.50, graphite 0.10 & MgO 0.15%; calorific value 730kcal/kg (Ref 4, p 145). A tubular proplnt for Naval guns: NC (12.0% N) 65.53, DEGDN 23.87, EtCentr 9.00, phthalate 1.25, graphite 0.10 & MgO 0.25%; calorific value 730kcal/kg (Ref 4, p 145). A universal proplnt for rocket launchers: NC (12.5% N) 60.17, DEGDN 35.33, hydrocellulose 1.50, DPhUret 1.00, EtPhUret 1.40, IG Wax E 0.35 & MgO 0.25%, with K nitrate 0.80 added; calorific value 900kcal/kg (Ref 4, p 145)

The commercial DEGDN used in "G" proplnts was prepd by nitration of the product known in Germany as "Polyglykol", which was a mixt of DEG (known in Ger as "Diglykol") with small amt of EG (ethyleneglycol), called in Ger Glykol and in Engl glycol. Polyglykol was a product easily available from non-feed materials

The advantage of DEGDN over NG, included (besides producing much "cooler" proplnts) more effective gelatinization for NC's. This permitted one to prepare more homogeneous proplnts and with smoother surface grains, while using smaller quantities (as low as 20% of DEGDN vs 40–45% of NG) and in a wider range. Another advantage of DEGDN was that it permitted incorporation, without becoming brittle, of materials which do not take part in gelatinization, such as K sulfate (flash reducer), NGu, MgO, graphite, etc

The disadvantage of DEGDN is its high volatility (4–5 times more volatile than NG) and for this reason the "G" Pulver was found to be unsuitable for hot climates

Inasmuch as the Ger troops had trouble with DEGDN proplnts during WWII in African Campaign, Gen Gallwitz proposed replacing DEGDN with a less volatile product (only 1.5 times more volatile than NG), TEGDN (Triethyleneglycoldinitrate), which could be prepd by nitration of a commercial product known in Germany as "Triglykol". The incorporation of TEGDN not only gave a low volatility product, but it also permitted obtaining even lower calorific value products (such as 650kcal/kg vs 670–690 for DEGDN proplnts)

A typical TEGDN proplnt contained: NC (a blend of sol & insol giving an av N content of 12.2%) 58.55, TEGDN 25.10, EtCentr 12.00, MgO 0.25, graphite 0.10 & K sulfate 4.00%; calorific value 650kcal/kg. Gelatinizing props of TEGDN were found to be similar to those of DEGDN (Refs 1, 2 & 3 and Ref 4, pp Ger 71 & Ger 145)

All "G" proplnts were slow-burning and were efficient only in weapons with long barrels, which allowed complete combstn of proplnts. They were unsuitable, even when used in small grains (flakes), in medium and small caliber howitzers and in mortars because their barrels were too short to allow complete combstn of proplnt while the shell was still in the barrel (Refs 1 & 2 and Ref 4, pp Ger 70 & Ger 71). For these weapons (as well as for the others), the *Gudolpulver*, developed in 1937 at the Dynamit AG laboratories, was quite suitable

In Ref 4, pp Ger 143 (Table 44), Ger 144 (Table 45a), Ger 145 (Table 45b), Ger 146 (Table 46) and Ger 147 are given several other Ger WWII formulations of NC-DEGDN proplnts. They are similar to the ones described above

The **Gudolpulver**, described in Refs 1, 2 & 3 and in Ref 4, p Ger 81 and mentioned above, was a "G" Pulver in which a large amt of NGu (Nitroguanidine) was incorporated. It was actually a "triple-base propellant"

As was mentioned above "G" proplnts were unsuitable for use in short-barrel weapons. In order to obtain satisfactory results with such weapons (without resorting to NC-NG proplnts, contg flash reducing compds), it was decided at Dynamit AG laboratories to modify "G" Pulver in such a manner that its speed of burning was increased without increasing the muzzle and breech flashingness. Such a proplnt was obtd in 1937 on incorporating a large amt of NGu (Nitroguanidine) in "G" Pulver. Due to the fact that DEGDN or TEGDN used in "G" Pulver is a good gelatinizer of NC, incorporation of large amts of NGu (which is not a gelatinizer for NC and is not gelatinized by nitrated glycols) did not produce brittle proplnts. In order to have a proplnt of good performance, the crysts of NGu should be short and fine and evenly distributed throughout the mass of proplnt. Good blending of Gudolpulver ingredients was achieved by kneading in a Werner-Pfleiderer apparatus the required amt of NC with nitrated glycol until a uniform jelly was obtd. While continuing to knead other ingredients, including short-fibered NGu, were gradually added and thoroughly incorporated. Then the mass was rolled between heated rollers for about 25 mins and the resulting sheets cut to the desired size (Refs 1 & 2 and Ref 4, p Ger 81)

Following are compns of some Gudol proplnts:

a) *Flaked for Various Howitzers:* NC(13.0% N) 38.03, DEGDN 31.12, NGu 30.00, Acardite 0.50, MgO 0.25 & graphite 0.10% (Ref 4, pp Ger 81 & Ger 145)

b) *Tubular for 88mm Army AA & AP Guns:* NC (12.0% N) 39.48, DEGDN 16.98, NGu 30.00, DPhUret 4.25, EtPhUret 5.00, K nitrate 4.00, graphite 0.10 & MgO 0.25%; calorific value 730kcal/kg (Ref 4, p 145)

c) *Tubular for Various Army Guns:* NC 12.0% N) 44.00, DEGDN 18.85, NGu 20.00, DNT 3.50, α-MNN 2.00, DPhUret 1.50, PhEtUret 1.50, hydrocellulose 4.00, K nitrate 4.00, Acardite 0.40, graphite 0.10 & MgO 0.15%; calorific value 720kcal/kg (Ref 4, p 145)

d) *Tubular for Naval Guns:* NC (12.0% N) 42.45, DEGDN 18.20, NGu 25.00, DPhUret 4.5, EtPhUret 4.50, K sulfate 5.00, MgO 0.25 & graphite 0.10%; calorific value 730 kcal/kg (Ref 4, p 145)

e) *Square for 75mm Guns:* NC (12.4% N) 38.4, DEGDN 31.5, NGu 29.0, graphite 0.2 & unaccounted 0.9% (Ref 4, p 143)

f) *Flaked for 76.2mm Russian Gun (captured):* NC (12.2%N) 38.6, DEGDN 30.9, NGu 30.2 & graphite 0.3% (Ref 4, p Ger 143)

Several similar formulations of Gudol proplnts are given in Ref 4, pp Ger 143 (Table 44), Ger 145 (Table 45b) & Ger 146 (Table 46). They were listed in Refs 1, 2 & 3

Proplnts contg NGu were found to be especially suitable for use in modern rapid-firing weapons, such as AA guns and guns used in armored vehicles. Here the problem of breech-flash became of utmost importance because the breech must be opened immediately after each firing and less time is given for cooling the chamber gases than in the case of slow-burning weapons. The problem was complicated because most of the rapid-firing weapons were provided with semi-automatic breech closures and muzzle brakes. Application of brakes after each firing tended to retain the gases back in the barrel and, when the breech was opened the gases emerged in a glowing condition, endangering the personnel near the breech and even causing ignition of combustible material located in their vicinity

With the use of Gudol proplnt, the breech as well as muzzle flash were practically eliminated and so was the smoke. The erosion of gun barrel was very low (Refs 1, 2 & 3 and Ref 4, p Ger 81)

Refs: 1) U. Gallwitz, "Die Geschützladung" (Propellant Charge), Heereswaffenamt, Berlin (1944) (English translation is available at PicArsnLibrary) 2) O.W. Stickland et al, "General Summary of Explosive Plants", PBRept **925**(1945) 3) H.H.M. Pike, "Report on Visit to Düneberg Factory of Dynamit AG", CIOS Rept **31–68** (1946) 4) B.T. Fedoroff et al, "Dictionary of Explosives Ammunition and Weapons" (German Section), PicArsnTechRept **2510** (1958) 5) G.A. Heath & R. Hirst, "Some Characteristics of High Pressure Combustion of Double-Base Propellant", pp 711–20 in 8thSympCombstn (1960) (publd 1962) 6) V. Lindner

in Kirk & Othmer **8**, 2nd edit (1965), 705 (DB solventless Mortar Sheet Proplnt M8), 706 (Rocket Proplnt JPN) and 705–08 (DB cast rocket proplnt)

Double- and Triple-Base Propellants. Following are some more or less recent references which were not included in Encycl, Vol 2, under BALLISTITE, Vol 2 under CANNON PROPELLANT and Vol 3 under CORDITE

Refs: 1) B. Troxler, BritP 568721(1945) & CA **41**, 4312(1947) (Flashless proplnt for 37mm AT & AA gun: NC, NG, $Ba(NO_3)_2$ & KNO_3) 2) T. Thomson & ICI Ltd, BritP 578372(1946) & CA **41**, 1843(1947) (Flashless tubular double-base proplnt for 3.7-inch AA gun: NC (12.2% N), NG, DBPh, DPhUrea & 0.4–1.4% K as K_2SO_4 or KNO_3) 3) B.L. Crawford, USP 2440327(1948) & CA **42**, 5230 (1948) (Double-base proplnts having controlled ignition by incorporating C black or other coloring compd) 4) S. Gordon, E. Whitworth & ICI Ltd, BritP 616898(1949) & CA **43**, 4856(1949) (Low-burning & low-erosion flashless proplnt: NG 13, NC (colloidable) 20, NGu 55, DBPh 10 & diethyldiphenylurea 2%) 5) D.S. Bruce & E.L. Klein, USP 2499295 (1950) & CA **44**, 4680(1950) (Use of lamp black in NC-NG proplnts) 6) G.K. Adams & L.A. Wiseman, Selected Combustn Problems: Combustn Coloquim, Cambridge Univ, England **1953**, 277–88, 381–402 & CA **48**, 10345(1954) (Combustn of double-base proplnts) 7) G.C. Whitnack et al, AnalChem **27**, 899–901(1955) & CA **49**, 11283(1955) (Polarographic detn of NG in double-base proplnts) 8) A.J. Camp et al, ChemEngrgProgr **52**, 79–82F (1956) & CA **50**, 5291(1956) (NOTS China Lake, Calif small-scale facilities for developing double-base rocket proplnts) 9) J.O. Watts & H. Stalcup, AnalChem **28**, 975–78(1956) & CA **50**, 11669(1956) (Chromatographic-acidhydrolysis method for detn of triacetin in double-base proplnts) 10) V. Lindner in Kirk & Othmer **8**, 2nd edit (1965), 701–04 (Double- and triple-base solvent extruded artillery proplnts, such as M2 & M17)

Double Base Cannon Propellants. See Vol 2 of Encycl, p C33-L

Double Base Howitzer Propellants. See Vol 2 of Encycl, p C34, Table V

Double Base Mortar Propellants. See Vol 2 of Encycl, p C35, Table VI

Double Base Recoilless Rifle Propellants. See Vol 2 of Encycl, p C35, Table VII

Double Cartridge Test, Ardeer, abbr **ADC Test.** The test described by McAdam & Westwater (Ref) is used for detn of "sensitivity to propagation" of blasting expls. It is similar to the *Gap Tests* described in Vol 1 of Encycl, pp XIV & XV. In the ADC test, two cartridges of the same compn & weight are placed end to end on a stout iron rail or bar. One cartridge called "primer" is provided with a standard detonator, while the 2nd cartridge is known as "receptor". After firing a "primer", a search is made for any residual portions of the "receptor". If both cartridges detonate completely then the test is repeated with two new cartridges of the same expl but leaving a "gap" of 0.5 inch betw the ends. Then the tests are continued with 1.0, 1.5, 2.0 etc gaps until the receptor fails to detonate. For example, if complete detonation takes place at 3-inch gap, but at 3.5-inch the deton of receptor is incomplete, the result of test is reported as 3–3.5 inch

Fresh expl would detonate across a bigger gap than same expls stored for some time; also larger diam cartridges deton over bigger gap than smaller diam chges. Cartridges confined in stemmed shot-holes would deton at bigger gap than those in the open

Ref: McAdam & Westwater (1958), 19–20

Double-Cone Mixer. This term may be applied to the "Twin-Shell" laboratory blenders

made by The Patterson-Kelley Co, East Stroudsburg, Pa. These blenders have shells made of heavy transparent Lucite or 304 stainless steel. A double-cone mixer is particulary useful in blending pyrotechnic compns, vacuum drying of heat-sensitive materials, or uniformly dispersing liquids into solids and controlling the granulation of the product
Ref: ChemEngineeringCatalogue, Reinhold Publishing Co, NY, 52nd ed (1968) p 20

Double-Effect Powders (Poudres à double effet, in Fr). A series of expls developed in 1881–1883 by Turpin. One of its varieties, which consisted of K chlorate 80 & coal tar 20%, may be considered as a predecessor of *Pyrodialites*, also developed by Turpin. Part of the chlorate could be substituted by K or Pb nitrate or K permanganate. Some kieselguhr or silica could be incorporated to prevent exudation in cases of high fluidity of tar

Pyrodialites were expls based on K chlorate and coal tar, but the tar was previously purified to remove the acids. In order to take care of acidity which could develop in storage, some alkaline carbonate or bicarbonate was incorporated. Some charcoal and other ingredients could also be incorporated and part of chlorate (or even total) could be substituted by other oxidizers, such as nitrates, perchlorates, bichromates, or permanganates

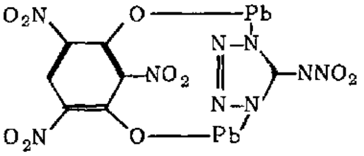
Following are some typical examples of Pyrodialites:
a) Forte (strong): K chlorate 80, tar 10, charcoal 6 & Na or Amm bicarbonate 4%
b) Lente (slow): K chlorate 40, K nitrate 40 & tar 20%, with 4–5% of bicarbonate added
c) K chlorate 15, Amm perchlorate 60, tar 10, Amm trinitrophenate 10 & Amm permanganate 5%
d) K chlorate 40, Amm perchlorate 40, tar 10 & Amm trinitrophenate 10%
e) K perchlorate 80, tar 10 & Amm trinitrocresylate 10%
f) Amm perchlorate 50, GuN (guanidine nitrate) 40 & tar 10%
Refs: 1) Cundill (1889) in MP **6**, 105–06 (1893) 2) Daniel (1902), 207 [Double effet (Poudres dites à)]; 661–64 (Pyrodialites); 777–78 [Turpin (Poudres) dites à double effet] 3) Fedoroff & Clift 4 (1944), p29

Double Lead Salt of Formic and Nitric Acids, $Pb(NO_3)(OOCH)$, "active"; and $Pb(NO_3)_2-[Pb(OOCH)_2]_x$, "inactive". These two samples were investigated for possible military use as suggested by their originator who also furnished their formulas and descriptive terms (Ref)

It was found that the two samples could not be detonated by any of the std test procedures used at Picatinny Arsenal. The lead salts were non-hygroscopic and possessed the same order of stability as TNT in the 120° Vacuum Stability Test. Because of their insensitivity to initiation, these Pb salts were considered to be unsuitable for practical military applications (Ref)
Ref: K.S. Warren, PATR **1283**(1943), "Investigation of Double Lead Salt of Formic and Nitric Acids Submitted by R.M. Cole, Fort Washington, Pa"

Double Salt of Cesium Dichromate and Cesium Decahydrodecaborate. USP 3256056 of June 14, 1966 of R.K. Armstrong (to DuPont Co) is described in CA **65**, 8660(1966) as follows: A B-contg compd is prepd which is suited for use in delay-producing compns, which ignite reliably and uniformly and has excellent resistance to impact and to static electricity. The compd has the formula $(Cs_2B_{10}H_{10})_2Cs_2Cr_2O_7$. Triethylammonium decahydrodecaborate 3.2, and 2.94 $K_2Cr_2O_7$ were dissolved in 50 parts water. Five parts 25% aq CsOH was then added to the soln, and the soln was heated on a steam bath 2 hrs; ~70% of the water was evapd from the resulting soln and the concentrate was cooled; a fine, yellow, needle-like ppt formed. The concentrate was filtered off and the filter cake was washed with 95% EtOH and dried. The IR spectrum of the yellow cryst product showed that a true mol compd, i.e. the double salt of $Cs_2Cr_2O_7$ and Cs decahydrodecaborate, was formed. The product was recrystd 3 times from water, 260–70°(with discoloration). The product flashed at 290°. A series of 8 conventional elec blasting caps were assembled. The bronze shell was 1.125-in. long with an outer diam of 0.272 in and an av inner diam of 0.26 in. Into this shell was loaded 5 grains of Pentaerythritol Tetranitrate pressed at 200 lbs. Above this base charge was loaded 3 grains $Pb(N_3)_2$ pressed at 200 lbs. In 4 shells, 3 grains of the double salt pressed at 200 lbs was inserted as the ignition chge, adjacent the primer chge. In the remaining 4 shells, 2 grains of loose double salt was inserted as the ignition chge, adjacent the primer chge. In each shell was inserted a conventional rubber plug assembly in which the 0.0019-in diam bridge wire was soldered to the lead wires sepd to provide a 1/8-in span and projecting 1/8-in from the base of the rubber plug. When a 5 amp DC was applied to the lead wires, each of the blasting caps detonated. The uniformity in delay within each group of caps was good as compared to conventional delay blasting caps

Double Salt of Lead Nitroaminotetrazole and Lead Styphnate. USP 3310569 of March 21, 1967 of E.A. Staba (to Olin Mathieson Corp) is described in CA **67**, 2273(1967)as follows: Crystals of the double salt (I) are formed by



(1)

reaction of a soln of sol Pb salt, desirably in excess, with a soln of an alkali or alk earth salt of Styphnic Acid and of Nitroaminotetrazole. For example, 1M aq Mg styphnate and K Nitroaminotetrazole were prepd, the solns having, resp pH values of 4.5±0.5 and 7±0.5. Five ml of one soln was added to 5ml of the other in a suitable beaker and the resulting soln dild to a total vol of 50ml, the pH being adjusted to 6.3±0.3 by the addn, as reqd, of MgO or of Styphnic Acid. The soln was heated in a water bath to a temp of 75–80°. While the soln was well stirred at that temp, 15ml *M* $Pb(NO_3)_2$, dild with water to a vol of 50ml, was added dropwise in 15–25 mins to ppt the cryst double salt. The product was washed 4–6 times with water and then decanted to obtain a 90–95% theoretical yield of I. A dry product may be obtained by drying in air or in a vacuum at 25 to 80°. In concordant 5-shot series, 0.4g charges crushed the following wts of 20/30 Ottawa sand to particles finer than 30 mesh: Pb azide 14.2, Hg fulminate 18.9, Pb styphnate 16.1 and I 24.0g, resp. The proprietory name of double salt is **Stabanate.** It is stable and suitable as an initiating compd

BritP 1069440 of May 17, 1967 of E.A. Staba (to Olin Mathieson Corp) and described in CA **67**, 5265(1967) seems to be identical with the *Stabanate* described in USP 3321343 (1967)

Double Salts of Basic Lead Picrate With Other Organic Lead Salts. USP 3262956 of July 26, 1966 of J.F. Kenney (to Remington Arms Co, Inc) is described in CA **65**, 13450 (1966) as follows:

Procedures are given for the prepn of a new series of primary explosives useful in compns for ammunition primers. Thus, 9g NaOH in 50cc water is added to 23g Picric Acid in 250cc water with stirring. After the temp is raised to 50–5°C, 110g lead acetate in 300cc water is added over 10 mins. The temp is held at 50–5°C for 30 mins. The soln is filtered, washed, and dried. The compn (yield, 60g) is *Monobasic lead picrate-lead acetate*, $Pb(C_6H_2N_3O_7)_2.Pb(OH)_2.Pb(C_2H_3O_2)_2$, explosion point 300°F. It is a weak but sensitive primary explosive. Other compds prepd and resp explosion points are:

Dibasic lead picrate-lead acetate, $Pb(C_6H_2N_3O_7)_2.2Pb(OH)_2.2Pb(C_2H_3O_2)_2$, 276°C
Tribasic lead picrate-lead acetate, $Pb(C_6H_2N_3O_7)_2.3Pb(OH)_2.3Pb(C_2H_3O_2)_2$, 277°C
Monobasic lead picrate-lead formate, $Pb(C_6H_2N_3O_7)_2.Pb(OH)_2Pb(HCO_2)_2$, 258°C
Dibasic lead picrate-lead formate, $Pb(C_6H_2N_3O_7)_2.2Pb(OH)_2.2Pb(HCO_2)_2$, 277°C

Tribasic lead picrate-lead formate,
$Pb(C_6H_2N_3O_7)_2.3Pb(OH)_2.3Pb(HCO_2)_2$, 247°C
Monobasic lead picrate-lead propionate,
$Pb(C_6H_2N_3O_7)_2.Pb(OH)_2.Pb(C_3H_5O_2)_2$, 291°C
Dibasic lead picrate-lead propionate,
$Pb(C_6H_2N_3O_7)_2.2Pb(OH)_2.2Pb(C_3H_5O_2)_2$, 265°C
Tribasic lead picrate-lead propionate,
$Pb(C_6H_2N_3O_7)_2.3Pb(OH)_2.3Pb(C_3H_5O_2)_2$, 255°C
Monobasic lead picrate-lead lactate,
$Pb(C_6H_2N_3O_7)_2.Pb(OH)_2.Pb(C_3H_5O_3)_2$, 276°C
Dibasic lead picrate-lead lactate,
$Pb(C_6H_2N_3O_7)_2.2Pb(OH)_2.2Pb(C_3H_5O_3)_2$, 263°C
Monobasic lead picrate-lead acrylate,
$Pb(C_6H_2N_3O_7)_2.Pb(OH)_2.Pb(C_3H_3O_2)_2$, 268°C
Dibasic lead picrate-lead acrylate,
$Pb(C_6H_2N_3O_7)_2.2Pb(OH)_2.2Pb(C_3H_3O_2)_2$, 261°C
Tribasic lead picrate-lead acrylate,
$Pb(C_6H_2N_3O_7)_2.3Pb(OH)_2.3Pb(C_3H_3O_2)_2$, 254°C
Monobasic lead picrate-lead methacrylate,
$Pb(C_6H_2N_3O_7)_2.Pb(OH)_2.Pb(C_4H_5O_2)_2$, 268°C
Dibasic lead picrate-lead methacrylate,
$Pb(C_6H_2N_3O_7)_2.2Pb(OH)_2.2Pb(C_4H_5O_2)_2$, 278°C
Tribasic lead picrate-lead methacrylate,
$Pb(C_6H_2N_3O_7)_2.3Pb(OH)_2.3Pb(C_4H_5O_2)_2$, 283°C
Monobasic lead picrate-lead aminoacetate,
$Pb(C_6H_2N_3O_7)_2.Pb(OH)_2.Pb(C_2H_4NO_2)_2$, 274°C
Dibasic lead picrate-lead aminoacetate,
$Pb(C_6H_2N_3O_7)_2.2Pb(OH)_2.2Pb(C_2H_4NO_2)_2$, 216°C
Tribasic lead picrate-lead aminoacetate,
$Pb(C_6H_2N_3O_7)_2.3Pb(OH)_2.3Pb(C_2H_4NO_2)_2$, 219°C

Double Salts in Explosives. Following are examples of expl compns using various double salts: 1) Imperial Chemical Industries Ltd, GerP 574271(1933) & CA **27**, 3612(1933) [A priming chge contg double salt $K_2Ba(NO_3)_4$ 30–60, MF 20–45 & $Pb(CNS)_2$ 10–40%] 2) A. Weale CanP 348291(1935) & CA **29**, 3518(1935) (Same compn as above) 3) A. Le Roux, MP **35**, 121–32(1953) & CA **49**, 5842(1955) [Tetramethyl Ammonium Nitrate (TMA) 25 & NH_4NO_3 75% form a eutectic mixt, mp 138°; its zero OB mixt (TMA 13.4 & AN 86.6%) can be detonated with 1.5g MF while the eutectic mixt requires 2.0g MF; rate of deton is 3020 at d 1.0g/cc; power 112% of PA; with 15% RDX added to zero OB mixt, it is 120% of PA, requires 0.5g MF and has rate of deton of 3920m/sec at d 1.0g/cc] 4) F.P. Bowden & J.D. Blackwood, BritP 715828(1954) & CA **49**, 2736(1955) [Fast burning BkPdr: Eutectic mixt (KNO_3 54, $LiNO_3$ 30 & $NaNO_3$ 16%) 75, alder charcoal (70% C) 15 & ground roll sulfur 10%] 5) S. Singh & K. Singh, Chem & Ind (London) **1957**, 1297 & CA **52**, 1831(1958) (Prepn & props of KPb Styphnate, $K_2Pb(C_6HN_3O_8)_2.5H_2O$, scarlet-red crysts, d 2.37; hydrated salt is insensitive to impact; anhyd salt is very sensitive to impact and explodes with great violence in a flame) 6) A. Rost & G. Knöffler, GerP 1057514(1959) & CA **55**, 6868(1961) [Castable expl compn AN 50.7, $NaNO_3$–$Mg(NO_3)_2$ eutectic 12.7, PETN 11.7 & RDX 24.9%] 7) L.F. Audrieth & G.C. Hale, USP 2929698–99(1960) & CA **54**, 12588(1960) [Expl salts having general formula $HN{:}C(NH.NH_2)_2.HX$ (diaminoguanidine salts) & $H_2N.N{:}C(NH.NH_2)_2.HX$ (triaminoguanidine), where X=nitrate, picrate, perchlorate, etc, are useful in military & other application; a 50/50 mixt of the nitrate salt & TNT gives a castable expl]

Douglas Powder. See Vol 3 of Encycl, Table on pp C452–C453, under British Permitted Explosives (Sheathed and Unsheathed)

Douille. Fr for Cartridge Case. One of the recent Fr cartridge cases, the "Douille d'acier en tole enroulée", has been manufd from steel and rolled sheet iron by the Forges et Ateliers de Construction Electrique de Jeaumont, Paris, 8^e

DOVAP. See under Doppler Effect (or Principle) and Its Application for Military Purposes

Dove. An Air-to-Surface Missile developed by the USNavy but never became operative. It was built around a 1000-lb GP (General Purpose) Bomb and was controlled in flight and homed in on its target by infrared seeking

device in its nose
Ref: F. Ordway & R. Wakefield, "International Missile and Spacecraft Guide", McGraw-Hill, NY (1960), p118

Dowmetal. Trade-mark of Dow Chemical Co, Midland, Mich, applied to a series of light and tough metals contg up to 85% Mg
Ref: CondChemDict (1961), 419
Note: It seems that Dowmetal is similar to Mg-Al alloy, contg ca 80% Mg, known as "Elektron". It was used by the Germans during WWII for construction of casing of Incendiary Bombs filled with Thermite or other incendiary material. Such a bomb is described in Vol 2 of this Encycl, pp B235 & B236 [See also PATR **2510**(1958), p Ger99-R, under Incendiary Compositions]

DP. Chemical Warfare Service (CWS) symbol for Dichloromethylchloroformate (Diphosgene). See Vol 2 of Encycl, p C167-R

DPA or **DPhA.** Abbr for Diphenylamine

DPE. Abbr for Dipentaerythritol

DPhA. Our abbr for Diphenylamine

DPEHN. Abbr for Dipentaerythritol Hexanitrate

DP Powder. Abbr for DuPont Powder

DPT. Same as DNPT (Dinitropentamethylenetetramine) described as 1,5-Endomethylene-3,7-dinitro-1,3,5,7-tetraza-2,4,6,8-cyclooctane

DPT (homo). Designation for Dinitrohexamethylenetetramine described as 1,5-Endoethylene-3,7-dinitro-1,3,5,7-tetraza-2,4,6,8-cyclooctane

Dragon. An Amer antitank weapon that hurls guided missiles which can be fired from a soldier's shoulder. It weighs 27 lbs and is powerful enough to destroy tanks and other targets; highly accurate and far superior in range and lethality than the current medium antitank assault weapons. It was designed to replace the 90-mm recoilless rifle

With Dragon, which employs a command-to-line-of-sight guidance system, no ranging or leading the target is required; all the gunner has to do is to keep his sight on the target. It was developed and is in production for the Army by McDonnel Douglas Corp; also is to be produced by Raytheon Company, provided the initial Raytheon lot passes qualification tests

The US DRAGON (XM47 Surface Attack Guided Missile System) is a medium range, antitank/assault weapon which is being developed for the Army infantryman. It is lightweight (30 lbs), highly accurate and lethal. The weapon can be carried, set up, and fired by one man. Two major assemblies comprise the Tactical System: Tracker (reusuable) and the Round consisting of: a) disposable launcher and b) missile. The system utilizes a command-to-line-of-sight guided missile that is tube launched with minimum recoil and is automatically guided to the target by means of a tracker and wire link. Dragon will replace the 90mm Recoilless Rifle and will be deployed at the platoon level to provide infantry companies with an antitank and assault fire capability
Refs: 1) Anon, Ordn **52**, 86(1967) 2) The Common Defense Bulletin No 385, Nov 15, 1972, in Ordn **57**, Nov-Dec, 1972

Dragon. French Ground-to-Air Two-Stage Rocket launched in Sahara on Oct 30, 1963
Ref : Anon, Missiles & Rockets, **13** (11 Nov 1963), p 12 (No description of rocket is given)

Dragonite. One of the older British permitted expls used for coal-mining: KNO_3 44.5, NG 35.5, Nitrocotton 2.5, woodmeal & charcoal 12 & vaseline 5.5%
Ref: Marshall, Dict (1920), 30

Dragon's Breath. (Drachenatem in Ger & Aliento del dragón in Span). An incendiary agent for use in flame throwers (Flammenwerfer in Ger & Lanzallamas in Span), developed in GtBritain before WWII and then improved in the USA. As this liquid becomes viscous and harder the moment it comes in contact with air, it is possible to throw it as far as 100 meters (Refs 1 & 2)
Refs: 1) Stettbacher (1948), 129 2) Stettbacher (1952), 159

Dragon's Teeth. Wedge-shaped concrete anti-tank obstacles laid in multiple rows
Ref: Webster's 3rd New International Dictionary (1961), p 685

Dranite. One of the perchlorate expls developed by Alvisi
Ref: U. Alvisi, RassMin **37**, 213–15, 243–46, 276–79, 299–303 (1913) & CA **7**, 1974 (1913) (No compn given)
Note: Giua, Trattato **6**(I)(1959), p 401, lists, among others, the following expls developed by Alvisi: a) *Cratiti* (See Vol 3 of this Encycl, p C555-L, where the page of Giua's book is erroneously listed as 451 instead of 401) b) *Nitrocratiti* – mixts of Amm perchlorate with NG, NC, dynamite and blasting gelatin c) *Cremoniti* – they were listed in Vol 3 of Encycl, p C556-L, as Cremonita together with a Cremonita contg AN 80 & cannel coal 20% and d) *Manlianiti* – mixts of perchlorate with sulfur & carbon. No Dranite was listed by Giua

Dreadnaught Powder. A permissible expl: AN 73–77, Na nitrate 17–14, TNT 3–5 & Amm chloride 4–6%
Ref: CondChemDict (1950), 288 (Not found in later editions)

Drehspiegelkamera (Rotating Mirror Camera). See under CAMERAS in Vol 2 of Encycl, p C14-L

Dreyse, Johann Nicolas von (1787–1867). German gunsmith, inventor of Needle Gun (1836), the first successful turn bolt action breechloader. He was enobled for this invention
Ref: James R. Newmann, "The Tools of War", Doubleday, Doran & Co, Garden City, NY (1943), 43–44

Driers. See under Drying of Materials Used in Ordnance

Drift Signals and Markers. These are devices used over water as an aid to navigation by providing a stationary reference point for determination of drift of an airplane with respect to its course. They are also used to mark the location of a submarine or other underwater object for the attention of surface vessels; also for determining the wind direction before landing, or to mark the location of the surface for emergency landing at night

At night the spot is marked by a pyrotechnic pellet or candle, weighing 2 to 4 lbs, which is ignited by a fuse on impact with water. It then floats on the surface, producing a light for ca 3 mins, visible up to 7 miles in clear weather. These signals are usually known as *Night Drift Signals*, also called *Aircraft Float Lights*

By day the spot is marked by a slick-producing device called *Slick Marker*. This signal consists of a cylindrical paper bag contg either a finely divided metallic powder (such as bronze), or a special dye (such as Uranine dye, which gives a yel-grn fluorescent slick). The paper bag, which weighs with contents ca 2.5 lbs, breaks on hitting the water, thus allowing the metal- or dye-powder to spread as a slick. This serves as a reference point which may be visible up to 10 miles at an altitude of 3000 ft

In Refs 1, 2 & 3 are described the following Amer signals: 1) Day Drift Signal AN-Mk 1 2) Slick Marker AN-Mk 59 3) Night Drift Signal AN-Mk 4 and 4) Night Drift Signal AN-Mk 5

Current US military ground smoke & marine dignals are described in Ref 4

Refs: 1) Ohart (1946), 313–14 2) Anon, "Military Pyrotechnics", **TM 9–1981**(1951) 122–29 3) Anon "Ammunition, General", **TM 9–1900** (1956), 171–75 4) Anon, "Military Pyrotechnics", **TM 9–1370–200/ TO 11A10–1–1**(1958), 96–104

Drill (or Dummy) and Practice Ammunition. All three types of ammo are designed or adapted for use in the training of a weapons crew, but there is the following difference between Drill (or Dummy) and Practice Ammunition:

a) *Drill or Dummy Ammunition.* Completely inert ammo serving to train crew in assembling, fuzing, unfuzing and other handling of bombs, projectiles, etc (Ref 3). Hayes (Ref 1, p605) lists also *Gage Bombs.* They are drill bombs designed for use in the testing of new types of airplanes for clearances, capacity and functioning of bomb racks

b) *Practice Ammunition.* An ammo designed for training troops in marksmanship. It has the same characteristics as *Service Ammunition,* but instead of being loaded with expls, it contains only a small spotting charge. Sometimes paractice ammo is inert (Refs 1, 2 & 3)

Refs: 1) Hayes (1938), 604–05 2) Ohart (1946), 8, 99, 114, 172 & 221 3) Encycl of Expls, PATR **2700**, Vol 1(1960), p A383-R, under AMMUNITIONS 4) Encycl of Expls, PATR **2700**, Vol 2 (1962), p B229-R, under BOMBS

Drilling Booster Cavities. When TNT (or other HE, such as Composition B) is loaded into a shell by casting, provision should be made to have a booster cavity of proper dimensions. (See BOOSTER in Vol 2 of Encycl, pp B243-R to B246-R). If an HE is insensitive to friction, such as TNT, the cavity may be produced by drilling the surface of cast HE in the center. For this operation the shell is secured in a horizontal position and the cast is drilled out with a twist drill of high-speed steel, rotating at not more than 120 rpm. All loose particles should be removed from the booster cavity by using a vacuum unit approved for use in hazardous operations. If there is any cast expl in the thread of a shell, it should be scraped off using a bronze pick before starting to drill

According to Ref 2, booster cavity forming tools should be maintained in a polished condition and should be handled to prevent marring their surfaces. Ammo items contg expls may be drilled either while in a vertical or horizontal position. Vertical drilling is preferred, since withdrawal of expl chips & dust is easier, and proper drill alignment is more easily attained & maintained in such a position. To protect adjacent operators, HE's should be drilled, faced, milled, sawed or otherwise machined within rooms or cubicles having safety approved & specified reinforced concrete walls

The following HE's, cased or uncased, may be machined w/o protection being afforded the operator & w/o coolant:

a) Amatol b) Black Powder (in components only) c) Composition B d) Explosive D and e) TNT

The following HE's, cased or uncased, may be machined w/o protection being afforded the operator provided a suitable noncombustible, nontoxic coolant (sometimes tap water) is directed on the tool & expl at their point of contact: f) Baratols g) Cyclotols h) 50/50 & 10/90 Pentolite and i) Tetrytol. When essential, any other HE may be machined by remote control, with the operator protected by a suitable shield. Initiating expls should not be machined if other means of forming can be used. When machining expls contg Al and using a noncombustible, nontoxic coolant, the coolant must not consist of nor contain water (Ref 2)

If drilling is being accomplished w/o the operator being afforded protection, only a single drill, having a diam greater than 1/4 inch must be used. Machining of cased expls is permitted if the operation requires the tool to remove metal prior to or after contact with the expl, provided it is performed by remote control. Where wet machining is to be performed, positive automatic interlocking devices must be provided to insure that machining cannot be started until the coolant is flowing (Ref 2)

Refs: 1) Anon, "Ammunition Inspection Guide", **TM 9–1904**(1944), 145 2) Anon, "AMC Safety Manual", AMC Regulation **385–224**(1964), 26–10

Drillingspulver. Ger short tubular powder for howitzers (Haubitze) such as the 10cm Haubitze

Ref: H. Brunswig, "Das Rauchlose Polver", W. DeGruyter, Berlin (1926), p 131

Drip Oil (Dinitro Oil, Liquid DNT, or DNT Low Oil) (Tropföl or Dinitrotropföl in Ger). A black tarry oil consisting of a mixt of various isomers of DNT with a small amt of MNT's and TNT's. As the purified DNT has been used in some Amer proplnts (See Ref 6, pp C34 & C35) several TNT plants used to stop at dinitration in order to obtain DNT. After separating the crude product from nitrating acid, while still warm, and the excess of acidity in crude product, known as *Dioil*, it was neutralized with soda soln. After allowing the Dioil to cool and to set to an oil-solid mass, it was broken into lumps, heated to ca 40°, and placed in an open trough with the bottom inclined from both sides toward the middle and provided with a slot at the lower part. The temp of the air surrounding the trough was ca 40°. During this process, called *sweating*, the more fusible portion (usually located on the surface of DNT crysts) flowed away leaving the purified product, mostly 2,4-DNT. The dark liquid was caught in sump tanks and sold under the name of Drip Oil. More efficient method of separation was by centrifuging. This was done in Italy

The other type of Drip Oil was fomerly obtd in the US and other countries when crude TNT (known as *Trioil*) was purified by washing with volatile solvents, such as alcohol or xylene. This took place during WWI and also later before the *sellite* treatment (Na sulfite soln) was used for purification. For washing crude TNT, it was packed into a large Büchner type funnel, known as *Nutsche*, and a volatile solvent poured over the product. After allowing it to stand for a few minutes, a vacuum suction was applied and the washings collected. This operation was repeated until the color of the product on the funnel became buff or light brown, which indicated that it was nearly pure α-TNT. Purification could also be achieved by using a centrifuge. The liquid was evaporated under vacuum to distil off the solvent which was collected and saved for washing other batches of crude TNT. The residue, which was a tarry oil known as *Liquid TNT* (Flüssige Tri in Ger), or *Drip Oil* consisted of an eutectic mixt of β– & γ– isomers of TNT with isomers of DNT and some other impurities

Accdg to G.D. Clift, for many years no use was found in the US for Drip Oil, although an attempt was made to utilize it in low-freezing Dynamites, such as *Lydol*. As there was no market for this oil betw WWI and WWII, some of it was destroyed by burning. During WWII the DuPont Co proposed using it in RDX Compositions C-2 and C-3

Because the oil is a good gelatinizer for NC, it could be used in some proplnts if its compns were not so variable and if it were not so reactive (Refs 1, 2 & 4)

While Drip Oil (Liquid DNT) was not used much in US Dynamites, its use in other countries was not so limited. For example, Naoúm (Ref 3) lists the following two Dynamites using this product: 1) NG 39,, liq DNT 24, collod cotton 2, woodmeal 5 & Na nitrate 30% (Ref 3, p 362) 2) NG 48, liq DNT 15, collod cotton 2, woodmeal 5 & Na nitrate 30% (p 363)

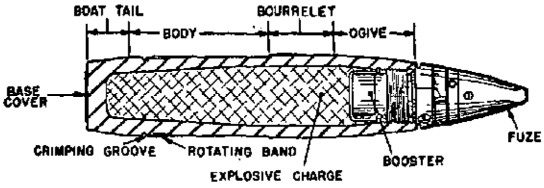
Stettbacher (Ref 4, p 240) who calls "Flüssige Tri" the liquid brn-blk product which could be separated from crude TNT, stated that it was used in great quantities in plastic, low-freezing Gelatin-dynamites. The same author called (Ref 4, p 261) "Tropföl" the liquid which dripped from crude DNT crysts and stated that it was used in AN and chlorate expls. We have listed in our Encycl (Ref 6, p C159) several French, Italian and Swiss Cheddites which contained 70–79% Na chlorate & 16 to 23.8% liq DNT together with other ingredients

In our work on German expls (Ref 5) are listed among others: Ammonit I (1932) & Gelatine-Donarit (1936), each contg 6% of liq DNT (p Ger 30, Table 9) and Nitrobaronit A, which contained 5% of liq DNT (p Ger 118, Table 28)

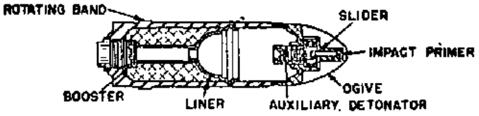
Refs: 1) Marshall 1(1917), 259 2) Colver (1918), 178 3) Naoúm, NG(1928)
4) Stettbacher (1933), 240 & 261 5) PATR **2510** (1958), pp Ger 30 & Ger 118 6) PATR **2700**, Vol 2 (1962), pp C34 & C35, Tables V & VI and p C159, Table 4

Driving (or Rotating) Band of an Artillery Projectile and Explanation of Terms **Grooves, Lands, Muzzle** and **Rifling.** The term **Barrel** is defined in Vol 2 of Encycl, p B22-R, **Bore** on p B248-R and **Bourrelet** on p B258-R

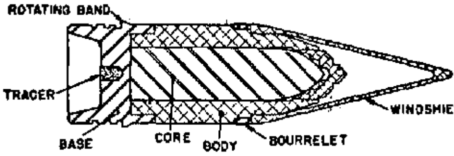
Accdg to Hayes (Ref 1, p 558) and to Ohart (Ref 2, p 93), all artillery projectiles greater than 0.60 inch in diam, except those for muzzle-loading weapons (such as trench mortars, which use fin-stabilized projectiles), are equipped with *rotating bands*, called by the British *driving bands*. Typical projectiles equipped with such bands are shown in Fig. The bands are made either of a soft metal (Cu or gilding metal) or a plastic ring and are usually seated in a circumvential cavity *(groove)*, machined around the body of the projectile near its base (See Fig)



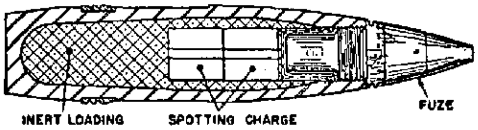
TYPICAL H.E. SHELL



POINT-FUZED H.E.A.T. PROJECTILE



TYPICAL H.V.A.P. SHOT



TYPICAL PRACTICE PROJECTILE

VARIOUS ARTILLERY PROJECTILES

For assembly, the band, which has an interior diameter smaller than that of the seat into which it is to be fitted, is expanded by heat, slipped over the base of the projectile, and forced into the groove by means of a banding press. The groove is previously roughened to prevent slippage of the band due to stresses set up by driving edge of the rifling. After assembly, the band is machined to final form and dimensions. The central section of a band is usually cylindrical in shape, with annular grooves to take care of excess metal displaced by *lands of the rifling* (See below). The front end of each band is tapered to insure proper seating in the bore while the rear usually has a raised lip to assist in centering the projectile initially, and as a gas check. The grooves cut into driving band serve to collect metal displaced from the band during its travel thru the bore caused by engraving, pressure of propellant gases and centrifugal force. Excess metal so collected is prevented from forming a skirt, known as *fringe*, in rear of the band. As fringe formation is a cause of excess dispersion and short range, it is very undesirable, and for this reason the *non-fringing* types of bands are used now. Typical band cross-sections are shown in Fig 40, p 93 of Ohart (Ref 2) reproduced here

SINGLE GROOVE 37 M/M. M54

DOUBLE GROOVE 90 M/M. M82

TRIPLE GROOVE 240 M/M. M114

TYPES OF ROTATING BANDS
FOR ARTILLERY PROJECTILES

The band, which is larger in diam than the bore of the gun and tapered at the forward end corresponding to the *forcing cone* at the origin of rifling, performs the following functions:

a) Centers the rear of projectile in the bore, and supports it in its travel, while the *bourrelet* (qv) supports its middle part on the *lands*, which are defined below under rifling
b) Locates the projectile initially in a fixed position, axially in the gun, and holds it in place when the gun is elevated. This is necessary to secure uniform ballistics from round to round
c) Acts as a gas check, preventing movement of proplnt past the proj in the bore of the gun then escaping into atmosphere without performing propelling work
d) Imparts *spin*(rotation about the longitudinal axis) to the projectile necessary for its stability in flight. This occurs because the band, being of soft metal (or plastic), becomes engraved by the lands of the rifling during its passage thru the bore of the gun, caused by the pressure of proplnt gases on the base of the projectile. This engraving is accomplished partly by cutting and partly by forming

In *recoilless ammunition*, the force required to accomplish engraving of the band would interfere with accomplishment of the recoilless feature, and the projs for this ammunition are provided with *pre-engraved* bands. Provision is made to insure that the proj will be inserted in the recoilless rifle in proper relationship to the rifling (Ref 5, p 31-R & Ref 6, p 35)

There is also the so-called *welded overplay driving band*, which consists of a band formed on projectile by depositing molten metal by welding technique, followed by machining to the required diameter. This avoids weakening of the proj wall as would occur in machining the band seat deep enough to maintain a swaged band and to reduce the possibility of band loss (Ref 5, p 31-R & Ref 6, p 35)

Ohart (Ref 2, p 93) gave formulas for calculating the width of driving band and the stress in it caused by centrifugal force

Rifling consists of helical *grooves*(channels) cut in the bore of a rifled gun, beginning at the front face of the *chamber* (space at the *breech* end of a gun occupied by *cartridge*) (See Vol 2 of Encycl, p C70-R and C147-L, under "Chamber of a Weapon") and extending to the *muzzle* (the end of the barrel from which projectile emerges). The purpose of the rifling is to impart *spin* to the proj, so the proj will travel nose first to the target. Engagement of the proj with rifling is accomplished by means of driving (or rotating) band defined at the beginning of this description. Such bands are used in guns of greater than 0.60 inch in diameter. The gun bore diam is detd by the *ridges* between the rifling grooves. These ridges are referred to collectively as the *lands*, and the sides of the lands are called *edges*. The *twist of rifling* at any point is the inclination of a groove to the element of the bore and it is expressed as the number of *calibers* (See Vol 2, p C5-R) of length in which the helix makes one complete turn as, for example, one turn in 40 calibers. The direction of rifling is usually right hand, causing the proj to rotate in clockwise direction, as viewed from the base. This is known as the "right-hand twist". There are also weapons with "left-hand twist." The above mentioned twists are *uniform* because the angle of the helix to the element of the bore is constant. There are, however, weapons (outside of the USA) with *increasing* (or *gaining*) *twist*. The twist may start either at zero or at some low value and then increase accdg to some function of travel, either to the muzzle, or to some point short of muzzle at which point the twist would become uniform up to the muzzle

The present practice of the US Ordnance Corps is to use rifling of uniform twist (Ref 5, p 245-L)

Refs: 1) Hayes (1938), 558–59 2) Ohart (1946), 93 & 99 3) Anon, "Artillery Ammunition", **TM 9–1901** (1950), 36 & others 4) Anon, "Ammunition General", **TM 9–1900** (1956), 122 5) Glossary of Ord (1959), 31-R (Band, Rotating); 124-L (Fringing Groove); 245-L (Rifling) 6) OrdTechTerms (1962), 35 (Band, rotating; pre-engraved and welded overlay)

Drone. A term which has been used to designate pilotless aircraft, target missiles and the like (Ref 2). Also a remotely controlled, pilotless aircraft used as a target or to perform tasks which are hazardous to a human pilot, such as probing a nuclear cloud, surveying enemy territory, and target practice (Ref 1)

Refs: 1) Capt G. Merril, Edit, "Principles of Guided Missile Design", Van Nostrand Co, Princeton, NJ (1959), 203-R 2) OrdTechTerm (1962), 105

Drone Charges. These are used where it is desired to load the max wt of expl in min time in a boat or other carrier which is to be run up to the firing point by remote control and detonated en masse

Ref: NDRC Summary Rept of Div 8, Vol 1 (1946), 62

Drop-Ignition Method. For detn of ignition point of combustible liquids and gases the standard procedure (D-286-30), described in Ref 1, can be used. One or more drops of the liq to be examined are dropped onto the bottom of a 125cc Pyrex Erlenmeyer flask suspended in a bath of molten solder or lead. An elec heated "Alundum" cylinder is specified in the revised method & procedure (Ref 1)

A modified method was proposed by Scott et al (Ref 2), who substituted an elec heated stainless steel block for the molten metal

Refs: 1) ASTM Standards, Pt **3**(1942), 90–91 (ASTM D286–30), now replaced by ASTM D2155–66, Pt **17**(1967), 737 (Autoignition temp of liq petroleum products) 2) G.S. Scott et al, AnalChem **20**, 238–41(1948) & CA **42**, 4755(1948)

Drop Safety of Bomb Test or **Bomb Functioning Test.** Drop safety of bomb may be defined as the maximum height of drop for a given bomb on a given surface without causing the bomb to fail or without detonation

An empirical formula computed at Aberdeen PG before WWII as a result of dropping various bombs from an airplane at a height of ca 2000 ft on 2 ft deep reinforced concrete hard surface laid over a suitable ballast is:

$$M = 1000 + \frac{16000}{\sqrt{7.5 + W/3 - 1}}$$

where M is the *setforward ratio* which is equal to height of drop/Deceleration distance or Total decelerating force/Weight of bomb) and W is weight of bomb. The above formula was computed for bombs with shear strength of the body equal to 50000lb/sq in, when dropped on hard concrete surface, such as was constructed at Aberdeen PG prior to 1940. The new surface constructed in 1940 gave higher values of setforward ratios

The value of M can also be calcd from the formula:

$$M = \frac{2\pi t S}{Wm/(D-t) + 2We(D-2t)}$$

where: D = outside diam of bomb in inches, S = shear stress of bomb walls in lb/sq in, due to the combination of: a) tangential stress on bomb walls due to setforward of filler and b) longitudinal compressive stress in bomb walls due to setforward of bomb

t = thickness in inches of bomb walls in cylindrical position

We = total wt in lbs of expl in bomb, exclusive of nose

Wm = wt in lbs of metal back of nose (Ref 2, pp 209–10)

Hayes (Ref 1, p 612–13) lists the following two tests which are actually drop tests:
1) *Safety Test.* It was to determine whether the bomb can be dropped safely, that is without detonation on impact. It was concerned with the sensitivity, strength, and general design of the fuze, the sensitivity and suitability of the bomb filler, and the structural strength of the bomb assembly
2) *Hard Surface Test.* It was used to determine the strength of demolition bomb cases by dropping them on the surface of concrete block one ft thick over crushed rock 2 ft thick. The bombs (except the 100-lb size) had to penetrate the concrete without breaking or becoming unduly deformed when the maximum diam of the bomb had passed thru the concrete. The altitude of drop varied betw 1500 & 4000 ft, the greater height used for the smaller bombs

The tests specified in Ref 3 for inert & HE-loaded bombs are as follows:
Drop Tower Test. Loaded, unfuzed bombs are dropped from varying heights up to 50 ft onto a well supported steel plate 3–5 inch thick. Each bomb undergoes only one drop. The test is conducted in 3 cycles, using 3 separate bombs. Each cycle is to consist of, and be in order as follows:
a) base down b) nose down and
c) side down (on lugs). The height of drop is increased from 15ft to the point of failure or 50ft whichever is lower. In some cases, it is more economical to start at 50ft and decrease the height. The bombs are observed for evidences of deformation, fracture, or functioning
Drop Test from Airplane – Hard Surface Test. Bombs are inert loaded to proper wt and center of gravity. They are released to impact from various heights (not over 10000ft). Bombs weighing less than 800lbs are dropped on 2-ft thick, reinforced concrete. Bombs over 800lbs are dropped on 7-ft thick, reinforced concrete. These surfaces are superimposed on crushed stone placed on firm ground. SAP-bombs weighing less than 1000 lbs are dropped on a 2-ft thick surface, those aver 1000lbs on a 7-ft surface. The degree of damage, its location on the bomb body, and penetration or perforation of the concrete is observed

HE-loaded bombs with inert fuzes are dropped on concrete surfaces of thickness & at altitudes of release which conform to those specified for inert bombs. Observe penetration, deformation of bomb body, and functioning of the expl
Bomb Functioning Tests. Bombs which are loaded with service chges and equipped with std fuzes are released for impact on land & water using the full range of fuze settings. This requirement applies to all bombs, except Cluster Bombs & Practice Bombs
Refs: 1) Hayes (1938), 612–13 2) Ohart (1946), 208–11 3) Anon, "Ordnance Proof Manual", AberdeenProvingGround, Maryland, Vol 1, Nos 9–11(1949) & Nos 10–86(1957)

DROP TESTS. There are many tests which may be called drop tests. Some of them are conducted by dropping the object to be tested onto a hard surface as for testing bombs, described under Drop Safety of Bomb Test. Testing of projectiles, rockets and of explosives in packing containers is also conducted by dropping them on a hard surface (See below). On the contrary, testing of loose, cast or pressed expls to sensitivity to shock (or impact) is conducted by dropping on them various weights from various heights. These methods, known as *Impact Sensitivity, Shock Sensitivity, Drop Weight* or *Falling Weight Tests,* are briefly described in Vol 1 of Encycl, p XVII, under PHYSICAL TESTS. The item includes 17 refs, but the Ref listed here as No 2 was not included and also there was not included the following test for sensitivity of *primers:*

The drop-weight apparatus is represented in Fig 14 of Ref 3. The test is conducted by dropping a ball of known weight, from specific distances, on the firing pin of a test fixture in which the primer is supported. For many years it has been customary to require that sample primers from a given lot fire at a certain height, and fail to fire at a certain lesser height, this last requirement supposedly insuring against accidental firing in handling and transportation

It has been recently shown that more satisfactory results are obtd if a given number of primers are fired at heights betw the all-fire and all-misfire heights, and the percentage firing at each height recorded. The average height and standard deviations are then computed and the satisfactoriness of the lot is detd by comparing with standards established from a consideration of the quality level it is desired to maintain. This modification of drop test is known as *run-down test* (Ref 3, p 48)

A test by dropping a loaded (but unfuzed) projectile, rocket, JATO or separate fuze from predetermined heights onto a hard surface is usually conducted to determine if the proj can withstand, without damage, the *setback force.* Drop test is the nearest substitute for more expensive and time consuming tests of actual firing the proj and is considered to be satisfactory (Ref 3, pp 96 & 103)

Setback force is the rearward force of inertia which is created by the forward acceleration of a proj during its launching phase. The force is directly proportional to the acceleration and mass of the parts being accelerated (Ref 5, p257)

In the drop test there is actually determined the minimum height of drop that will damage the item under test to a degree which will cause the round to fail to function in accordance with design requirements. The procedure as conducted at Picatinny Arsenal consists of suspending the item on a cable of the so-called *Drop Test Tower* (See Fig 53, p 134 of Ref 3) and raising it (on a crane, or chain block hoist) to a predetermined height (such as 50ft for 37 to 155mm projs, or 40ft for a rocket). By means of a special mechanism, the item is released so that it can drop onto a hard surface base (such as reinforced concrete or steel). After this, the item is examined for deformation in metal parts. It is disassembled to determine if the contents are damaged. The item may also be examined by X-rays (Ref 3, p 134 & Ref 4, p 3)

The requirement for satisfactory *packing* of ammo or expls is that it protects the contents until the time comes for its use, no matter what the handling, storage, and shipping conditions may have been to that point. One of the most important requirements for a packing container is that it should not break when dropped. This may be detd by using the *drop test,* which consists of dropping a packing (either loaded with ammunition or explosive, or inert loaded) from a height of 4ft on a steel plate supported by a concrete column. It is usually repeated four times, and if the packing is in the shape of a box, it is dropped so that it impacts on a diagonal and is stressed the worst way. In addition, if containers might be damaged by a fall on the side, or if their contents would be damaged by such a fall, the packing is dropped so that its side strikes the narrow edge of a 2 by 4 inch piece of soft wood (Ref 3, pp 380–81)

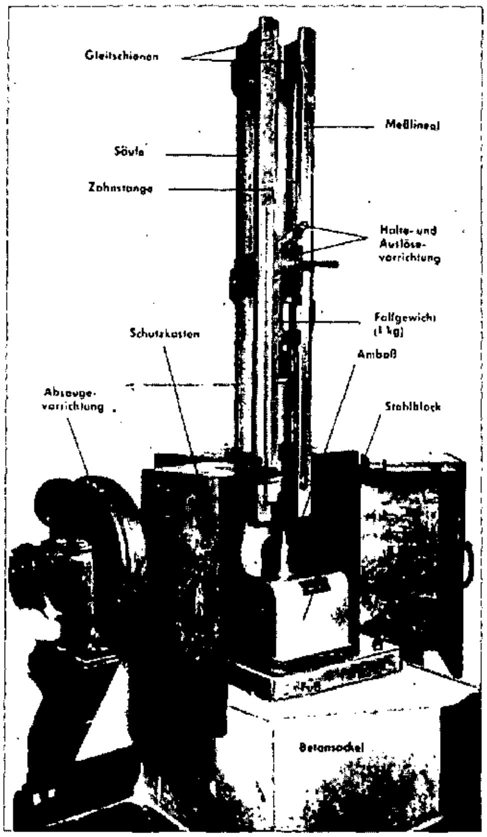
This drop test is sometimes run in conjunction with the *rotating-drum test,* which may also be considered as a kind of drop test. In this test a drum 14ft in diam, which carries inside plates or baffles, rotates at 1 rpm. A packing item, such as a box, is placed inside the drum and, as the drum rotates, the item is raised by baffles and then dropped to the bottom of the drum. After each series of 24 falls, the item is removed and subjected to one fall from a height of 4ft in the drop-test apparatus. These procedures are repeated, until a total of 96 falls in rotating drum and 4 falls in drop-test app are completed. (Ref 3, p 380–81; the drum is shown in Fig 225, p 381)

Hayes (Ref 1, pp 137–38) describes under Impact Tests devices for testing tensile strength (toughness) of metals or plastic materials under conditions of impact or shock. These devices may be subdivided into:
a) Machines for breaking a given specimen at a single blow and b) Repeated-impact machines for delivering a number of blows before fracturing the specimen

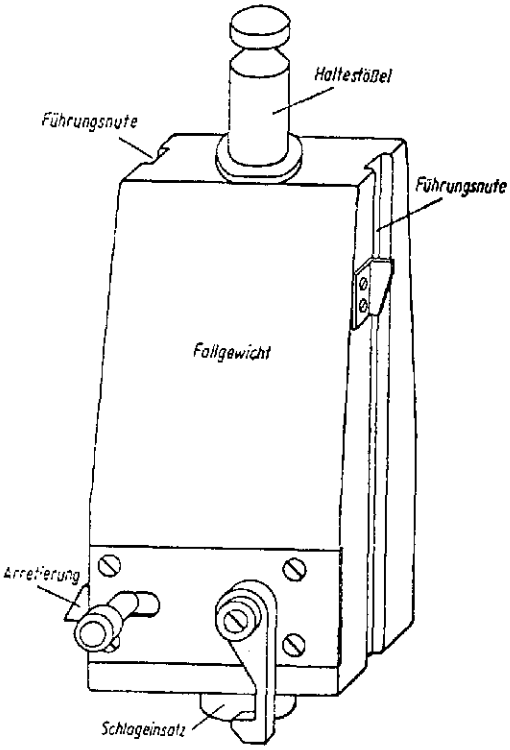
To the first class belong the *Charpy* and *Izod* machines, both used in ordnance work. These machines are briefly described in Vol 2 of Encycl, p C154-L. A photograph of the Charpy machine is shown in Fig 50, p 138 of Ref 1)

To the second class belong the Cambridge Drop Hammer, the Eden-Foster and the Vibratory Spring Testers (Ref 1, p 138)

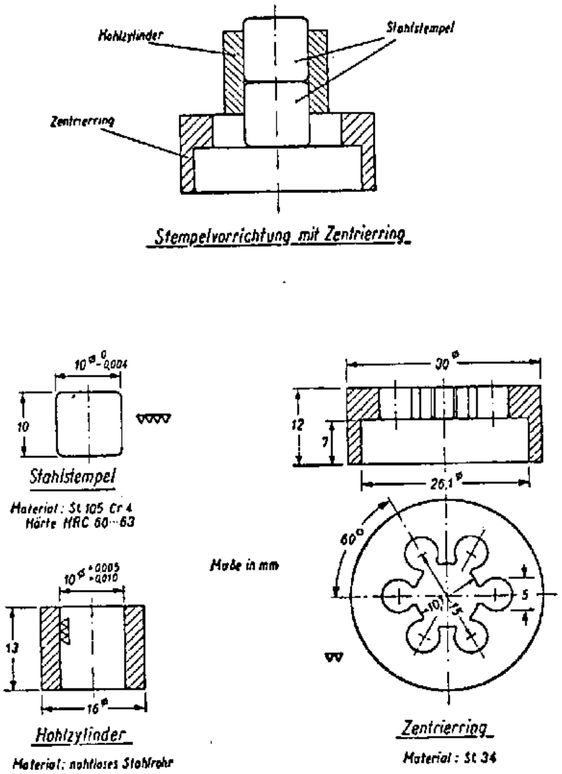
In Germany the sensitivity to impact of solid, liquid & paste expls is detd according to the drop hammer method of the Bundesanstalt für Materialprüfung (BAM) Berlin-Dahlem. This method, which uses a die device, with either a large or small drop hammer, is described by Koenen et al (Ref 7). The large drop-weight app of BAM consists of a cast steel block (200 x 250 x 200mm) with a foot (400 x 400 x 50mm), and a steel anvil (100mm diam x 70mm) which is attached to a column made of seamless steel tubing. The tubing has affixed to it two guides with a toothed rachet to arrest the recoiling drop wt, as well as



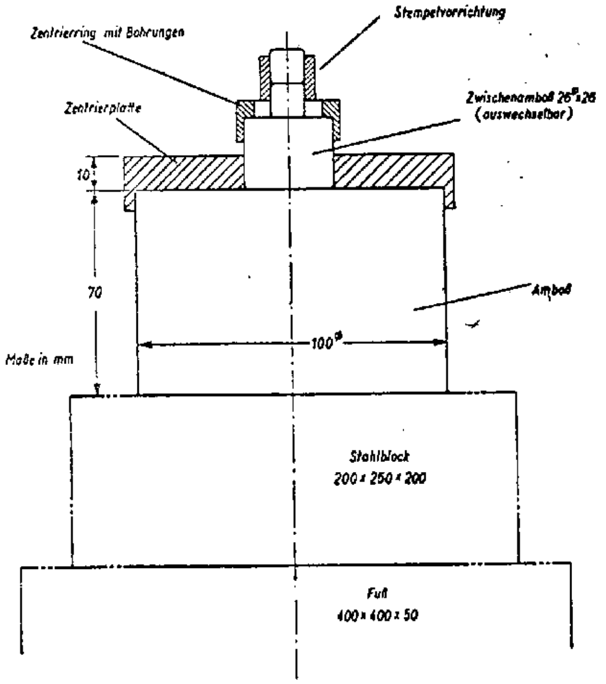
Large drop hammer of BAM with protective box and ventilating equipment used for determining the sensitivity to impact of explosible substances



5 kg drop weight for the large drop hammer of BAM



THE DEVICE WITH CENTRING RING AND DETAILS



ARRANGEMENT OF THE DIE DEVICE UNDER THE LARGE BAM DROP WEIGHT APPARATUS

an adjustable meter stick. The drop weight is held absolutely vertical by guide rails. Protection from any possible splintering is provided by an easily opened wooden box having an internal layer of 2-mm Pb sheets. In order to remove expln gases & dust during testing, a suction device is attached. The smaller drop-weight app is constructed on the same principle but is intended for use with a smaller drop weight

In conducting the test, the expl (dry & sieved, if solid, except pastes) is placed in the die device consisting of two coaxial steel cylinders, a hollow steel cylinder guiding ring, and a centering ring. The device, with test sample carefully pressed with index finger between the steel cylinders, is placed centrally on the anvil and the drop wt is released from a first previously agreed height. The surfaces of the steel die are used for only one impact test. If an expln occurs, both the steel die & hollow cylinder are replaced. Weights of 1, 5 & 10kg are used for the large drop hammer, whereas weights of 100 & 25g are used for the small drop hammer. Falling heights for the 1 kg weight are 10–50cm, 5kg wt 15–60cm and the 10kg wt 35–50cm. For the small drop hammer, impact energies are less than 0.1kgm. The test is conducted six times at each height of fall

In reporting tests results the sample is noted as "no reaction", "decomposition" (w/o expln) and "explosion". The drop weight in kg and the lowest height in cm (calculated as impact energy in kgm) are given as a measure of the sensitivity to impact, if in six tests at least one expln occurs. The sensitivity to impact of a substance is greater the smaller the impact energy is in kgm (Ref 7)

Much more info on drop tests will be found in the Refs and Addnl Refs cited below

Refs: 1) Hayes (1938), 137–38 2) E.H. Eyster & R.F. Davis, OSRD Rept **5744**(1945) (One type of Drop-Weight Impact Machine developed in the US during WWII) 3) Ohart (1946), 48–9, 96, 131–32, 210 & 380–81 4) Anon, "Picatinny Arsenal Testing Manual", **No 2–2** (1951), p 3 5) Glossary of Ord (1959), 257 (Setback force) 6) F.D. Altman, "Drop Test of Large Castings of Experimental Explosive PAX–5", Naval-ProvingGround, Rept No **1665** (1959) (Conf) (Not used as a source of info) 7) H. Koenen et al, "Safety Characteristic Data of Explosive Substances, Part I. Methods of the Bundesanstalt für Materialprüfung (BAM)", Berlin-Dahlem, Explosivst **9**, 4–13, 30–42 (1961) 8) A. Maček, "Sensitivity of Explosives", ChemRevs **62**, 58–60 (1962) (US Naval Ord Lab Impact Test)

Addnl Refs on Drop Tests:

A) J.J. Fox et al, "Sensitiveness of High Explosives", OSRD Rept **1288**(Mar 1943)

B) Hercules Powder Co, "Sensitiveness of High Explosives", OSRD Rept **3991**(Aug 1944)

C) Hercules Powder Co, "Sensitiveness of High Explosives", OSRD Rept **4962**(April 1945)

D) E.H. Eyster & R.F. Davis, "Physical Testing of Explosives; Part II, Sensitivity Studies with the Drop-Weight Impact Machine", OSRD Rept **5744** (Dec 1945)

E) J.M.Downard et al, "Sensitiveness of High Explosives", OSRD Rept **5632**(Jan 1946)

F) J.M. Downard, "Sensitiveness of High Explosives", OSRD Rept **6627**(Feb 1946)

G) J.M. Downard & R.W. Lawrence, "Sensitiveness of High Explosives", OSRD Rept **6629** (Mar 1946)

H) NDRC Summary Tech Rept of Div 8, Vol **1** (1946), 38–41 (Sensitiveness & drop-weight tests)

I) E.H. Eyster & L.C. Smith, "Studies of the ERL Type 12 Drop-Weight Impact Machine at NOL", NOL Memo **10003** (1949)

J) O.E. Sheffield, "Application of the Impact Test to the Study of Cast and Pressed Explosives", **PAMR–24** (1952)

K) P.E. Braid & R.C. Langille, "A New Type of Impact Machine for Evaluating Sensitiveness of Explosives", CanJTech **33**, 28 (1955)

L) R.L. Wagner & M.E. Pollack, "Development of an Impact Sensitivity Test for Cast and Pressed Explosives", PATR **2290** (1956)

M) A. Bulfinch, "Improved Methods and Techniques of Testing Impact Sensitivity of Explosives", PATR **2282** (1956)

N) P.W. Levy, "The Impact Testing of Explosives", Nature **182**, 37–39 (1958)

O) R.C. Kopituk, "A New Impact Detonability Tester for Evaluating Materials with Highly Reactive Oxidizers", Reaction Motor Div, Thiokol Chem Corp, Denville, NJ. Paper **1707–61** presented at the American Rocket Society Conference on Propellants, Combustion and Liquid Rockets, Palm Beach, Fla (26–28 April 1961)

P) L.E. Starr et al, "NOL Drop Test", Naval Ord Lab, White Oak, Md, **TR 62–150** (June 1962)

Q) H. Koenen & H.H. Ide, "New Testing Methods for Explosive Substances", GB Ministry of Aviation **TIL T–5194**(Oct 1962)

R) M. Kusakabe & N. Ishikawa, Kôgyo-KayakoKyokaishi **24**, No 6, 12–23 (1963) [Engl translation Picatinny Arsenal Tech Memo **TM–1734** (Jan 1966)]

S) C. Boyars, Western States Sect – Combstn Inst Papers **WSCI 65–27** (1965) & CA **64**, 4852 (1966) (A review of drop-weight sensitivity testing of expls, with 38 refs)

T) A.J. Clear, "Standard Laboratory Procedures for Determining Sensitivity, Brisance, and Stability of Explosives", PATR **3278**

(Dec 1965), pp 2–7, 32–37 (Impact test procedures & apparatus)

U) W.E. Voreck & E.W. Dalrymple, "Development of an Improved Stab Sensitivity Test and Factors Affecting Stab Sensitivity of M–55 Detonators", PATR **4263** (June 1972)

Drop Safety Test is, accdg to Ohart (1946), p210, the maximum height of drop for a given bomb on a given surface without causing the bomb to function or detonate. It is desirable that the bomb be drop-safe against the reinforced concrete "hard surface" at Aberdeen Proving Ground (which consists of 2 ft of concrete with suitable ballast) when dropped from a fairly high distance, say 2000 ft

Drop Safety Test for Fuzes. See under Fuze and Fuze Components, Environmental & Performance Tests for, in Vol 6 of Encycl

Drop Test for Packing is described in Ohart (1946), pp380–81. There is also in Expls & Pyro Vol 3, No 2 the following:
New Drop Test Fixture. A test tower (patented) is claimed to keep expl hardware reliably in the five attitudes required by the MIL-STD-331 drop test (nose-up, nose-down, horizontal, nose 45° up and nose 45° down). Hardware is kept in position by having fixture fall with it for 32 of the 40 feet
Refs: 1) Atlas/Aerospace, No 8, Dec 1969. Atlas Chemical Industries, Inc., Valley Forge, Pa, 19481 2) E&P **3**(2)(1970), p3

Drop Test Tower. See under DROP TESTS and in Ohart (1946), pp133–34

Drop Weight (or Impact) Tests. See Impact Sensitivity or Shock Sensitivity Test in Vol 1 of Encycl, p XVII; also in Vol 4, pp D321-R and D322

Drozdov (or Drosdov), N.F. (1862–1954). A Rus ballistician, former professor of Mikhailovsky Artillery Academy at StPetersburgh. He gave in 1903 a complete vigorous integration of basic equations of Interior Ballistics without any simplifying assumptions and in 1920 he published tables for practical solutions of various ballistic problems, especially of those required for the design of guns. In later years he published many other papers on ballistics and among them (in 1947) on the characteristics of the so-called "Optimum Gun", briefly described in Ref 1, p446, Ref 2, p556 and also below:
Optimum Gun, called in Rus: "Orudiye Naibol'shego Mogushchestva" (Gun of Maximum Power). Accdg to Drozdov such a gun must possess at a given length of the barrel and a maximum pressure, maximum barrel energy equal to $(mv_{\delta}^{2})/2$. Here m is mass of the projectile and v_{δ} its starting velocity in the barrel. Accdg to Prof Serebryakov, Drozdov's formula has advantage over the formula of French ballisticians: $(\psi m v_{\delta}^{2})/2$, which expresses maximum total work. Here ψ is relative part of consumed proplnt
Refs: 1) M.E. Serebryakov "Vnutrennyaya Ballistika" (Interior Ballistics), Oboronghiz, Moscow (1949). Abbreviated English Translation by Dr V.A. Nekrassoff, Contract NOrd **10260**(1954), Washington, DC, pp32, 38–9 & 446 of Engl Transln 2) M.E. Serebryakov, "Vnytrennyaya Ballistika Stvol'nykh System i Porokhovykh Raket" (Interior Ballistics of Gun Barrel Systems and of Solid Propellant Rockets", Oboronghiz, Moscow (1962), pp 19, 23 & 566

DRPC 02 (Dünneberg Röhrenpulver für Canonen, 1902). A tubular proplnt similar to the British **CSP2**, described in Vol 3 of Encycl, p C571-R. It was manufd since 1902 by the Vereiniate Köln-Rottweiler Fabriken, Dünneberg a/d Elbe for the use of German Navy. It was used also in Brazil
Ref: Admiral Alvaro-Alberto; private communication, Rio de Janeiro, Oct 14, 1958

Drum Camera. See item D, p C14-L in Vol 2 of Encycl, under CAMERAS, HIGH-SPEED PHOTOGRAPHIC

Drums for Blending (Mixing) of Explosives and Their Components. See Drums, Rotary (Rotating) for Blending (Mixing) of Explosives and Their Components

Drums for Drying. See under Drying and Dryers (Driers)

Drums for Filtering. See Drums, Rotary (Rotating) for Filtering

Drums, Flaker. See Drums, Rotary (Rotating) for Flaking

Drums for Glazing Grains of Explosives and Propellants. See Drums, Rotary (Rotating) for Glazing Grains of Explosives and Propellants

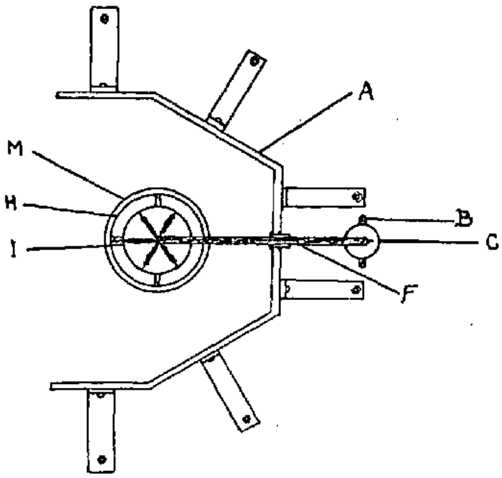
Drums for Mixing Very Sensitive Explosives. See under Drums, Rotary (Rotating) for Blending (Mixing) of Explosives and Their Components

Drums, Rotary (Rotating) for Blending (Mixing) of Explosives and Their Components.

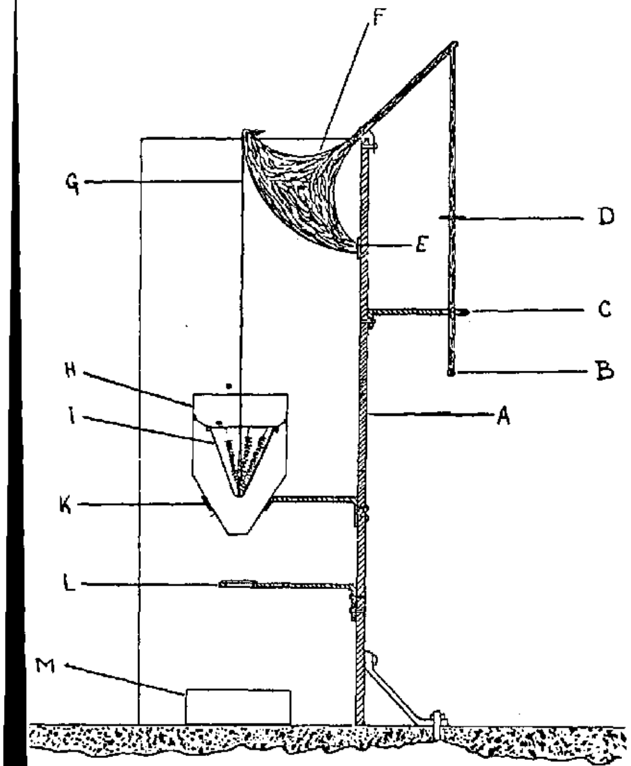
Sensitive ingredients of explosive compositions such as of primers can be blended either by "wet" method such as described by Marshall (Ref 1, p514) or by "dry" process, such as by means of rotary drums, described by Marshall (Ref 1, p515) and Colver (Ref 2, p522). Each drum was of papier-mâché, 6 by 6 inches, varnished inside and they all rotated in groups of two within strongly constructed enclosures. Into the first drum of each pair were loaded, previously pulverized, insensitive ingredients of primers or detonators together with soft rubber balls (to assist mixing). After rotating the drum, by an operator placed behind a barricade, the contents were emptied out thru a funnel and a coarse sieve (to retain the rubber balls) into the 2nd drum. After introducing the sensitive ingredients (such as MF, LA, etc), the 2nd drum was rotated from behind a barricade until complete blending of ingredients was achieved. Each charge was ca 250g

It was claimed that the rotary drum method offered a number of advantages over the *jelly bag method* of blending and yielded a more intimate mixture. The "jelly bag method", which originated in France and adopted in GtBritain was conducted in a conical bag of soft material (such as cloth or leather) with an upper (wider) end of the cone being attached to a frame of a thick leather enclosure resembling on the outside a tall Büchner funnel (See Fig 1), but having no perforated disc inside. To the inside of the bag's apex was attached a cord, carried over pulleys to the operator placed behind a barricade after the bag was loaded with components of primers or detonators. In loading the bag (which could hold as much as 2 lbs), the insensitive ingredients were placed first and then the sensitive components like MF or LA, were put in. On pulling the cord the bottom of the bag was raised so that components could roll from one place to the other inside the bag. A movable stop was provided to prevent the cord being pulled too far and thus cause the contents of the bag to be spilled. On releasing the cord the bag was returned to its original position and then the operations were repeated. Sometimes soft rubber discs or balls were added to the contents of the bag in order to make the mixing more thorough. Before the operation a vessel of water was placed under the bag to catch any material which could be accidentally spilled. After completing the mixing (which took about 3 minutes), the operator replaced the water vessel with a thick paper box and retired again behind the barricade. After removing the "movable stop", the operator pulled the cord until the bag turned completely upside down, thus spilling the mixed material thru a Büchner into the paper box

Mechanical power, used to replace manual labor, gave more uniform mixtures. Over the top of funnel a cardboard sheet was placed to make sure that no impurities could fall into the bag



PLAN OF "JELLY BAG" MIXING PLANT

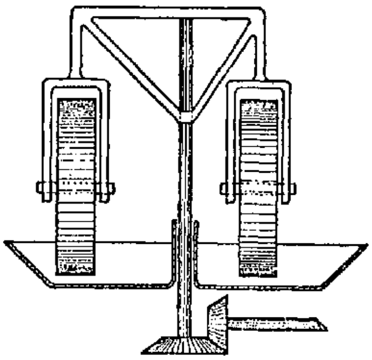


VERTICAL SECTION OF "JELLY BAG" PLANT FOR MIXING FULMINATE COMPOSITIONS

FIG 1

The jelly-bag method was considered as extremely safe, because if even the charge should explode during mixing, the operator was well protected (Refs 1, 2 & 3)

Mixing of insensitive ingredients of explosives also can be done in rotary drums, but if the components are not previously pulverized, the so-called *pan mixers* also known as *wheel mills* or *edge runners* are used. Riegel (Ref 6, p249) calls the device *pan mixers* or *mullers.* Such devices are used for mixing ingredients of *dynamite dopes* and in manuf of BkPdr. Fig 2 is the copy of Fig given on p20 of Barnett (Ref 3). These wheels are also described under "incorporating or milling" of BkPdr in the books of Marshall (Ref 1, p76) and Davis (Ref 5, p46). The process of manuf of BkPdr is also briefly described in Vol 2 of Encycl, p B168, but no Fig is included



GUNPOWDER INCORPORATING MACHINE

FIG 2

The modern "wheel mill", such as described by Marshall, Barnett, Davis under manuf of BkPdr, consists of stone runners acting on a stone bed or of iron runners on an iron or a hardwood bed. The runners used to weigh 4 to 8 tons in GtBritain and 8 to 10 tons in the USA. They are set at different distances from driving shaft so that they do not run on the same path. Each runner is provided with a scraper to prevent its picking up cake, and with a phosphor bronze or hardwood plough to push the charge into its path. In some types of mills, the runners do not rest on the bed, but are suspended a short distance above it, and each is capable of an independent vertical movement, so as to allow it to pass over any extra hard lumps without undue friction. Water is added from time to time so as to maintain a moisture content of 3–6%. Usual charges were 80 lbs in GtBritain, while in Canada and the USA, they milled up to 650 lbs (Ref 4, p13). In the wheel mill, described by Davis (Ref 5, p46), the charge was 300 lbs and the wheels, each weighing 10 tons, rotated for about 3 hrs at a rate of about 10 turns per minute (See also Ref 6, p249 and Ref 7, p1214)

Another type of mixer is known as *mixing rolls.* It consists of two cylindrical rolls turning in opposite directions (and sometimes at different speeds) between which the materials to be mixed are passed. A kneading, tearing, stretching, folding, and shearing action is produced. This type of mixer is shown in Fig 199, p247 of Ref 6 and Fig 39, p1214 of Ref 7. It is used for blending the components of double-base and triple-base components and for rolling the resulting blends into sheets of desired thicknesses

Still another mixing device can be used in explosives industry. It is the *tumbling mixer* which, accdg to Perry (Ref 7, p1212), exists in three varieties: a) "tumbling barrel" or "ball mill" (Ref 7, Fig 32); b) "double-cone mixer" (Fig 33) and c) "mushroom mixer" (Fig 34), which is reproduced here as Fig 3. This mixer is called "sweetie barrel" by Davis (Ref 5, p290 and is shown as Fig 71 on p291)

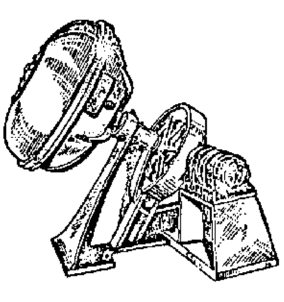


FIG 3 – MUSHROOM MIXER

Refs: 1) Marshall **1** (1917), 76; Marshall **2** (1917), 514–15 2) Colver (1918), 521–22 3) Barnett (1919), 19–21 4) Marshall **3** (1932), 13–14 & 160 5) Davis (1943), 46 & 291 6) Riegel, ChemMach (1948), 234–52 and (1953) 7) Perry (1950), 1195–1231 and (1963), pp 19–3ff 8) Kirk & Othmer, 2nd edit **13**(1967), pp577–613

Drums, Rotary (Rotating) for Drying. See under Drying and Dryers (Driers)

Drums, Rotary (Rotating) for Filtering. Many rotary drum filters are described in the books of Riegel (Ref 1) and Perry (Ref 2). Following are examples of such filters:

Continuous-Suction (Vacuum) Filters with Rotating Drum. The earliest and probably the best known filter of this type is the *Oliver.* They are described in Ref 1, pp 283–87 and Ref 2, pp976–78 and shown in Figs 75 & 76 on p976 of Ref 2 and here as Figs 4 & 5. Essentially they consist of a cylindrical drum lying on its side and rotating about its axis. The cylinder is mounted on a horizontal shaft, set on a slurry tank, in which the drum dips to varying depths. The surface of the cylinder is made up of compartments, which are shallow, and terminate in a pipe first running radially, then turning along the shaft to reach the stationary part of automatic valve. Each compartment is so connected. The compartment offers numerous points of support to the filter cloth. As a drum compartment enters the slurry, the automatic valve connects the suction line to it, and filtrate is drawn in; then it is pulled to a separator and receiver and the cake is deposited against the surface. This continues while the compartment is submerged; the suction remains connected for a short distance after it emerges, in order that all adhering filtrate may be sucked in. A spray of water meets the compartment as it slowly rises away from the tank. Suction is again applied by the automatic valve; the wash water is sucked in, and pulled away thru a second line to a second separator, receiver and pump. The cake now approaches the dis-

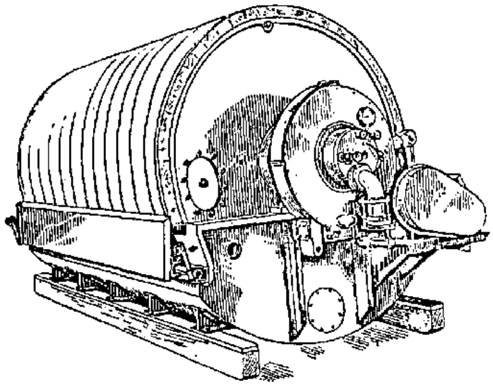


FIG 4 – OLIVER FILTER
(DISCHARGE SIDE)

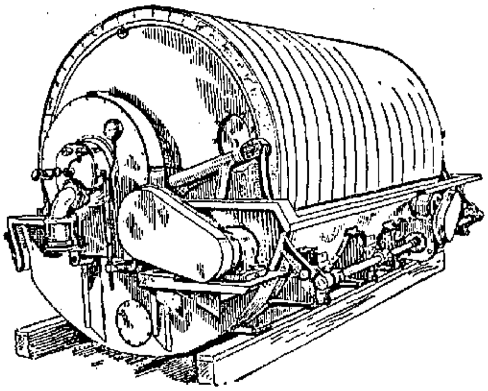


FIG 5 – OLIVER FILTER
(AGITATOR SIDE)

charge knife; a pressure line brings a positive-pressure air blow which bulges the filter cloth outward, loosening the cake, so that when it reaches the knife, it is easily scraped off. The rotary-drum suction filter is possible only because of the automatic valve, which places a compartment under suction alternately for filtrate, for wash, and blowback for bulge. The valve is described on p284 and shown in Fig 227 of Ref 1. A detailed cross section of Oliver filter is given in Fig 77, p977 of Ref 2, which includes a table with a complete list of parts

Other types of Oliver filters include the top-feed filter (Ref 2, p980, Fig 82) and its modification by Robinson (Ref 2, p980)

Other examples of rotary-drum vacuum filters are:

Dorrco Continuous Suction Filter has, in contrast to Oliver, its filtering surface on the inside of the drum, and the suction line on the outside. It is described by Riegel (Ref 1, p287–88; Fig 232) and by Perry (Ref 2, p979; Fig 81)

American Continuous Suction Filter consists of a number of filter discs mounted around a hollow cast-iron center shaft, as illustrated by Fig 233, p289 of Ref 1 and Fig 83, p981 of Ref 2

Bird-Young Continuous Suction Filter is designed for slow-filtering slurries because of its ability to operate with very thin cakes. A detailed description is given in Perry (Ref 2, p983) and its cross section is in Fig 84c

Refs: 1) Riegel, ChemMach (1944), 282–91
2) Perry (1950), 976–83; Perry (1963), 19–76
3) Kirk & Othmer, 2nd edit 9(1966), 264–68 (Filtration)

Drums, Rotary (Rotating) for Flaking. Under this term are known devices in the form of rotary drums for prepg various substances in the form of small scales (flakes). Such drums, known as *flakers* are used in the manuf of TNT. Flaking can also be done by using water-cooled belts, such as in the Stengel process for making flaked Ammonium Nitrate. This process is briefly described in this Encycl, Vol 1, p A315-R. On the same page are described two other processes for manufg AN – the *prilling* and *crystallization* methods

In the case of TNT, crystallization was used in the USA prior to WWII, but during the war this method was practically replaced by flaking on rotary drums. It must be noted that "flakers", such as now used, function also as "driers"

A typical "flaker", such as used in manuf of TNT and some other HE's consists of a horizontal, hollow, metallic (usually cast-iron), water-cooled, drum, with hollow trunions mounted in bearings. It is given in Perry, p1164-L as Fig 68 and reproduced here as Fig 6. The lower part of drum dips into molten material located in a hollow, steam-

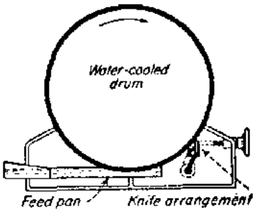


FIG 6 – TYPICAL DRUM FLAKER

heated pan. The moment the material touches the water-cooled drum, a thin film is formed on the surface of the drum. As the drum rotates, the film hardens on cooling and when this brittle film reaches the knife (known as the "doctor"), held firmly by means of a spring to the surface of the drum, it is scraped off in the form of flakes (scales). The reduction to the flake size by chipping action of the knife results in grains which are not very regular

For products which do not adhere readily to cold surfaces, or which drop very quickly before they reach the knife, a double-drum flaker is used, such as shown in Fig 69, p1164-R of Perry and reproduced here as Fig 7. Here the drums are placed close together and they rotate towards each other, as shown by arrows. The liquid material is fed between the drums, being prevented from flowing out at the ends of the drums by end plates, held against the heads. With the drums turning as shown in Fig, the liquid passes thru a narrow space between them and then gradually solidifies. The thickness of film is determined by the clearance between the drums. When solidified film reaches knives, it is scraped off as described under single-drum flaker

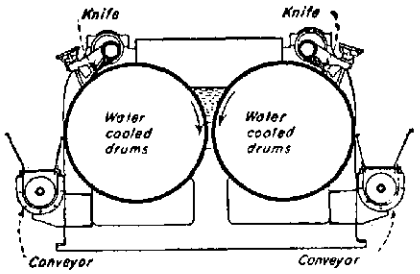


FIG 7 – DOUBLE-DRUM FLAKER

Some devices which produce flaked material are listed by Perry as "driers", because their principal purpose is to dry the materials. They resemble in appearance the flakers, but their hollow drums are heated. Some of these devices are described under "Drying and Driers"

Flakers are also described by Riegel (Ref 1, pp347–50) and a rotary, single-drum flaker is shown in Fig 271, p348

Refs: 1) Riegel (1944), 347–50 2) Perry (1950), 1164–66 3) Kirk & Othmer, not found

Drums, Rotary (Rotating) for Testing Packing of Ammunition. In the book of Ohart (1946), pp980–81, is briefly described a rotating (revolving) drum used to provide a controlled rough-handling test, which has long been used for all types of military and commercial packing. In this test, a horizontal drum, 14 ft in diameter, rotates at 1 rpm. On the inside of the drum are plates or baffles that serve to carry a box or other packing item up a certain distance before the packing falls over the baffle and lands inside the drum at its bottom. There are 6 falls per revolution of the drum. The usual test consists of 96 falls, but after each series of 24 falls, the packing is removed from the drum and subjected to one fall from a height of 4 ft on a steel plate supported by a concrete base. Photographic view of the testing equipment is shown in Fig 225, p381 of Ohart's book

Drying Agents. See under Dehydrating and Drying Agents in Vol 3 of Encycl, p D43

Drying. This term refers to the removal of volatile materials from liquids or solids. In case of removal of moisture, the term *dehydration* may be used. Most common methods of drying are heating and passing dry preheated air (or other gas) thru the substance or over it. Drying by freezing and drying by sublimation are also types of thermal drying (Refs 10 & 14). Non-thermal methods of drying include: filtration, centrifuging, decantation and drying by means of drying agents

Drying is a very common operation in explosives laboratories and plants. All explosives must be carefully dried before loading them into ammunition because wet materials are less powerful, less brisant and less sensitive to initiation. There are, however, expls and proplnts which are too sensitive for use in projectiles, unless they contain some moisture. For example, Guncotton, formerly (end of 19th century) used for press-loading Amer and Rus shells and torpedoes contained ca 18% of moisture

There are several methods for drying explosives

For laboratory drying a crystalline or powdered sample of expl is spread in a thin layer on a sheet of filter paper and left overnight in a warm room. The next day, the sample is placed in a vacuum oven at ca 55°C with 18–25 inches of vacuum and kept there until constant wt (2 or more hrs). Expls can also be dried by the methods for drying NC described in detail in Vol 3 of Encycl, p D45-L

For drying of expls at the plant, Olsen patented (Ref 7) the following method: Grains of an expl, deposited in a layer 1/8 to 1/2 inch thick on a belt, travel for about 95 mins thru a radiation chamber, ca 30 ft long, maintained at 65–75°C. Heating is produced by radiation which is nearly infra-red

Explosives which are produced in flaked form (like TNT) lose moisture during the process of flaking (See under "Drum, Flaker")

Methods used for drying expls in Germany during WWII were described by Glücklich (Ref 6). Holland-Merten (Ref 15) described vacuum drying techniques used in explosives industry

Drying of smokeless propellants was described in Vol 3, p D47

Davis (Ref 4a) described solvent recovery and drying of single-base propellants as conducted during WWII at the E.I. duPont de Nemours and Company plants:

The colloided NC proplnt, known after graining and cutting as *green powder*, was put into a closed system and warm air (at 55–65°) was circulated thru it in order to pick up volatile solvents (alcohol & ether). This air went then thru a condenser where most of the volatile solvent was removed. The stripped-of-solvent air was reheated and passed thru a fresh portion of proplnt

The above operation did not remove solvent to the extent of rendering proplnt suitable for loading in ammunition. For a more complete removal of volatile solvent, the process known as *drying* is used. This can be done either by *air-drying* or by *water-drying*. The "rapid water-drying process" has the advantage because the formation of a "skin" of colloided NC upon the surface of grains is prevented. In this method, water at 65° is circulated thruout the proplnt. The water causes the production of microscopic cracks and pores thru which the residual solvent escapes more freely and is dissolved in water. Circulation of water is continued until the solvent content of proplnt is sufficiently reduced. Then the grains of proplnt are transferred to another building, where they are dried by a continuous method at 55–65°. The finished proplnt usually contains 3.0 to 7.5% of volatile solvent in the interior of the grain (the amount depending upon the thickness of the web) and 0.9 to 1.4% on the exterior of the grains (mostly in cracks and pores of the surface). The amount of moisture which the proplnt thus holds upon its surface is an important factor in maintaining its ballistic stability under varying atmospheric conditions. The amt ought to be such that there is no great tendency for the moisture to evaporate off in dry weather, and such also that there is no great tendency for the proplnt to pick up moisture in damp weather

Note: The importance of surface moisture is so considerable that in France, long before the "hot-water drying process" was invented proplnts were submitted to a *trempage* (or tempering) by immersion in water for several days at temperatures as high as 80°C

Mathematical study of drying process was conducted in France by P. Arribat and published in MP **29**, 12–49 (1939), under the title: "Étude Analytique du Séchage"

In the *rotary drum driers*, a liquid material, a solution, slurry, or paste is transferred in the form of a film to the surface of a revolving metal drum heated inside by means of steam. The heat conducted to the film evaporates the water (or other volatile substance) during partial revolution of the drum and then the dry material is scraped from the drum by a stationary knife

Drum driers can operate either at atmospheric pressure or under vacuum

The simplest type of drum drier contains only one drum and is known as:

Atmospheric Single Drum Drier with Dip Feed. It is identical in appearance with a "rotary single drum flaker", shown here in Fig 6, except that the drum is heated instead of being cooled by water as in the case of flaker. This type of drier is described by Riegel (Ref 10, p362) and shown in Fig 276

When two drums rotate around parallel horizontal axes they are known as *double drums* (rotation toward each other) or as *twin-drums* (rotation in opposite directions from each other). Following are examples:

Atmospheric Double-Drum Drier with Pipe Feed is described in Perry, p863-R and shown in Fig 62 (See our Fig 8). A similar apparatus is described in Riegel, p364, Fig 277 under the name of "atmospheric double-drum drier with center feed"

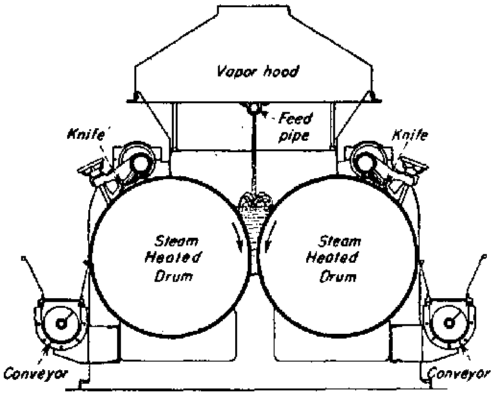


FIG 8 – DOUBLE-DRUM DRYER WITH PIPE FEED
(Buflovak Equipment Div, Blaw-Knox Co)

Atmospheric Twin-Drum Drier with Splash Feed is described in Perry, p864-L and shown in Fig 64, which is reproduced here as Fig 9. A similar apparatus is described in Riegel, p365 and shown in Fig 278

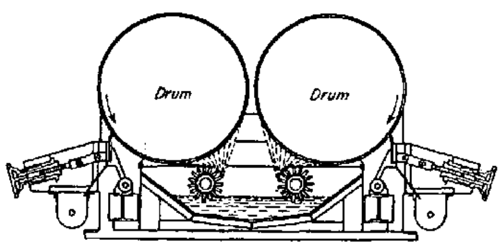


FIG 9 – TWIN-DRUM DRYER WITH SPLASH FEED
(Buflovak Equipment Div, Blaw-Knox Co)

All driers listed above are steam-heated

Vacuum drum driers operate on the same principle as the corresponding atmospheric driers, except that the drums are enclosed in vacuum-tight casing and a vacuum is created either by a pump or a steam jet. They are used in drying materials which are heat-sensitive, easily oxidizable or when it is desired to recover a volatile solvent

Following are examples of vacuum rotary driers:

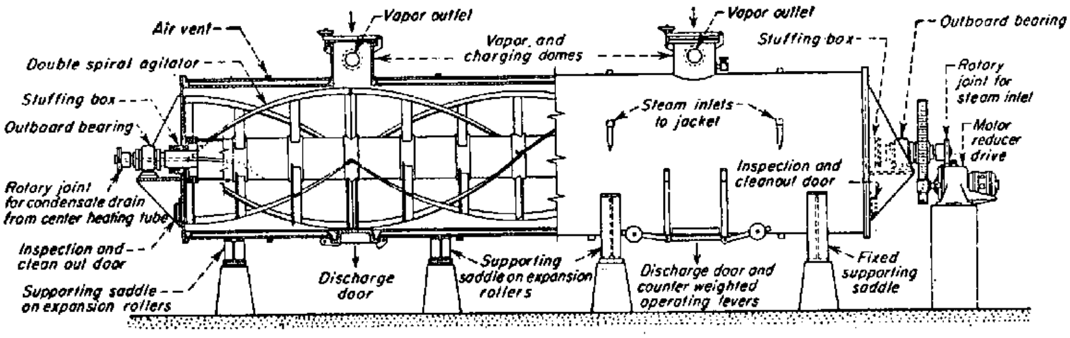
Vacuum Single-Drum Drier of Blaw-Knox Co. is described by Perry on p858 and shown in Fig 57. It is reproduced here as Fig 10

Vacuum Single-Drum Drier of Blaw-Knox Co with Patented Spray Film Feed is described in Perry, p864-R and shown in Fig 65, which is reproduced here as Fig 11

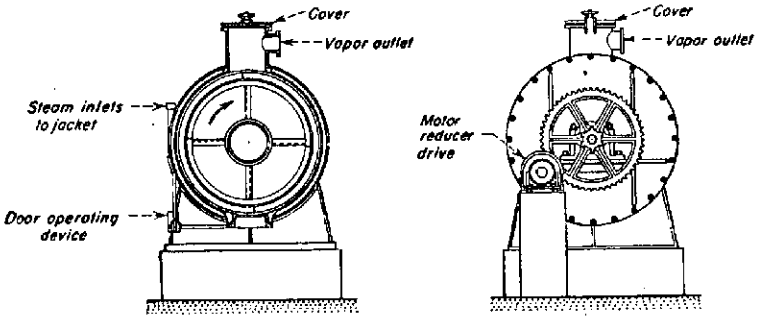
The above described driers consist of closed, short, large diameter drums, which heat and dry on their surfaces the wet materials

The driers described below consist of long, open drums (cylinders), which heat and dry the wet materials fed inside of them

The simplest and commonly used of these driers is the *Single-Shell, Direct-Heat, Revolving Rotary Drier,* such as described by Perry (pp828–29, Fig 27), given here as Fig 12. It consists of a cylinder rotating on suitable bearings and slightly inclined to the horizontal. The length of the cylinder may range from 4 to 10 times its diameter, which may vary from 1 to 10 ft. Wet material fed into one end of the cylinder progresses thru, by virtue of the rotation and slope of the cylinder, and discharges dry from the other end. Heated air or flue gas is passed either counter-current to or parallel with the flow of material



Elevation and partial cross section



Cross sectional view

Drive end view

FIG 10 – A TYPICAL VACUUM ROTARY DRYER
(Blaw-Knox Co)

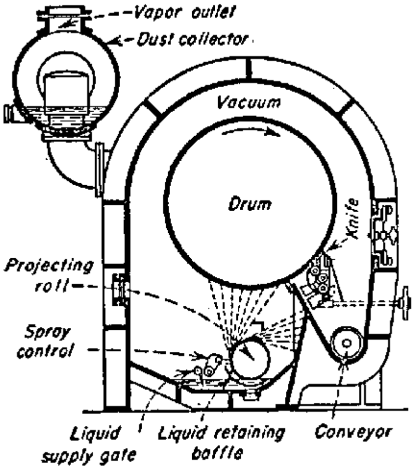


FIG 11 – VACUUM SINGLE-DRUM DRYER SHOWING PATENTED SPRAY FILM FEED
(Buflovak Equipment Div, Blaw-Knox Co)

A similar drier is described by Riegel (p367, Fig 280)

Some special types of rotary driers not described in Perry are described by Smith (Ref 5)

Rotary driers may also be used for combined drying & roasting and calcining. In this case they are known as *rotary kilns*. A typical kiln is briefly described by Perry (p289) and shown in Fig 26, reproduced here as Fig 13

This device contains a refractory-lined cylinder, rotating around its axis. Roasting and calcination are achieved by burning gases entering at one end of the cylinder and moving counter-current to crude material entering at the other end of the cylinder

The rotary-kiln type incinerator mentioned by S. Slemrod of Picatinny Arsenal (Ref 24) for destruction of "red-water" syrup, obtained as waste in US plants manufacturing TNT by the batch method. One of such kilns was in operation at Keystone

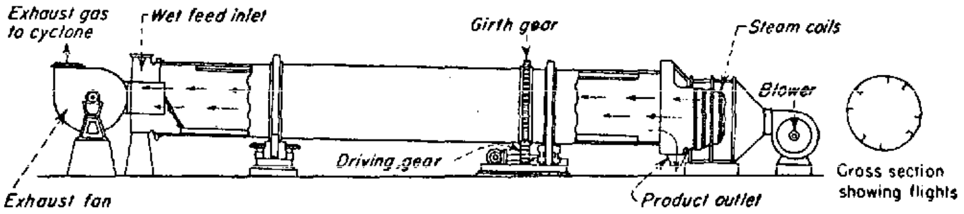


FIG 12 – SINGLE-SHELL, DIRECT ROTARY DRYER USING STEAM-HEATED AIR AND BALANCED PRESSURE BY MEANS OF A BLOWER AND EXHAUSTER
(Hardinge Co)

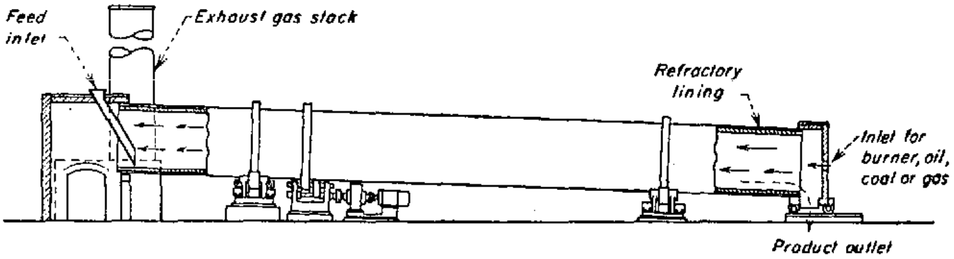


FIG 13 – ROTARY KILN, REFRACTORY-LINED, FOR ROASTING AND CALCINING. IN SPECIAL CASES, DRYING IS ALSO ACCOMPLISHED
(Hardinge Co)

Ordnance Plant, Meadville, Pa, where senior author of this book was chief chemist

The "red water" was obtd as a result of treatment of crude TNT with the soln of Na sulfite, known as *sellite* (See Ref 6, pp 1–2). The resulting red water consisted of aqueous soln of organic nitrogen salts (such as Na dinitrotoluene sulfonate, Na nitrodiazotoluene sulfonate and unattacked alpha TNT) and inorganic salts (such as Na sulfate and Na nitrate). This liquid was concentrated in vacuum evaporators and the resulting "syrup" was introduced thru a feed-inlet into upper part of preheated rotary kiln similar to that shown in Fig 13. The syrup, first spread on the hot refractory lining, started to run by gravity towards the outlet of the cylinder. The hot furnace gases, moving in opposite direction, heated the syrup and evaporated the water. As the flow of syrup progressed, its consistency thickened to form a dark-red paste, which on further heating dried to a black cake. Finally, an incineration took place with formation of a grayish ash consisting mostly of Na sulfate and small amounts of Na nitrate and carbon. Water vapor and gases (such as carbon and nitrogen oxides) were removed thru the exhaust stack, while the ash fell onto a conveyor and was disposed of by drowning it in water

Rotary kilns used for incinerating solid materials, such as cements, lime, magnesite, etc are described in Perry (Ref 14, pp1608–12 and shown in Fig 8, which is not reproduced here

In addn to heating and vacuum, drying can be done by freezing and sublimation, as described in Ref 14

Although our sources of information for this section on drying were mostly the books of Riegel and Perry, we list several other references for those desiring more detailed information

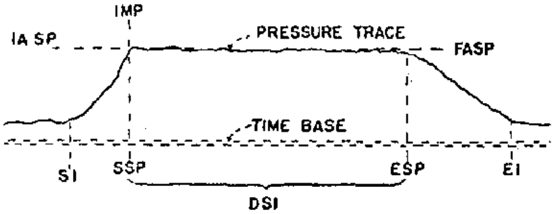
Refs on Drying: 1) Marshall **1** (1917), 289 (Drying of NC); 294 (Drying of proplnts) 2) T.K. Sherwood et al, IEC **21**, pp 12 & 979 (1929) and in succeeding years; series of papers on "Drying of Solids" 3) Marshall **3** (1932), 85 (Drying of NC); 89 (Drying of proplnts) 4) N. Hirsch, "Die Trocken-

technik'', J. Springer, Berlin (1932) 5) B.A. Smith, IEC **30**, 993(1938)(Rotary Dryers) 6) Clift & Fedoroff, Vol **1**(1942), Chap VI, pp 1–2(Purification of TNT by Na sulfite) 7) J. Reilly & W.N. Rae, "Physico-Chemical Methods", Van Nostrand, NY (1943), 193–209(Drying) 8) E. Glücklich, SS **38**, 48–50 & 66–8(1943) (Die künstliche Trocknung von Sprengstoffen) 9) F. Olsen, USP 2349300(1944) & CA **39**, 816(1945) (Drying of expls and proplnts) 10) Riegel, ChemMach (1944), 351–81(Driers) 11) D.B. Broughton, IEC **37**, 1184–85(1945) (Drying of solids) 12) S.J. Friedman, IEC **38**, 22(1946) (Drying) (In "Unit Operation Review") and in succeeding years 13) E.W. Flosdorf, "Freeze-Drying; Drying by Sublimation", Reinhold, NY (1949) 14) Perry (1950), 799-884 (Drying) 15) A. Weissberger, ed, "Physical Methods of Organic Chemistry", Interscience, NY (1950), 605–34(Drying) 16) Kirk & Othmer **5**(1950), 232–65(Drying) 17) R.J.C. Harris, "Freezing and Drying", HafnerPubCo, NY (1952) 18) Dept of SciIndRes, "Bibliography of Industrial Drying", (1952); Reviewed in ChemAge(London) **67**, 196–97(1952) 19) S.P. Artyushin, "Dehydration and Dust Collection in Coal Concentration Plants" (in Russian), Ugletckhizdat, Moscow (1956) 20) M.I. Belin, "Dehydration of the Products of Coal Enrichment" (in Russ), GosNauch-TekhIzdatel, Moscow (1958) 21) E.L. Holland-Merten, "Explosivstoffe, **7**, 1–9 (1959)(Vacuum drying in the explosives industry) 22) Perry (1963), pp 11–45 & 15–32 (Drying of Solids); 15–34 (Mechanisms in Drying) & 17–26 (Freeze Drying) 23) Kirk & Othmer, 2nd edit **7**(1965), pp326–78 (Drying) 24) S. Slemrod, "Producing TNT by Continuous Nitration", Ordnance **54**(1970), p527

DSI. Abbr for "Duration of Sustained Injection" (of a liquid rocket propellant). It is calculated as the difference between ESP & SSP, which terms are illustrated in the Fig, where:

ISAP – Initial Average Sustained Pressure
IMP – Initial Maximum Pressure
FASP – Final Average Sustained Pressure
SI – Start of Injection
SSP – Start of Sustained Pressure
ESP – End of Sustained Pressure
EI – End of Injection

Ref: F. Bellinger et al, IEC **38**, 166–67(1946)



DEFINITION OF VALUES MEASURED
ON FILM STRIP

DSSP. Abbr for "Deep Submergence Systems Project", the objective of which is the development by the US Navy of a deep submarine rescue and salvage capability
Ref: Anon, "The Common Defense", Bulletin No 305 of AOA, Washington, DC, 20006, p2

D-Stoff. German designation for Dimethyl Sulfate, $(C_2H_5)_2SO_4$, a blister gas, listed in Vol 2 of Encycl, p C169-L. It was known in France as "Rationite"

DTA. Abbr for Differential Thermal Analysis(qv)

Du-1 Ammonal. A German formulation: TNT 50, RDX 25 & Al 25%; was used in shells, mines and bombs
Ref: Ye.Yu. Orlova, "Chemistry and Technology of High Explosives", translated from Russian by the Technical Documents Liaison Office, Ohio, Pt 1 (1961), p187

Dualine Stumping Powder (Canad). It was prepd in 1893 in Nanaimo, British Columbia, by incorporating 3 parts of BkPdr (previously pulverized by "wheel mill") with 1 part of NG
Ref: Van Gelder & Schlatter (1927), 706

Dualines. Schultze patented in 1868, under the name of *Dualin*, an explosive consisting of NG, nitrated woodmeal and sawdust. Dittmar of Sweden modified its compn to have NG 50, partially nitrated sawdust 30 and K nitrate 20%. This expl was manufd beginning in 1869 at Charlottenburg, Sweden (Refs 1, 2, 3, 5, 6 & 7). Modified Dualines were manufd in San Francisco, Calif. They contd NG, NC, saltpeter, Ba nitrate and resin (Ref 3)
Refs: 1) Daniel (1902), p208 2) Thorpe **2** (1917), p438 3) VanGelder & Schlatter(1927), p417 4) Naoúm, NG(1928), p282 5) G.D. Clift & B.T. Fedoroff, "A Manual of Explosives Laboratories", Lefax, Phila, Vol **2** (1944), p D13 6) PATR **2510**(1958), p Ger 38-R 7) Giua, Trattato **6**(I)(1959), p343

Dubois Explosives consisted of liquefied acetylene (or ethylene) mixed with an oxidizer, such as nitrogen oxide or peroxide. They were proposed as military expls
Ref: Daniel (1902), 208

Dubois-Raymond Explosives (Fr). Proposed in 1892, mixtures which contained combustible materials such as naphthalene, anthracene, camphor, etc together with oxidants, such as nitrates, chromates, etc. To these was added tar, varnish, lacquer or a drying oil. Some of these expls contained picrates

Following are two examples: *Mixture No 1:* AN 81.4, naphthaline 7.8 and tar 7.8; *Mixture No 2:* K picrate 45, anthracene 45 and tar 10%
Refs: 1) Daniel (1902), p209 2) Giua, Trattato **6**(I)(1959), p373

Dud. An explosive loaded item that fails to explode when subjected to treatment that should cause it to function. A dud can be a bomb, shell, grenade, etc that has been discharged from a weapon, dropped from a plane, or thrown by hand, that failed to explode on reaching the target. This usually happens when the explosive train is broken and firing does not take place in the regular manner. Defective fuze, booster or main charge are the most frequent causes of duds. As soon as a dud is located, it should be (if possible) examined to determine the cause of failure and then destroyed as soon as possible, because it is a source of danger. If left unattended, it might explode by an outside shock or when handled by an inexperienced person
Refs: 1) Anon, "Ammunition Inspection Guide", **TM 9-1904**(1944), p131
2) Anon, "Ordnance Train Designers' Handbook", NOLR **1111**(1952), p G2
3) FranklinInst Newsletter, G. Cohn, Editor, Explosives & Pyrotechnics, Vol 1, No 8 (1968), p2

Duda's Ballistograph. See Ballistograph in Vol 2 of Encycl, p B10-L

Duds Destruction. Destruction can be achieved either by burning (for small items of ammunition) or, preferably, by detonation. As soon as a dud is located, it should be marked with a red flag and reported to personnel trained for destruction of live ammunition. Whenever possible the dud should be destroyed at the spot where it is found. If it is, however, necessary to remove the dud to another location, great care should be exercized not to cause movement of internal parts of a dud (especially of fuze)

In destruction by detonation each dud is placed with its fuze towards, and as near as possible, to the demolition explosive which will be detonated by means of a blasting cap (electric or nonelectric). If a barricade is not available in immediate vicinity, all personnel should be removed to a safe distance, before the demolition charge is fired

A similar procedure for destruction of any undesirable live ammunition can be used, even if it does not consist of duds. Such is the case when it is necessary to destroy captured enemy projectiles, or when it is necessary to destroy projectiles so that they cannot be captured by the enemy
Ref: Anon, "Ammunition Inspection Guide", TM **9-1904**(1944), p771

Duffek, Victor, Dr (1898–1957). An Austrian engineer specializing in metallurgy, who invented during WWII the "Deep Bonding Process" (Tiefbonder Verfahren, in Ger). This process greatly reduced wear in the bore of guns when fired with AP projectiles provided with sintered iron sheaths

A fairly comprehensive description of this process is given in Ref 1, pp Ger 198–99

Dr Duffek worked after WWII at Picatinny Arsenal until he was killed in his car by a train at a grade crossing in Sparta, New Jersey (Ref 2)
Refs: 1) B.T. Fedoroff et al, PATR **2510** (1958) 2) Private Communication from Mrs V. Duffek thru Henry A. Tisch of Picatinny Arsenal, Dover, NJ (Feb 1965)

Dufour's Bolting Cloth. Silk screens of various apertures used for separating grit from smokeless propellant grains
Ref: TransAmerInstMining **19**, 580(1909–1910)

Dugway Proving Ground, located at Tooele, Utah, has been used for large area testing of Chemical Corps toxic agents and other chemical warfare materials
Ref: R. Clinton, ArmedForcesChemJ **5**, No 4, pp 14–19(1952) & CA **46**, 6285(1952)

Duke University, located at Durham, North Carolina was the headquarters for the Army Research Office which prepd the Engineering Design Handbook Series of the Army Materiel Command. This coordinated series of handbooks contains basic information & fundamental data useful in the design & development of Army materiel & systems. They are authoritative reference books of practical information & quantitative facts helpful in meeting the tactical & technical needs of the Armed forces. Information and/or copies of the handbooks may be obtd from Headquarters, US Army Materiel Command, Washington, DC 20315

DUKW. One of the amphibious vehicles used during WWII. Another vehicle was BARC [See Vol 1 of Encycl, p A393-L and also in Ordnance **38**, 529–31(1953)]

DULCITOL AND DERIVATIVES

Dulcitol *(Dulcite, Hexanhexol or Melampyrit)* (Dulcit in Ger), $HO.CH_2(CHOH)_4.CH_2.OH$; mw 182.17, colorless monoclinic crysts, d 1.466 at 15/4°, mp 188.5°, bp – dec; sol in hot w, sl sol in cold w; v sl sol in alc, insol in ether. It occurs naturally in Madagascar manna, from which it can be extracted with water; can also be obtd by treating aqueous sol of lactose or of d-galactose with sodium amalgam. When treated with nitric acid or with mixed nitric-sulfuric acids it forms nitrates of which tetra-, penta- and hexanitrates are explosive
Refs: 1) Beil **1**, 544, (286), [612] & {2405} 2) CondChemDict (1961), p424

Azido, Diazido, Mononitrate, Dinitrate and *Trinitrate* derivs of Dulcitol were not found in the literature thru 1966

Dulcitol Tetranitrate, $C_6H_8(OH)_2(ONO_2)_4$; mw 362.17, N 15.47%; prismatic ndls (from alc), mp 120–30°. It can be prepd by gently heating Dulcitol Hexanitrate (qv) (Refs 1 & 2)
Refs: 1) Beil **1**, 547 2) Béchamp, CR **51**, 257(1860) 3) Not found in later refs thru 1966

Dulcitol Pentanitrate, $C_6H_8(OH)(ONO_2)_5$; mw 407.17, N 17.20%; ndls (from alc + w), mp – sinters at 71° & melts at 75°; has bitter taste; mod sol in alc, eth, acet & benz; sl sol in petr eth; v sl sol in water. It was prepd by gently heating Dulcitol Hexanitrate (qv) in pyridine (Refs 1, 2 & 3)
Refs: 1) Beil **1**, 547 2) J.H. Wigner, Ber **36**, 799(1906) 3) G.G. McKeown & L.D. Hayward, CanJChem **33**, 1392(1955) & CA **51**, 3587(1957) [Report colorless ndls (from aq alc, eth-petr eth or CCl_4), mp 85–86°]

Dulcitol Hexanitrate (Nitrodulcite), $(O_2NO)CH_2.[CH(ONO_2)]_4.CH_2(ONO_2)$ or $C_6H_8(ONO_2)_6$; mw 452.17, N 18.59%, OB to CO_2 +7.1%; wh ndls (from alc), d 1.62 mp 94–95°(Refs 1 & 5); bp 290–95° at

3mm Hg; heated at atm pressure expl at 205° within 1 sec; heat of combustion 670.9 kcal/mol (Refs 2 & 4). It is insol in w and sol in pyridine at 30°. It may be prepd by treating dulcitol with concd mixed nitric-sulfuric acid, as was done in France by Béchamp & Champion (Ref 5, p30)

Dulcitol Hexanitrate, isomeric with Mannitol Hexanitrate and similar to it, is a very powerful and brisant explosive. It is less sensitive to impact than LA (47cm with BurMinesApp and 0.5kg weight vs 32cm for LA), but it is more sensitive than PETN (Refs 2 & 4)

Uses: Might prove suitable in primer mixtures. When heated gently slightly above mp, it is converted into Dulcitol Tetranitrate (Ref 2)

Refs: 1) Beil 1, 547 & [612] 2) C.A. Taylor & W.H. Rinkenbach, JFrankInst **204**, 374 (1927) 3) Davis (1943), 238 4) A.H. Blatt, "Compilation of Data on Organic Explosives", OSRD **2014**(1944) 5) B.T. Fedoroff et al, "A Manual of Explosives Laboratories", Lefax Soc, Phila, Vol **4**(1946), p30 6) G.G. McKeown & L.D. Hayward, CanJChem **33**, 1392(1955) & CA **51**, 3587 (1957) (Report mp 98–99°) 7) H.J. Wiemann & J. Gardan, BullFr **1958**, 433 & CA **52**, 18200(1958) (Report mp 84–85°)

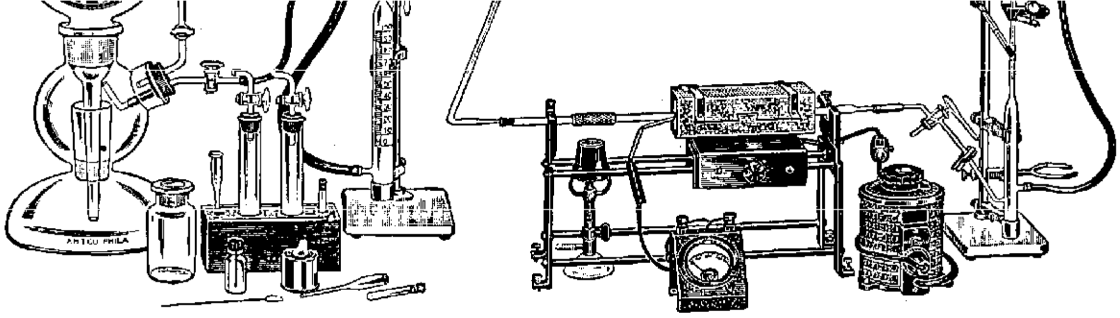
Dulitz Explosive (1886). Mixtures of K chlorate (or other oxidizer) with various quantities of gel prepd by treating NC with nitrobenzene. Eg: 10 parts of K chlorate & 1 part gel

Refs: 1) Cundill (1889) in MP **5**, 323(1892) 2) Daniel (1902), p209

Dumas Apparatus. An apparatus for determination of nitrogen in organic compds, invented by Fr scientist J.B.A. Dumas (1800–1884)

The above assembly consists of a self-sealing Kipp-Hein gas generator for furnishing comparatively air-free CO_2, a Trautz-Nierderl gasometer to measure the amt of gas used either in the determination or in the blank test; a gas burner for heating the sample and temporary filling of the combustion tube; adjustable combustion stand with electric heating elements; pyrometer with thermocouple designed for microchemical combustion procedures; a Precision Micro Nitrometer (Microazotometer) made of Pyrex glass, meniscus reader & thermometer, mounted on corrosion resistant support, with components as suggested by Nierderl & Nierderl (Ref 5), for the volumetric measurement of elementary nitrogen

Refs: 1) O.R. Trautz & J.B. Nierderl, IEC (Anal Ed) **3**, 151(1931) 2) R.T. Milner & M.S. Sherman, IEC(Anal Ed) **8**, 331(1936) 3) D.F. Hayman & S. Adler, IEC(Anal Ed) **9**, 197(1937) 4) J.R. Spies & T.H. Harris, IEC(Anal Ed) **9**, 304(1937) 5) J.B. Nierderl & V. Nierderl, Micromethods of Quantitative



Organic Analysis, 2nd edit (1942), pp79–99 6) F. Pregl & J. Grant, Quantitative Organic Microanalysis, 4th English Edition (1946), pp63–78 7) A. Steyermark et al, Analytical Chem **21**, No 12, 1559–61 (1949) (Report on recommended specifications for microchemical apparatus)

Dumdum Bullets. See Bullets, Dumdum in Vol 2 of Encycl, p B331-R

Dumorite. Accdg to Refs 1 & 3 it was a Guncotton Dynamite of high density contg 35 to 40 more 1¼ x 8 inch sticks to the case than the ordinary Dynamite. It was waterproof, and non-freezing, but too insensitive to be of use in ditch-blasting by the propagation method

Accdg to Van Gelder & Schlatter (Ref 2), it was an American low-grade blasting explosive utilizing in its manuf the NC recovered from smokeless propellants left as surplus after WWI

Refs: 1) Anon, DuPont Mag **16**, 4–5 (1922) & CA **16**, 1667 (1922) 2) Van Gelder & Schlatter (1927), p355 3) G.D. Clift & B.T. Fedoroff, "A Manual of Explosives Laboratories", Lefax, Phila, Vol **2** (1944), p D13

Dumping at Sea of Ammunition, Explosives, Propellants and Pyrotechnic Items. One of the permitted US methods of disposal of ammunition, etc is their dumping at sea. US Ordnance Regulations require that expls, proplnts and pyrotechnics be removed from their containers and dumped not less than 10 miles from shore at a depth of not less than 500 fathoms. The regulations permit dumping only expl materials that contain a water-soluble constituent and that are desensitized by partial solution in sea water

Ref: Anon, "Military Explosives", TM 9–1300–214/TO 11A–1–34 (1967), p18–1 (Compare with "Destruction of Ammunition, Explosives, Propellants and Pyrotechnics" listed in Vol 3 of Encycl, p D92-L)

Dünneberg (Duenneberg) Fabrik. One of the plants of DAG (Dynamit Aktien Gesellschaft). Founded in 1880 and located on the north bank of the Elbe river west of Geestacht ca 4 miles from Krümmel in Germany. It was famous before 1900 for the excellent quality of its BkPdr, which was prepd by the most careful blending of its ingredients. Beginning in 1902 it manufd single-base cannon, smokeless proplnt, known as DRPC-02 (Dünneberg Röhrenpulver für Kanonen, 1902). During WWII it was the principal Ger plant for the development and production of double-base, solventless proplnts, both for cannons and rockets. The total area of the plant was ca 1.8 sq miles and it employed 7600 in 1942 (mostly foreigners). This plant also prepd some of the ingredients used in manuf of propellants, but NC, glycol and acids were obtd from Krümmul Fabrik of Dynamit AG

The personnel at Dünneberg developed, in collaboration with General Uto Gallvitz, the "cool propellants": "G" or "K" Pulver (Ref 3, p Ger 70) which contd DEGDN or TEGDN and also "Gudolpulver", which contained NGu in addn to DEGDN or TEGDN (Ref 3, p Ger 81) (See also "Cool (or Cooled) Propellants" in Vol 3 of Encycl, p C511-R)

Refs: 1) Daniel (1902), 210 2) O.W. Stickland, "General Summary of (German) Explosive Plants", Dynamit AG, PB Rept No **925** (1945) 3) B.T. Fedoroff et al, PATR **2510** (1958), p Ger 70 ("G" Pulver & "K" Pulver); p Ger 81 (Gudolpulver); p Ger 218 (Dünneberg Fabrik)

Dunn, B. W. (1860–1936). Colonel, US Ordnance Dept. Introduced Ammonium Picrate for loading AP (armor-piercing) projectiles. This expl became known as "Explosive D" or "Dunnite". Col Dunn also constructed one of the modern impact test machines designed to determine sensitivity of expls by means other than an actual firing test so as not to risk destroying a gun if an explosive is too sensitive

Refs: 1) VanGelder & Schlatter (1927), pp 373 & 942 2) Army Ordnance **16**, 364 (May-June 1936) (Describes his contributions as developing an impact-testing machine, developing War Dept Factory at Picatinny Arsenal and organizing the Bureau of Explosives for the American Railway Association)

Dunnite, Explosive D, or Ammonium Picrate. See under "Phenol and Derivatives", 2,4,6-Trinitrophenol, Ammonium Salt

Duobel. Trade mark of duPont Co for high-velocity, non-gelatinous permissible explosives furnished in two grades based upon velocity and cartridge count; fair water resistance; used for mining coal where lump coal is not a factor
Refs: 1) Bebie (1943), p67 2) CondChemDict (1961), p424 3) Blasters' Hdb (1966), p44

Dupléxite. A powder proposed in 1888 by Turpin of France: K or Ba nitrate 70, DNB 10, charcoal 10 & coal tar 10%. It could be initiated by ignition
Refs: 1) E. Turpin, EnglP 189426(1888) 2) Cundill (1889) in MP **6**, 108(1893), under Turpin 3) Daniel (1902), 210 4) Escales, Chloratsprengstoffe (1910), 81 5) Giua, Tratto **6**(I)(1959), 395

Duponol. Trademark of duPont Co for a line of surface-active agents of the alcohol-sulfate type. They have detergent emulsifying, dispersing, and wetting properties; used in the textiles, paper, leather, electroplating, etc industries
Ref: CondChemDict (1961), pp424–25

DuPont & Co, Inc (E.I. duPont de Nemours & Company, Incorporated). Main Office at Wilmington, Del with plants scattered all over the US, Mexico, Canada, Europe and South America. It is the largest American concern manufacturing explosives and propellants (military and commercial) and explosive items used in coal & ore mining, rock blasting, ditch digging, etc. It manufactures also various organic and inorganic chemicals, synthetic fibers (such as Nylon, Dacron, Orlon, etc),Total number of their employees is probably ca 100,000 and of stockholders 250000

This concern originated in 1802 when a French chemist (who arrived in the US at the end of the 18th century), Eleuthère Irénée duPont de Nemours, established a BkPdr mill on the banks of Brandywine Creek near Wilmington, Delaware. The compn of powder was: saltpeter 75, charcoal 15 & sulfur 10%. From the start the duPont product enjoyed a a superior reputation. Production continued at the Brandywine Mills until 1921 when the site was abandoned because of the encroachment of the city of Wilmington. In 1857 Lammot duPont (qv) replaced the costly K nitrate with a much cheaper Na nitrate (Chile saltpeter). This invention was instrumental in increasing the consumption of duPont's BkPdr to ca 98 million pounds in the year of 1900. While the duPonts had bought an increasing interest in one company which manufactured Dynamite [invented ca 1866 by Alfred Nobel (1833–1896)], their entry into the Dynamite business is usually associated with the formation in 1880 of the Repauno Chemical Co and erection of Repauno Works on the Delaware River at Gibbstown, New Jersey. Its President, Lammot duPont, intended to make the plant so thoroughly mechanized that workmen would not be subjected to hazardous operations. His idea was not fulfilled until after his untimely death in 1884 caused by the accidental expln of Dynamite (Ref 6, pp3–5)

William duPont, who succeeded Lammot as President, carried on Lammot's plans and also made Repauno the largest Dynamite plant in the US. Here, Dynamite was manufd with wood pulp as an absorbent in place of sugar used by J. Howden since 1870 in Dynamite manufd near San Francisco, Calif. Other absorbents were K nitrate and Mg carbonate and amt of NG was 75%. Manufacture of Gelatin Dynamite began in 1893 (Ref 6, p5)

American railroads refused to transport Dynamites until duPont Co proved to them in winter of 1885–86 that it can be safely transported. In 1886 the duPont Co in collaboration with Pennsylvania RR worked up the first practical regulations governing the movement of Dynamite by rail. In rapid succession other railroads adopted similar regulations (Ref 6, pp5–6)

By 1900 the production of Dynamite in the US had reached a total in excess of 85 million pounds (Ref 6, p6)

The duPont Co started production of electric blasting caps thru the Electric Exploder Co in 1902 at Pompton Lakes, N.J. and in rapid succession three other companies were purchased and their operations, which included the manuf of fuse blasting caps, combined there (Ref 6, p6)

In 1902 the duPont Co established at Gibbstown, NJ, its research laboratory known as the Eastern Laboratory, so-called for its connection with the Eastern Dynamite Co. From a small beginning research was expanded to form three other laboratories with a combined personnel of chemists, engineers, physicists and assistants. The physical assets comprise more than 100 buildings distributed over several hundred acres. The duPont Co maintains four Development Laboratories. Two of these are concerned directly with explosives research. They are the Pompton Lakes Development Laboratory near Pompton Lakes, NJ and the Potomac River Development Laboratory, near the duPont's new and modern Dynamite Plant at Martinsburg, West Virginia (Ref 6, pp7–10)

As of 1966, the duPont Co operated one BkPdr plant, the Belin Works at Moosic, Pennsylvania, erected in 1916 to replace the Moosic Mills of 1849. Moosic discontinued commercial operations in 1973

The Repauno Works became by 1910 the largest in the World and remained so until the middle of 1930 when its production was exceeded by a plant near Modderfontein, S. Africa. In January 1954 the manuf of expls was discontinued at Repauno which thruout the years had become a large chemical plant. To supply the requirements formerly handled by Repauno, the new, ultra-modern Potomac River Works was constructed near Martinsburg, W. Virginia (Ref 6, p17)
As of 1973, duPont maintains two dynamite and two high explosives plants:
Barksdale Works, established in 1905 near Ashland, Wisconsin, manufd NG expls till 1961, but continued to manuf TNT, and non-NG expls and blasting agents. It closed in March 1971
Louviers Works, built in 1908 at Louviers, Colorado, 20 miles south of Denver, has been supplying expls for gold, silver, uranium, zinc and potash mines; also for tunnel construction, seismic prospecting and coal mining. It closed in June 1971
DuPont Works, built in 1909 at the village of DuPont, Washington, near Tacoma. It fronts Puget Sound for low-cost shipments to Alaska, Hawaii, the Philippines and some west coast South American ports. The most important outlet of this plant has been, however, to the copper, silver and zinc mines of Idaho and Montana. Expls for quarrying, lumbering, land clearing and seismic prospecting, were also important items
Birmingham Works, established in 1927 at Mineral Springs, Alabama, manufd expls except dynamite, for iron ore, limestone and coal mines. Also for granite & cement rock quarrying, copper, salt & phosphate rock mining, seismic prospecting, etc
Seneca Works, built in 1928–29 at Seneca, Illinois (90 miles southwest of Chicago), manufd prills only for coal mines, quarrying, zinc, salt & feldspar mines and agricultural work
Pompton Lakes Works, established ca 1902, in northern New Jersey, remains the only duPont plant manufg fuse- and electric-blasting caps, and other related items. The plant has been expanded from time to time as required to take care of both domestic and export markets (Ref 6, pp18–19)

Developments achieved by the duPont Co in this century include:
DuPont's Blasting Agents, such as Nitramons (expls, introduced in 1935, contain no NG or other ingredient which can be classified as an expl by itself; they cannot be initiated unless a booster is used); Packaged nitro-carbo-nitrates known as NCNs ("Aluvite", "ANFO HD"); and a new series of small-diameter water-gel explos-

ives ("Tovex", 1" to 1-3/4" dia) and intermediate-diameter ("Tovex" 1-3/4" to 4" dia), "Tovex" Extra and "Pourvex" Extra; various prill-fuel formulations (Such as "Nilite", ANFO and ANFO-P). Ref 6, pp12–14 & 48–71

DuPont's Blasting Caps, such as Fuse Caps and Electric Caps. Plain MF caps were manufd at the Pompton Lakes, NJ plant beginning in 1902. In 1912 plain caps were replaced by "composition" caps which contained a powerful base charge underneath MF, but real advances were made in 1916 when Tetryl was introduced as a base charge and another in 1929 when LA replaced MF. Eventually PETN and RDX replaced Tetryl as a base charge. Two major improvements in electric blasting caps were introduced in 1939. These were the adoption of nylon-insulated leg wires and rubber plug closures. In the fall of 1961, duPont Co introduced "Acudet" Mark V electric blasting caps. This revolutionary series consists of 14 regular delay periods, plus a "0" period, in which overlapping is eliminated and an assured time interval is provided between successive periods to allow rock movement. The "Acudet" Mark V delays cannot fire out of turn, resulting in more advance per round, better fragmentation and a lower powder factor, all of which gives the operator savings in time and money (Ref 6, pp 15–16 & Ref 7, pp6–7)

Other important developments in blasting supplies have been a "no-lag electric cap for seismic exploration", "millisecond (MS) delay electric blasting caps", "static resistant (SR) electric caps", "high temperature caps", "special oil-well detonators", "waterproof booster for exploding seismograph sleeper charges in very deep holes", a "millisecond (MS) delay connector for delay firing of Primacord", "Detaprime" Primers, and electric squibs for military and commercial uses (Ref 6, pp 16 & 87–99; also Ref 7, pp 6–7)

An electric cap developed for seismic prospecting is known as "SSS" Seismograph Electric Blasting Cap. It is supplied only in No 8 strength (Ref 6, pp90–1)

Another electric cap is known as the "jet-tapper cap". It was developed for use with *DuPont Jet Tappers*, which are expl devices utilizing the Munroe-Neumann (shaped charge) principle. These tappers, developed during WWII, found extensive use in tapping holes of open hearth furnaces (Ref 6, pp14 & 81–6)

DuPont's Delay Electric Caps, such as Acudet Mark V and MS (Millisecond) Delay (Ref 6, pp91

DuPont's "Detaclad" (Explosion-Bonded Clad Metal), Ref 6, op 14-15. See also Vol 3 of Encycl, p D96-L

DuPont's Detasheet (Flexible Sheet Explosive). See Vol 3, p D99-L

DuPont's Detonators. See Ref 7, pp6–7 and DuPont's Blasting Caps

DuPont's Dynamites, such as Straight and Ammonia Dynamites include: Red Arrow, Red Cross "Hi-Drive", Ditching, Red Cross "Extra", Loggers' 2, DuPont Extra, "Hi-Cap", Stripkolex; DuPont Gelatin, High-Velocity Gelatin, special Gelatin, Toval Gelatin Dynamite, Gelex and Seismograph Types Gelatins (Seismograph 60% High-Velocity Gelatin,) (Ref 6, pp33-42). Properties of Dynamites are described on pp 73-80

DuPont's Electric Squibs. See Ref 6, pp 94–5 and Ref 7, pp6–7

DuPont's Explosive Releases are described in Ref 7, p14, but not listed in alphabetical index of Ref 6

DuPont's Extruded El-506 Cord (Ref 7, p9)

DuPont's Flexible Cord Explosive. See Detaflex

DuPont's Flexible Linear Shaped Charge (FLSC). See Ref 7, pp8 & 12; not listed in Ref 6

DuPont's HDP (High Detonation Pressure) Primers for water gel expls (Ref 6, pp64–7)

DuPont's Jet Tapper Electric Blasting Cap. See here under Blasting Caps and Ref 6, p86

DuPont's Mechanical Dipper Process for Manufacturing Nitrocellulose. A brief description of the process is given in Vol 2, p C102 of Encycl and a schematic view of machinery is given in Vol 3, p C539-R

DuPont's Nitrometer is described in Vol 1, pp A373-L to A377-L and its illustration is on p A374-L

DuPont's Permissibles, such as Duobel, Monobel, Lump Coal and Gelobel (Ref 6, pp43–6)

DuPont's Plastisol Nitrocellulose (Ref 7, p15)
DuPont's Pourvex Extra is one of the water gel expls (Ref 6,pp 70-71)
DuPont's Prills, such as SP-2 (Ref 6, pp62–3)
DuPont's Primers, Non-NG. See Ref 6, pp63–8
DuPont's Pyrocore. See Ref 7, p9
DuPont's Seismic Prospecting Explosives, such as Seismograph 60% "High Velocity" Gelatin; Nitramons WW, WW-EL, S & S-EL; Seismogel A; Seismex and Seismex PW (Ref 6, pp358–62) (See also Seismograph Type Explosives, pp41–2)
DuPont's Special Explosive Tacot. See Ref 7, p15
DuPont's Squib. See DuPont's Electric Squib
DuPont's "SSS" Seismograph Caps. See Ref 6, pp90–1
DuPont's Stripkolex (Ref 6, p37)
DuPont's Submarine (Underwater) Blasting and Explosives Used. See Ref 6, pp421–27)
DuPont's Trimtex Explosives (Ref 6, p43)
DuPont's Water Gel Explosives, such as Tovex Extras and Pourvex Extras (Ref 6, pp68 -71)

Note: Accdg to private communication of the late Mr George D. Clift only 1% of DuPont's activities were (as of 1960) in explosives, the rest being in various chemicals, synthetic fibers (Nylon, Dacron, Orlon, etc),

Refs for DuPont Co: 1) Daniel (1902), p210 2) Van Gelder & Schlatter(1927), pp 174ff and many others
3) Smokeless Powder Department of E.I. duPont de Nemours & Co, Inc, "A History of the duPont Company Relations with the United States Government 1802–1927", Wilmington, Delaware (1928) 4) W.S. Dutton, "DuPont in World War II", Armed Forces Chemical Journal **3**, No 7, pp4–5 & 38(1950) & CA **44**, 3178(1950) (Discussion on the manuf of chemicals by DuPont Co)
5) Anon, "DuPont Opens Old Powder Mills to 7000 at 150 Anniversary", Chem&Engrg-News **30**, 3097–3100(28 July, 1952)
6) DuPont's Blasters' Handbook (1966), pp3–20 and others indicated in the text
7) "DuPont's Explosives Specialties", Pamphlet **A32618** 8) "DuPont's Explosives Specialties Selector", Pamphlet **A-42939**

DuPont Family. The name of a family of French Huguenot origin which settled in the USA in 1800. Principal members of the family connected with the expl industry and one of the world's largest chemical companies follow in alphabetical order with brief biographical sketches

DuPont de Nemours, E.I. (Eleuthère Irénée) (1771–1834). French-American scientist and industrialist, who studied in France under famous chemist A.L. Lavoisier (1743–1794), unjustly accused and executed by French revolutionists. DuPont was, before emigrating to America, one of the first directors of L'Administration des Poudres et Salpêters created in France in 1777 under Minister of Finances, A.R.J. Turgot (1727–1781). DuPont arrived in this country at the end of the 18th century and in 1802 established a Black Powder Mill near Wilmington, Delaware. This was the forerunner of the E.I. duPont de Nemours & Co (Inc) of today, which is one of the largest chemical companies in the world.

Upon the founder's death, direction of the powder concern passed to Alfred Victor, scholarly chemical experimentator, later to Henry duPont, known as "General", who was president for a long time, including the Civil War period. Henry was succeeded by Lammot duPont

Brief biographies of prominent members of the duPont family are given below
Refs: Same as under DuPont & Co, Inc and W. Haynes, ChemInd **46**, 427–34(1940) (Founder of DuPont) & CA **34**, 3548(1940)
Note: In the dictionary: "Nouveau Petit LAROUSSE Illustrée", Paris(1953), p1341, he is called " Eleuthère Désiré duPont de Nemours, chimiste, née à Paris; collaborateur de Lavoisier"

DuPont, Henry B. (1812–1889). Second son of E.I., in 1850 became 3rd president of the company, succeeding his brother, Alfred Victor, and managed it for 39 yrs. Under his direction the production of gunpowder increased due to orders from the government for the Civil War and to orders from the British during the Crimean War. Private industry placed orders for gunpowder, blasting powder & dynamite. Henry DuPont, an able executive, greatly expanded the firm's operations. He was known as "General"
Refs: 1) D. Chandler Jr, Military Affairs **13**, 142–49(1949) & CA **44**, 881(1950) (DuPont, Dahlgren, and the Civil War Niter Shortage) 2) Colliers Encycl **8**(1965), 431

DuPont, Irénée (nicknamed "Bus") (1876–1963). American industrialist, MIT graduate in engineering; he succeeded in 1919 Pierre Samuel duPont as president of the Co and retired in 1926 at the age of 49 because he thought that he "slowed up" (Ref 1) and should be replaced by a younger man. This younger brother, Lammot, was only 4 years his junior
Refs: 1) Anon, Chem&EngrgNews **30**, 3097 (July 28, 1952) 2) Anon, Ordnance **48**, 571(1964) (Obituary)

DuPont, Lammot (1831–1884). Amer industrialist, grandson of founder of the company, E.I. duPont de Nemours. He expanded the duPont works and substituted the cheaper Chile saltpeter for K nitrate (imported from India) in the manuf of BkPdr. About 1880 he established the Repauno Chemical Co which manufd Dynamite contg wood pulp as an absorbent. He was killed by accidental expln in 1884 and was succeeded by William duPont
Refs: 1) Anon, C&EN **30**, 3097 (July 28, 1952) 2) Blasters' Hdb (1966), p4

DuPont, Lammot (1880–1952). American industrialist, brother of Pierre Samuel & Irénée, who succeeded Irénée duPont in 1926 as president of the duPont Co. He retired in 1940. He was the last of the family to head the company. During this period the company began production of nylon and participated in the atomic bomb program
Refs: 1) Anon, C&EN **30**, 3097 (July 28, 1952) 2) Colliers Encycl **8**(1965), 431

DuPont, Pierre Samuel (1870–1954). American industrialist and grandson of Alfred Victor DuPont; was president of the duPont Co from 1915 to 1919, when he was succeeded by Irénée duPont (Ref 2). He was called in Ger journal SS (Ref 1) "der Pulverkönig" (Powder King)
Refs: 1) E. Neumann, SS **5**, p87(1910) 2) Anon, C&EN **30**, 3097(July 28, 1952)

DuPont, William (1855–1928). An Amer industrialist who succeeded the senior Lammot duPont in 1844 as president of Repauno. He introduced at Repauno Works many improvements in Dynamites, such as introduction in 1893 of Gelatin-Dynamites
Refs: 1) W.S. Dutton, "DuPont–140 Years", Scribner's Sons, New York (1942) pp 132–33, 137 & 139 2) Blasters, Hdb (1966), p 5 3) Miss B.F. Sargeant, Private Communication, 24 Jan 1973

Dupré, August (1835–1908). British scientist (descendant from French Huguenots) who specialized in explosives
Ref: O. Guttmann, SS **3**, 181–82 (1908) (Obituary)

Duralumin. One of the first wrought "age-hardenable" aluminum-base alloys. It was proposed in 1911 by Wilm, but modified later. The wrought Al alloys considered current in 1957 contained either 4% Cu & 0.6% Mg or 1% Si & 1% Mg (Ref 1). The compn listed in Ref 2 contained 4% Cu, 0.5% Mg, 0.25–1.0% Mn and small amts of Fe & Si. It is resistant to corrosion by

acids and sea water. Its uses include aircraft parts, boats, railroad cars, machinery, etc (Ref 2)
Refs: 1) Clark & Hawley (1957), p47
2) CondChemDict (1961), p425

Duranickel. Trademark of DuPont Co for a wrought, "age-hardenable" alloy contg ca 94% of Ni. Has greater strength and hardness and high resistance to corrosion which is characteristic of Ni
Ref: CondChemDict (1961), p425

Duraplex. Trademark of Rohm & Haas Co for drying and nondrying oil-modified alkyd resins derived from phthalic anhydride, polyhydric alcohols, and vegetable oils (Ref 2). It was used by Aerojet Engineering Corp, Azuza, Calif as an ingredient of some solid propellants, such as RL-223 (Ref 1)
Refs: 1) AerojetEngrgCorpRept No **192** (1946), pp16–17 2) CondChemDict (1961), p425

Duration and Length of Flame. It is known that in explosion in fiery coal mines many factors have to be taken into consideration in selecting the explosives which will not ignite the firedamp. Such explosives are known in the US as permissible, and they are described in Vol 3 of Encycl, pp C444 to C459. The factors important in selecting such explosives include *duration and length* of flame

It was discovered by French scientists Mallard & LeChatelier that, in order to ignite a combustible gas by a flame, spark or explosion, a certain time interval has to elapse before ignition will take place. This means that there is a certain degree of delay in ignition, or a certain period of action of the high temperature required to cause ignition. For the same combustible gas, this interval is shortened with the increase of temperature produced on detonation of explosive which is used in mining. For example, for a temp of deton of permissible expl of 650°C, the delay of ignition of firedamp observed was ca 10 sec, for temp 1000° it was ca 1 sec and for 2000° there was no delay noticed

As the *length of the flame* is usually approximately proportional to the *duration of flame,* the shorter the flame is, the safer is the explosive (provided expls in comparison possess the same temp of deton). However, there are some exceptions to this rule as described in Ref 2, p C446-L, line 3ff

It has been observed that some brisant expls do not ignite firedamp although their flames are very hot. This is because their flames are of extremely short duration. As the flame produced by BkPdr is of short duration, some authorities consider it safe for use in coal mining and some states (such as Kentucky) used it for such purpose. Mr G.D Clift, who had nearly 50 years of experience with commercial expls, knew of many explns (some with many victims) of firedamp and coal dust caused by ignition with BkPdr

Other factors of safety in coal mining are described in Ref 2, pp C446 to C450
Refs: 1) Naoúm, NG (1928), pp384–87
2) Encycl of Explosives, PATR **2700**, Vol 3

Duration and Length of Flame Determination. This can be made by photographing the flame thru a narrow slit, using a rotating drum camera with very sensitive film similar to that used for the determination of detonation velocity (Ref 4). For a horizontal drum, the height of image shows the length of flame and the width its duration. Too great emphasis must not be placed on these values as an indication of their permissibility, for both the length & duration of flames of certain nonpermissible expls are less than those of some permissible expls. The flame-test apparatus & the test method are described in detail in Ref 2
Refs: 1) Naoúm, NG (1928), p385 2) US. BurMinesBull **346**(1931), pp67–71 3) Reilly (1938), p69 4) Fedoroff et all, "A Manual for Explosives Laboratories", Vol **4**(1946), p53 (Length, etc) 5) Encycl of Expls, PATR **2700**, Vol 2, p C14-L, "Drum Camera" 6) Encycl of Expls, PATR **2700**, Vol 4 (1970), p D157 (Determination of burning

rates of gases by photographing flames as was done in Russia by V.A. Mikhel'son)

Duration Time of Burning of a Rocket Propellant. A term defined by the Aerojet Engrg Corp, Azuza, Calif, as the time elapsing between the intersection with the time axis of the tangent to the final ascending and descending portion of the thrust-time curve. The first Fig illustrates a typical curve plotted from measurements of thrust vs time values of an actual firing of a rocket unit. This measurement can not be precise because of the freedom in choosing portions of the curve that best represent the build-up & decrease in the thrust

The US Army has used the expression

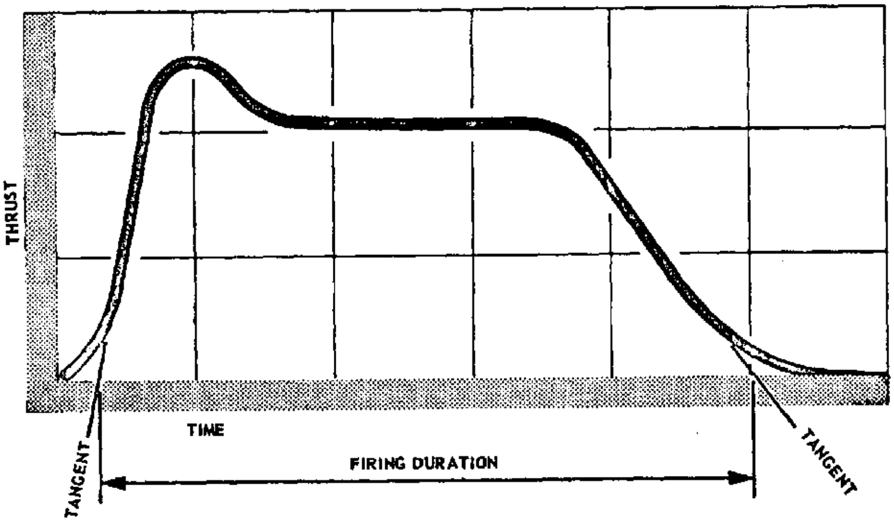


FIG – THE MEANING OF FIRING DURATION, AS APPLIED TO THE THRUST-VS-TIME PERFORMANCE OF A SOLID-PROPELLANT ROCKET UNIT

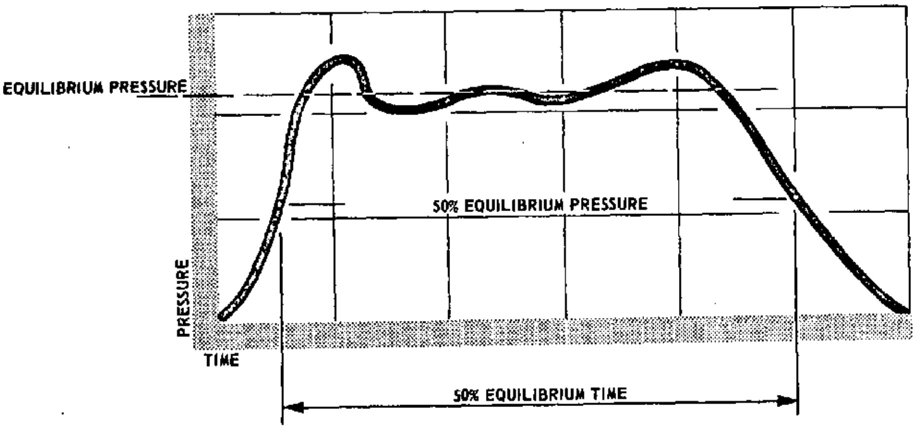


FIG – THE EXTENT OF A SPECIAL DURATION–50% EQUILIBRIUM TIME–GRAPHICALLY DEFINED ON A TYPICAL PRESSURE-VS-TIME CURVE OF A FIRING

50% equilibrium time. The second Fig explains the unique meaning of this term. It represents the duration during a firing when the internal chamber pressure at the nozzle entrance is 50% of the calculated equilibrium pressure. The curve is drawn from the recorded measurements of the chamber pressure over the entire duration of the firing, from the instant of the first rise in pressure to the instant of the last recorded measurement of pressure. In a properly operating solid-propellant power unit, the 50% point on the thrust-dieaway portion of the curve would be located approx at 50% of the max value obtainable from the curve
Ref: RocketEncycl (1959), pp140 (Duration, Firing), 165 (Firing Duration) & 586 (50-percent Equilibrium Time)

Durchschlags- und Strahlungsproben (Penetration and Radiation Tests). Ger tests similar to those of Lead Plate & Steel Plate Tests described in Vol 1 of Encycl, p XX under Plate Tests. The Ger test is called also Brisanzplattenbeschuss, which means Brisance Plate Shooting
Refs: 1) Stettbacher (1933), p361 2) Stettbacher (1948), p110

Durco. A series of cast alloys (manufd by the Duriron Co) which are resistant to acids and other corrosive liquids
Ref: CondChemDict (1961), p426

Durcon. A series of cast, plastic, epoxy formulations manufd by the Duriron Co for use in corrosion resistant equipment such as pumps, valves, fans and others
Ref: CondChemDict (1961), p426

Durene or Durol (sym-1,2,4,5-Tetramethylbenzene), $C_6H_2(CH_3)_4$, mw 134.21, colorless crysts with camphor-like odor; sp gr 0.838, mp 79–81°, bp 189–191°; sublimes and volatilizes with steam; sol in alc, eth & benz; insol in w; occurs in coal tar. It can be prepd by heating ortho-xylene & methyl chloride in presence of Al chloride; purified by crystn. It is used in plasticizers, polymers and in fibers
Refs: 1) Beil 5, 431, (207), [329] & {979}
2) CondChemDict (1961), p426

Durez. Trademark of Hooker Chem Corp, for a line of phenolformaldehyde resins and molding compds; also including diallyl phthalate resins
Refs: CondChemDict (1961), p426

Durichlor. A ferrous cast alloy, manufd by the Duriron Co. It contains besides iron, 14.5% Si, 0.85% C, 0.65% Mn & 3.0% Mo and is similar to Duriron in corrosive resistance. In addn it gives excellent resistance to hydrochloric acid and corrosive chloride salts
Ref: CondChemDict (1961), p426

Durimet-20. A ferrous alloy in cast or wrought forms manufd by the Duriron Co. It contains besides iron, 29% Ni, 20% Cr, 3.5% Cu (min), 2% Mo & 0.07% C (max) and is resistant to many acids
Ref: CondChemDict (1961), p426

Duriron. A ferrous cast alloy manufd by the Duriron Co. It contains besides iron, 14.5% Si, 0.85% C & 0.65% Mn and offers excellent resistance to cold & hot acids, but not to most strongly basic liquids
Ref: CondChemDict (1961), pp426–27

Durnford patented in 1892 in England a process of removing water (dehydration) from NC not by drying (as was previously done) but by replacement with alcohol (Ref 1). His process was essentially the same as described in Ref 2
Refs: 1) Daniel (1902), p214 2) Encycl of Expls, Vol 3 (1966), pp D44 to D47 (Dehydration or Drying of Nitrocelluloses)

Durnford Powder, patented in England in

1886 contained: K nitrate 80 parts, cork charcoal 20 & sulfur 1 to 20 parts.
Ref: Daniel (1902), p214

Duro. Trade name of Harbison-Walker Refractories Co, Pittsburgh, Pa for an acid-proof brick made from materials of very low flux content. When fired stable minerals are formed which are insol in various acids and in other corrosive liquids; used in tanks storing acids, supports for acid tower packing, etc
Ref: CondChemDict (1961), p427

Durol. See Durene

"Durol". Trademark of Allied Chem Corp for milling blocks for wool & Nylon
Ref: CondChemDict (1961), p427

Dust, Burning and Deflagration. See in Vol 4, p D154-L

Dust, Coal and Its Uses. See "Coal Dust and Its Uses" in Vol 3 of Encycl, p C358

Dust, Coal, Bomb. See "Coal Dust Bomb" in Vol 3 of Encycl, p C359

Dust, Coal and Firedamp Explosions. See under "Coal Mine Explosions and Fires", Vol 3, pp C360 to C367

Dust, Coal; Explosion Hazards from Its Use. See Vol 3, p C359-R

Dust Collector in Industry. See Fig E13 with accompanying description in this Vol under "Electrostatic Precipitation"

Dust Detonation (and Explosion). See Detonation and Explosion of Dusts, Mists and Vapors in Vol 4, p D253; also in Kirk & Othmer **12**(1954), p25, under "Safety" and K.C. Brown & G.J. James, "Dust Explosions in Factories: A Review of the Literature", SMRE Ministry of Power, Sheffield, England (1962), 714pp

Dust, Explosibility of. Dust expln data obtd in laboratory tests by the BurMines are discussed in Ref 1 for 181 miscellaneous materials that present an expln hazard. Data are given also for 50 dusts that present primarily a fire hazard and for 88 dusts that did not present an expln hazard
Refs: 1) John Nagy, A.R. Cooper & H.G. Dorsett, Jr, "Explosibility of Miscellaneous Dusts", USBurMines **R7208**, Dec 1968, 31pp. Publication Distribution Brancy, 4800 Forbes Ave, Pittsburgh, Pa, 15213. Single copy free 2) Explosives and Pyrotechnics, Vol 2, No 4 (1969), p4

Dust Explosions. Industrial dust explosions are a recognized hazard. The number of major explosions, fatalities and property loss during the years from 1900–1961 were as follows: 25 to 140 explosions per 5 year period with an average of 20 per year during the past 30 years, an average of 10 fatalities and an approx loss of 2 million dollars per year (Ref 7). This type of explosion is a hazard in all industries that involve the preparation, processing or handling of materials that have dust forming tendencies, such as coal, asphalt, pitch, etc; farm products, such as wheat, corn, starch, flour, sugar, etc; and metals such as magnesium, aluminum, titanium, etc and alloys of such metals (Refs 1, 2, 3, 6, 8 & 10)

The BurMines criteria for rating the potential explosion hazard of dust by the standard electric spark is defined by the ignition sensitivity, explosion severity and the overall explosibility indices or the correlative adjectives, weak, moderate, strong and severe (Ref 10)

It is categorically assumed that dusts ignited in the laboratory by a spark source are an explosive hazard in the industry. However, some dusts that do ignite with a spark source are also shown to present a hazard by the Godbert-Greenwald furnace test and by experience

The explosibility of a carbonaceous dust is governed by its fineness and its volatile content. A hazardous dust would, in general, have a fineness greater than 200 mesh and a volatility greater than 13%. A governing factor for a metal or alloy would be its fineness; and for a plastic, molecular configuration plays a part. Other factors that play important roles in the initiation and development of an explosion are composition of the dust, concentration of the dust cloud, composition of the atmosphere and the ignition source.

The overall explosibility of several materials when compared to Pittsburgh coal, the currently accepted standard with an explosibility index of 1, is as follows: Corn starch (coml) 7; wheat flour 4; sugar, powdered 9.6; methyl methacrylate polymer 6.3; cellulose acetate >10; magnesium, milled, 7.7; and aluminum, atomized, 2.5 (Refs 6 & 10)

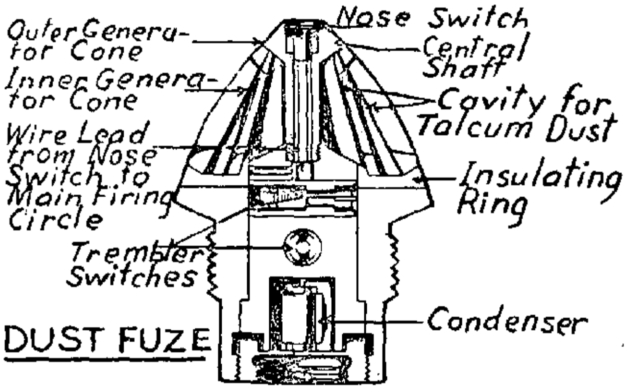
The following operating procedures will reduce explosion hazards to a minimum: good housekeeping, prevention of dust dissemination, elimination of igniting sources, use of inert atmospheres or explosion suppression devices and venting

Apparatus and procedures currently used for the examination of dusts are given in Ref 4

Refs: 1) I.G. Gwillim et al, "The Electrostatic Ignitability of Dust Clouds of Powdered Explosives", Atomic Weapons Establishment, GtBrit, Rept **ERN 25/55**(1955) 2) A.L. Golbert & L.O. Would, "The Inflammability of Magnesium, Aluminum, and Magnesium-Aluminum Alloy Dusts", TechInfoSect, Safety in Mines Research Establishment, Sheffield, England, ResRept **113**(1955) 3) W. Cybulski, Prace Glównego Inst Górnictwa No **198**, 24pp (1957) & CA **52**, 4182(1958) (The explosiveness of coal dust as a function of the distribution of coal & stone dusts) 4) H.G. Dorsett et al, "Laboratory Equipment and Test Procedures for Evaluating Explosibility of Dusts", USBurMines **RI 5624**(1960), 21pp 5) R.H. Essenhigh, "Dust Explosions in Factories. Ignition Testing and Design of a New Inflammator", Ministry Power, Safety in Mines Research Establishment (GtBrit) ResRept **188**(1960), 21pp & CA **55**, 3061(1961) 6) M. Jacobson et al, "Explosibility of Agricultural Dusts", USBurMines **RI 5753**(1961), 23pp 7) D. Kingery & J. Nagy, "The Dust Explosion Problem, Discussion on Dust Explosions", College of Metal Industries, Penn State University(1962) 8) M. Jacobson et al, "Explosibility of Metal Powders", USBurMines **RI 6516**(1964) 9) J. Nagy et al, "Preventing Ignition of Dust Explosions", USBurMines **RI 6543**(1964) & CA **62**, 394(1965) 10) J. Nagy et al, "Explosibility of Carbonaceous Dusts", USBurMines **RI 6597**(1965)

Dust and Foreign Materials. US Military Specification requirements are given in MIL-STD-286A

Dust Fuze. A German bomb fuze, developed during WWII at the Rheinmetall-Borsig laboratory, was based on the principle of charging a condenser electrostatically be means of a dust field. The fuze was located in the nose of a bomb and, prior to its dropping, the plastic cap covering the slits in the outer generator cone of the fuze was removed. As the bomb fell, the air stream entered the fuze via the slits and this action disturbed the talcum powder inside the cavity (See Fig), thus creating a dust cloud in and around



forward part of the fuze. When the dust particles came into violent contact with each other, also with the outer and inner generator cones, an electrostatic charge was developed. The condenser, which was connected to both generating cones, drew off the electric charge and built it up sufficiently to ignite the charge in detonator on impact. The size of electric charge was controlled by the quantity of dust within the fuze. The electric current could be closed for firing by any of the three switches: a nose contact switch or two trembler switches set at right angles to each other. An extremely low energy electric igniter was used with this type of switch so that even if a small part of the chge leaked from the condenser, the remaining chge would be sufficient to fire the fuze

This fuze was used in smaller Ger bombs and shells, such as 37mm

Ref: PATR **2510**(1958), pp Ger 38 & Ger 39

Dust Testing of Ammunition. The purpose of this test is to investigate the functioning of ammunition and weapons under simulated conditions likely to be encountered in the field in an extremely dusty surrounding, such as in desert countries. Detailed descriptions of the test are given in Refs 1 & 2

Essentially it consists of 1) exposing inert components (when possible) for 6 hrs at 73°±2.5°F & 22% max RH with dust (commercially known as "140 mesh silica flour) density of 0.3±0.2g/cu ft, and dust velocity of 1750±250ft/min

2) sixteen hrs at 145°±2.5°F, 10% max RH, and air vel of 300±200 ft/min, no dust feed and

3) six hrs at 145°±2.5°F, air vel 1750 ±250ft/min and dust concentration of 0.3 ±0.2g/cu ft

The test chamber barometric pressure must not exceed absolute ambient air pressure by more than 1% at any time. At the conclusion of the test, record the following: a) chamber pressure b) muzzle velocity c) target accuracy d) sabot discarding characteristics e) fuze functioning time and f) observation of loading & firing difficulties

Refs: 1) Anon, Ordnance Proof Manual **7-24**(1945), p13 2) US Army Materiel Test Procedure **MTP 4-2-819**(May 1969), Aberdeen Proving Ground, Maryland

Dutch Explosives, Ammunition and Weapons. No information at our disposal. Attempt to obtain it directly from the "Ministerie Van Defence", Netherland was unsuccessful. Our guess is that the NATO items are mostly used

Dutch Stability Test. This test, which may be considered as a modification of "Simon Thomas Fume Test", was devised in 1920 by Van Duin and Van Lennep (Ref 1). Here the loss of weight on heating of a propellant (or explosive) served as a criterion of stability

Procedure. Transfer into accurately tared glass tube, 18mm ID and 160mm long(closed with a glass stopper), an accurately weighed 4g sample of proplnt (finely ground to pass a 0.5mm sieve). Remove the stopper and heat the tube with sample for 8 hours at 104–106°C for NG proplnts or at 109–111° for straight NC proplnts. Remove the tube, close it, cool in a desiccator and weigh. Register the loss of wt. With the tube loosely closed continue heating at above temps and reweigh every morning and evening until total time of heating should reach 72 hours. Stable proplnts should not register more than 2% loss in 72 hrs and there should be no brown fumes

Refs: 1) C.F. van Duin & B.C.R. van Lennep, Rec **39**, 145–77(1920) and JCS **118**, I, p307 (1920) 2) A. Stettbacher, SS **24**, 176(1929 3) Reilly (1938), p85

Duttenhofer, Max von (1843–1903) German scientist-industrialist and one time Director of "Vereinigte Köln-Rottweiler Pulverfabriken". He made numerous contributions to the explosives industry; invented in 1882 the brown powder described below as Duttenhofer's Geschützpulver C/82, and also smokeless propellant known now as RCP (Rottweiler Cellulose Pulver). His invention was done slightly earlier and independently of P. Vieille who, in 1884, prepd the so-called "Poudre blanche", now known as "Poudre B" (See Vol 2 of Encycl, p B1-L). Duttenhofer's invention also was ahead of that of D.I. Mendeleeff's "Pyrocollodion", which contained NC of N=12.44% and served as a prototype for US Navy proplnt called "Pyro" Powder (See Bernadou in Vol 2 of Encycl, p B104). Max von Duttenhofer left a son (also called Max) who also worked in the field of expls

Refs: 1) Daniel (1902), p214 2) H. Brunswig, SS **23**, 257–60(1928) (Commemorating 25th anniversary of Duttenhofer's death)

Duttenhofer, Max von (1877–1935). German scientist-industrialist, who founded at Rottweil near Köln and later at Geeschacht on Elbe, plants for manuf of smokeless proplnt RCP, invented in 1884 by his father (See above). He died as the Director of Vereinigten Köln-Rottweiler Pulverfabriken

Ref: P. Müller, SS **31**, 1–2(1936) (Biography of Duttenhofer)

Duttenhofer's Geschützpulver C/82. A "brown" powder invented by Max von Duttenhofer (1843–1903) and successfully used (until replaced by smokeless propellants) in German large caliber cannons. It contained K nitrate 78, rye straw (slightly carbonized) 19 & sulfur 3%

Ref: H. Brunswig, SS **23**, 257–60(1928)

Duttenhofer's Smokeless Propellant, known as RCP (Rottweiler Cellulose Pulver), invented in 1884 by Max von Duttenhofer (1843–1903) consisted of fully gelatinized NC and was in the form of small, hard, irregular grains of black color (Refs 1 & 2). Stettbacher (Ref 3) called it "völlig gelatinierte Kompulver". Its manuf was done by the son of inventor at Rottweil near Köln and later at Geeschacht on Elbe. Both plants were bombed during WWII

Refs: 1) Daniel (1902), p214 2) H. Brunswig, "Das Rauchlose Pulver", DeGruyter, Berlin (1926), pp6–7 3) A. Stettbacher, "Schiess- und Sprengstoffe", Barth, Leipzig(1933), p14

Duval, Clément (1902–). His accomplishments in analytical chemistry have brought him international recognition. Although born of poor parents at Neaufles St Martin near Normandy, he obtd the doctorate in physical sciences from the Sorbonne at age 25 with a dissertation on cobalt ammines. His scientific achievements are in four categories: 1) Composition of complex compds in soln 2) IR absorption spectra of about 1000 inorg & org compds 3) discovered & developed specific spot reactions and 4) invented automatic method of gravimetry. Recipient of many honors

Ref: R.E. Oesper, JChemEd **28**, 36–37(1951)

Duxita B. An Italian expl consisting of RDX 94.5, K nitrate 3.0 & castor oil 2.5%; white, odorless pdr, d 1.74g/cc, mp ca 225°(dec); very sol in acet, sl sol in alc; Abel's Test at 80° – 30 mins. Impact Test with 5kg wt 55 to 85cm; suitable for pressure-loading shells and bombs

Ref: L.P. Pardo, Revista de Aeronautica **2**, 342(1941)

Duxite. One of the older (1914) British "permitted" explosives which passed the "Rotherham Gallery Test". It consisted of NG 31–33, CC (collodion cotton) 0.75–1.5, Na nitrate 27–29, WM (woodmeal) 8–10, Am oxalate 28–31 & moisture 0–0.5%. Maximum Charge 12 oz and Swing (Ballistic Pendulum Test) 2.45 inches for 4 oz sample vs 3.27 inches for 4 oz sample of 60% Gelignite (NG 63.5, CC 1.5, Na nitrate 27 & WM 8%)

Refs: 1) Marshall **1**(1917), 374 2) Barnett (1919), p136(Duxite); 123–25 (Rotherham Test); 182–84 (Ballistic Pendulum Test) 3) Encyclopedia of Explosives, PATR **2700**, Vol **1**(1960), pp VII–VIII (Ballistic Pendulum Test) 4) Ibid, Vol **3**(1966), p C372 (Rotherham Gallery Test)

Duxite T4. French designation for **Trimethylenetrinitramine** (qv)

DVS. It is described as "Dehydrating Value of Sulfuric in Mixed Acids" in Refs 3, 4 & 5. This value is also known as "Spent Acid Concentration", because the lower the DVS is, the weaker is the spent acid due to the presence of too much water. With such weak acid, further nitration might stop (or even reverse), even if there is still some nitric acid left. The importance of not having too much water in spent acid is discussed in the last paragraph on p D43-R of Ref 5

DVS is also known as "Driving Value of Sulfuric Acid" because it really promotes the nitration reactions (Ref 4)

Rinkenbach & Aaronson (Ref 1) calcd various values for DVS when they nitrated DEG (diethyleneglycol) to the dinitrate (DEGDN) and found that a DVS value of 3.15 represents an optimum with respect to the yield of DEGDN. They believed that reduction in yield when using a MA (mixed acid) of higher DVS value was due to the tendency of too strong sulfuric acid to dehydrate DEG with formation of polyethyleneglycols or dihydroxypolyesters

Refs: 1) W.H. Rinkenbach & H.A. Aaronson, IEC **23**, 160–67(1931) 2) Clift & Fedoroff, Vol 1(1942), Chapter XI, pp6–7 3) W.deC. Crater, IEC **40**, 1633(1948) 4) Groggins (1958), pp105–07 5) Fedoroff & Sheffield, EncyclExpls, Vol 3, pp D43-R to D44-R

Dwell or Pressure Dwell. In pressure-loading of powders into cavities, it is the interval of time that the powder is held at the full loading pressure

Ref: Anon, "Ordnance Explosive Train Designers' Handbook", NOLR 1111(1952), pG2

DXY Composition – NH_4ClO_4/Al/TNT/RDX/Desensitizer 50.5/22/13/5.5/9 (Densitizer, Micris Wax, 3717 Yellow)

Ref: G. Svadeba, NAVORD Rept **2832**, "Desensitization of Ammonium Perchlorate Explosives", Naval Ord Lab, White Oak, Md

DY Composition – NH_4ClO_4/Al/TNT/Desensitizer 45/23/23/9 (Desensitizer–Micris Wax, 3717 Yellow)

Ref: G. Svadeba, NAVORD Rept **2832**, "Desensitization of Ammonium Perchlorate Explosives", Naval Ord Lab, White Oak, Md

Dymnyi Porokh (Rus for Smoke Powder). There are three types of Russian powders known under this name:

a) *Chornyi Porokh* (Black Powder) (BkPdr). A mixt of KNO_3 75, charcoal 15 & sulfur 10%. Charcoal is prepd by calcination of alder wood at 350–450° and its free C content is 80–85%. Density of grains 1.5 to 1.93, rate of grain burning ca 0.4m/sec and of pressed powder in a fuse ca 0.9 at atm pressure (Ref 1, p 156–58). It is used for blasting in quarries when individual pieces of stone are desired, in sporting shot guns, safety fuses, delay charges, igniters for smokeless proplnts and in pyrotechnics (Ref 1, p159 & Ref 2, p26)

b) *Buryi Porokh* (Brown Powder) is similar to BkPdr but the wood is calcined at 280–320° to form a charcoal with C=70–75% (Ref 1, p156)

c) *Shokoladnyi Porokh* (Chocolate Powder) is light brown powder which contains charcoal with C=52–54% prepd by burning wood at 140–175° (Ref 1, p156)

Most commonly used "smoke powder" contained in 1957 charcoal with C=74–78%

Refs: 1) Gorst (1957), pp156–59 2) Vaskovskii (1957), p26

Dynagex. An explosive designed specifically for delay blasting in solid coal. The expl has NG content as high as possible, consistent with safety, to make it less liable to desensitization by shock compression. A high degree of safety against deflagration is achieved by eliminating cellulosic fuel

Ref: O.A. Gurton & W. Dick, IndChimBelge **1967**, 32(Spec No), 511–14(Engl) & CA **70**, 49149(1969)

Dynamagnite or Nitromagnite. A Brit 80% Dynamite contg, as an absorbent, magnesia alba (a hydrated Mg carbonate). It was patented in GtBritain in 1878 by E. Jones and authorized for manuf there in 1879. No factory was built, however, because the House of Lords declared that all expls of Dynamite-type are covered already by Nobel's patents

Refs: 1) Daniel (1902), 560 (Nitromagnite or Dynamagnite) 2) Clift & Fedoroff **2**(1943), p N35

Dynamics and Fluid Dynamics. *Dynamics* is the study of the relationship between forces acting on systems of particles & their resulting motions (Refs 1, 5 & 6)

Fluid Dynamics is the study of the relationship between forces & resulting motions of a medium that is continuously deformable by shear forces. The fluid medium is called a Newtonian fluid if the shear stress is proportional to the rate of shearing deformation. A perfect fluid has no shear stresses despite shearing deformations in the fluid (Refs 2, 3, 4, 6 & 7)

Refs: 1) S. Timoshchenko & D.H. Young, "Advanced Dynamics", McGraw-Hill, NY (1948) 2) V.L. Streeter, "Fluid Dynamics", McGraw-Hill, NY (1948) 3) L.D. Landau & E.M. Lifshitz, "Fluid Mechanics", Addison-Wesley Publg Co, Reading, Mass (1959) 4) S. Goldstein, "Lectures on Fluid Mechanics", Interscience, London, Vol 2(1960) 5) H. Goldstein, "Classical Mechanics", Addison-Wesley Publg Co, Reading, Mass (1965) 6) Kirk & Othmer, 2nd edit **9** (1966), 445–83 (Fluid Mechanics) 7) C.S. Yhi, "Fluid Mechanics", McGraw-Hill, NY (1969)

NOTE: See also Fluidics in Ordnance in Volume **6** of Encyclopedia

Dynamic Tensile Strength of Explosives. PETN, LX-04-1, PBX 9404, PBX 9011 and RX-08-AZ were tested using the Hopkinson split-bar technique. This technique allows data acquisition up to strain rates of ca 5×10^3 in/in/sec. The "dynamic" tensile strength of these expls is ca four times greater than their "static" strength.

Note: LX-04-1 consists of HMX 85 & Viton A (Vinylidene fluoride hexafluoropropylene copolymer) 14%. PBX 9404: HMX 94, NC 3 & CEF (Tris-β-chloroethylphosphate) 3%. PBX 9011: HMX 90 & Estane (Polyester urethane of adipic acid 1,4-butanediol-diphenylmethane diisocyanate) 10%. RX-08-AZ: HMX 80, FEFO (Fluorodinitroethyl formal) 13–16 & Polyester 3–6%

Refs: 1) K.G. Hoge, Explosivstoffe Vol **18**, 39–41 Feb 1970) 2) Explosives & Pyrotechnics, Vol **3**, No 12(1970), p3

Dynamite Industry is Fading Away. Since the appearance on the market after WWII of ANFO (Amm nitrate/Fuel oil), which sells at 4 to 6¢ a pound vs five times as much for Dynamite, many Dynamites plants closed down and many more are in jeopardy. Estimates are that Dynamite will drop to 5% of the expl market by 1980, while in May 1971 it held at about 30%

Refs: 1) Wall Street Journal, May 18, 1971, p40 2) Explosives & Pyrotechnics, Vol 4, No 8(1971), p 1

Dynamite Heart. An outbreak of heart disease at the Badger Army Ammunition Plant in Wisconsin has shown that workmen who handle NG can develop a dangerous dependency on it. They can suffer heart pains and even death when denied exposure to NG. Suffering usually occurs on weekends and in order to relieve pain doctors prescribed NG pills used medicinally for heart conditions

Refs: 1) Time, July 12, 1971, p41 2) Explosives & Pyrotechnics, Vol 4, No 9(1971), p4

DYNAMITE
AND
SUBSTITUTES

(Dynamite, in French; Dynamit, in German; Dinamite, in Italian; Dinamita, in Spanish; Dinamit, in Russian; and Dainamaito, in Japanese)

Dynamites are explosive mixtures which were originally based on NG(Nitroglycerin), as a sensitizer without another liquid explosive to serve for lowering the freezing point of NG. Now nearly all Dynamites manufd in US are LF(low-freezing) because they contain some NGc(Nitroglycol), known also as EGDN(Ethyleneglycol Dinitrate), mixed with NG. It replaced previously used Nitropolyglycerin, Nitro sugar, known also as SON(Sucrose Octanitrate), etc. DNCH(Dinitrochlorohydrin) has been used in Germany. Some liquid aromatic nitro compounds, such as Drip Oil(mixture of liq DNT with some TNT) are used to depress freezing point of NG. The term "Dynamite" is extended sometimes to expl mixtures contg no NG or NGc(as a sensitizer), but some other HE such as TNT, PETN or RDX. In order to avoid confusion, we are calling in this Encycl such mixts as **Substitute Dynamites** and are including under this term other industrial expls serving for the same purposes as Dynamites. Such Substitute Dynamites include **AN-FO** (Ammonium Nitrate-Fuel Oil) and their numerous variations — the most popular expls used at the present time as **Blasting Explosives.** Other Blasting Expls are described in Vol 2 of Encycl, pp B202 to B211. Many AN-FO expls are listed here under Additional References on Dynamites. Other Blasting Expls are listed under Commercial Explosives in Vol 3, pp C434 to C460

Some commercial Dynamites are used for military purposes, such as for cratering(See Vol 3, pp C553-L to C555-L) and for demolition(See Vol 3, pp D56-R to D61-R)

There are also Dynamites developed in USA especially for military purposes and some of these "Military Dynamites" contain no NG or NGc, but are based on solid HE's such as TNT or RDX plasticized by special oils. These Dynamites and anything pertaining to military uses of Dynamites are described under Class X of this section on DYNAMITE

Historical. Until about the middle of the 19th century, Black Powder (BkPdr)(See Vol 2 of Encycl, pp B165-R to B179-R) was the only explosive known and it was used not only for commercial, but also for military purposes, both as a propellant and as a bursting charge for filling bombs, grenades and artillery shells

As BkPdr was a very weak expl, attempts were made, beginning with the 19th century to replace it with something more powerful. One of such attempts was the invention of "Berthollet Powder", which consisted of K chlorate 75, sulfur 12.5 & charcoal 12.5%. This mixture was more powerful than BkPdr, but it was extremely sensitive and for this reason very dangerous to use(See Vol 2 of Encycl, pp B106 & B107)

Several other mixtures were proposed, but none of them proved to be suitable for industrial or military purposes

The first important invention before the invention of NC & NG was the prepn in 1833 of impure Nitrostarch(NS) by Braconnot. The next step was prepn in 1838 by Pelouze of nitrated paper and cotton, but it was not realized that these were actually impure, low nitrogen content, NC's. More important than the work of the above investigators were the prepn in 1846 of NC of high N content(known as Guncotton), independently by C.F. Schönbein (1799-1866) and F. Böttger (1806-1886) and in the same year of NG by an Italian chemist Ascanio Sobrero (1812-1888). The method of prepn and some props of NG were published in France in Feb 1847 by Th-J. Pelouze (1807-1867)

NG proved to be more powerful than any previously prepd expl, including Guncotton. Because of the extreme sensitivity of NG, its use on a large scale was considered to be dangerous and the fact that it was liquid made it difficult to transport or to handle. It was nearly impossible to use it in horizontal bore holes, but when using in vertical ones, there was a danger that NG might run into surrounding fissures in the rock, escape the detonation of charge, and, either burn giving deleterious nitrogen oxides, or, if unburned, remain a grave source of danger during removal of the rock by mechanical means. Attempts were made to overcome this difficulty by lining the bore-holes with wet clay, but this was time consuming and not always effective (Ref 15, p 261)

Nitroglycerin in liquid form found, however, extensive use for blasting oil wells in Pennsylvania where the petroleum industry started to develop in the middle of the 19th century. Here, near the town of Titusville, George Mowbray established sometime in the 1860's the first NG plant on the American continent. From this plant, liquid NG was transported to locations in small tanks placed on buggies with or without rubber tires. These buggies were pushed or driven by workers thru the fields. This method of transportation is described with an illustration, in the book of VanGelder & Schlatten (Ref 14, p 399). A buggy of Hercules Powder Co for transporting NG is shown in Davis (Ref 35), p 203, Fig 65

Less dangerous method was transportation of NG in a frozen state to the location and then defrosting it. Such method was practiced by Mowbray at his plant in North Adams, Massachusetts. This plant was larger than the one previously constructed at Titusville, Pa (Ref 63, p 4). Although the transportation of frozen NG is not as dangerous as that of liquid, the defrosting, which follows, is not safe unless it is conducted in a careful manner. Mowbray transported NG in tin cans and thawed them in warm water (Ref 15, p 7)

Straight NG was also used in construction of some tunnels, but it was replaced by *Guhr-dynamite* as soon as this was invented

Before inventing his Dynamite in 1863, Alfred B. Nobel (1833-1896) proposed a rather safe method of transporting liquid NG. He mixed it with 15-20 parts of anhydrous methyl alcohol and transported the resulting nonexplosive mixture in tanks to places of work. There NG was precipitated by adding water and the supernatant dilute methanol removed by decantation. As this method was time-consuming and rather wasteful (because it did not pay to recover methanol), it was seldom used in the USA. Here, where everything is done in a hurry and as cheaply as possible, it was preferred to transport NG in the liquid form, although it was more dangerous. Many accidents occurred and many lives were lost in connection with NG and as long as most of the workmen were foreigners, especially Chinese, the industrialists, who care only for profits, did not introduce any safety regulations until they were forced by the Government after establishing in 1910 the Bureau of Mines at Pittsburgh, Pennsylvania

Although NG is a very powerful explosive, it found use also in *medicine* against "angina pectoris", because it has a tendency to relax the arteries. The medicine, known as **Spirit of Glonoin** is a 1% soln of chemically pure NG in pure ethyl alcohol. The usual dose is one drop of this spirit taken in water or one lactose or dextrose pellet, contg 1/100 grain (0.0006 g) of NG, dissolved under the tongue (Ref 35, p 208)

As the accidents with NG continued to occur, some countries, among them Great Britain, prohibited its manuf and use beginning about the middle of the 1860's. But this did not stop the USA and, in fact the production increased after the construction of Mowbray's Massachusetts plant, mentioned above

Meanwhile, Alfred Nobel with the assistance of his father, Emmanuel (1801-1872), was engaged in attempts to utilize NG in explosives and to improve its method of prepn, naming their product *Pyroglycerin* or *Glonilöl*. It became later known as *Nobel Sprengöl*. It must be noted that Alfred's father, Emmanuel, was able to instruct his son in the use of expls, because he, himself, worked in this field in

Russia before and during the Crimean War (1854-1856) in installation of a sea-mine barrage to protect the Fortress Cronstadt, which served as defense of St Petersbourg against the combined Anglo-French-Turkish Fleet. This barrage of E. Nobel proved to be successful (Ref 27, p9 & Ref 37, p311)

Everything written to this point deals with NG at the time between its invention in 1846 by Sobrero and development of its preparation on an industrial scale by Alfred Nobel and his father Emmanuel during the years 1862-1863. More detailed description of this period may be found in Ref 15, pp1-7; Ref 27, pp8-13; Ref 35, pp196-97 and Ref 37, pp285-87

Accdg to Stettbacher (Ref 27, p9), A. Nobel started as early as 1862 in search of a suitable material for absorption of NG

Before starting, however, to describe the discoveries of A. Nobel, we must say a few words about attempts to render NG safe to handle by mixing it with absorbing materials, conducted prior to Nobel by other investigators. These earlier attempts were not as successful as the work of Nobel

For example, accdg to Gorst (Ref 54, pp9-10), a Russian Artillery Officer, V.F. Petrushevskii, prepared in 1854, on suggestion of Prof N.N. Zinin, a powdery explosive by mixing NG with pulverized magnesite. He named this expl *Magnesial'nyi* and proposed its use, during the Crimean War, for loading artillery shells. This was not, however, approved by military authorities, probably due to exudation of NG, since magnesite is a poor absorbent for NG. A few years later the same explosive was adopted for demolition purposes by Military Engineer, O.B. Ghern, and ca 1867 the "Magnesial'nyi" explosive was successfully used for blasting in gold mines of Eastern Siberia

There was a claim in the literature (Ref 30a) that previous to discovery by Nobel of Guhrdynamit (1867), a German mining engineer, F. Schell of Clausthal, Oberharz, used, as early as 1866, a mixture of NG and crushed "Pochsand" (ore sand) for blasting in mines of Harz mountains. After visiting Schell in 1866, Nobel found out that kieselguhr would make a better absorbent for NG than Pochsand

Prior to the discovery of Guhrdynamit, Nobel, who started to work as early as 1862 on the search for a suitable absorbent for NG, prepared a charge consisting of a cartridge made from Zn foil filled with pulverized BkPdr impregnated with NG. Such cartridges were successfully detonated under water and BritP 2359 was obtd in 1863 for their manuf. It began at his father's Helenborg plant near Stockholm, but was stopped in the fall of 1864 when a great expln took place killing Alfred's youngest brother and a chemist employee

Because BkPdr was not a good absorbent for NG and the mixture was not safe in transportation, Nobel started to investigate other possible absorbents. As he was not allowed to build another NG plant in Sweden he transferred his activity to a boat anchored in the middle of the lake, called Mälarsee. He was allowed, however, in 1865 to build a plant at Krümmel, near Hamburg in Germany. This plant, called "Alfred Nobel & Cie", was the forerunner of the famous Dynamit Aktiengesellschaft (abbr DAG), which operated during WWII 30 plants and 15 subsidiaries until most of them were destroyed by bombing (See Ref 56, ppGer 218 & Ger 219)

Later in 1865 the Swedish Govt gave A. Nobel permission to build a plant in Vintervicken near Stockholm and in 1866 permission was granted to construct a plant in Norway, which was at that time united with Sweden under the same King

In the same year, Nobel succeeded (after trying the following as absorbents for NG: charcoal, silica, powdered brick and shredded paper) in finding a good porous material, known as kieselguhr, guhr or infusorial earth. When pulverized it could absorb up to 4 times its weight of NG. On the strength of this he compounded a mixture of 75% NG with 25% guhr. This mixt proved to be satisfactory not only from a safety standpoint but also in its expl properties – being less sensitive to shock but more readily detonable than liquid NG. Nobel called the new expl *Dynamit*, derived from the Greek "dinamis", which means force. This particular expl became also known as "Guhrdynamite" and "Dynamit No 1" or "Kieselguhr Dynamite"

Accdg to Blasters' Hdbk (Ref 63, p 4), Theodore Winkler, an associate of A. Nobel, made in 1868 near San Francisco 3 pounds of Dynamite by mixing 1 part of Kieselguhr with 3 parts of NG. He used the facilities of Judson & Sheppard Chemical Works of San Francisco and then demonstrated the strong action of this new expl by blasting boulders along the line of Bay Shore Railroad. Directly thereafter an American firm started to manuf in San Francisco NG and Dynamite with an inactive base, under the name of "Nobel's Safety Powder" or *Giant Powder No 1*

Accdg to Stettbacher (Ref 27, p 9) the following plants for manuf of NG and Dynamite were established between 1868 and 1873: Zamky near Prague (Bohemia), Hangö (Finland), Ardeer (Scotland), Paulilles (France), Schlebusch near Köln (Germany), Bilbao (Spain), Isleten (Switzerland), Avigliana, near Torino (Italy), Trafalda near Lisbon (Portugal), Pressburg (Hungary) and some place near New York (USA)

As can be seen from the above list, none of the Dynamite plants constructed before 1873 in the USA belonged to the duPont & Co, the leading manufacturer of BkPdr known for its excellent quality thruout the world. There were two reasons for this in the opinion of Mr Clift (Ref 59): dangers involved in manuf, handling and transportation of NG and higher prices of Dynamites in comparison with the new formula BkPdr introduced in 1857 by Lammot duPont on replacing the Indian saltpeter (K nitrate) by a much cheaper Chile saltpeter (Na nitrate). When manuf of NG was improved in Europe and Dynamites with active base and with AN were introduced, the duPont Co started to realize the advantages of Dynamites over BkPdr for some works. They constructed in 1880 the Repauno Works at Gibbstown, New Jersey for manuf of NG and Dynamites, but continued to make BkPdr at their Brandywyne Mills plant in Delaware

Although Guhrdynamite proved to be a good, inexpensive expl (and it is still used in some parts of the world, although discontinued in the USA since ca 1908), its disadvantage was to contain 25% of an "inactive material" (kieselguhr) which did not participate in expln. To overcome this disadvantage, Abel in England and Trauzl in Austria proposed in 1867 to use NC as an absorbent for NG. As the NC used by them had a higher N content than Collodion Cotton, it could not be colloided by NG and explosives prepd by them were not of the gelatin-type, but powdery. As their NC was not a good absorbent for NG, explosives of Abel and Trauzl were exudable and for this reason not successful. Schultze of Prussia patented in 1868 an expl consisting of NG and partly nitrated wood ("nitrolignin") but this expl, known as *Dualin* (qv) also was exudable and for this reason considered unsuitable (See also under Dualin in this Vol)

It must be mentioned that at the time of Nobel's work in trying to find a suitable absorbent for NG, pulverized sand was proposed by Prof Seely and mixtures of pulverized AN with sawdust or charcoal were proposed in 1867 by Swedish inventors Norrbin & Ohlsson and also by Björkmann. When these mixtures were combined with NG, Dynamite-like expls *Ammiakkrut* and *Seranin* were obtd (See Ref 58, pp A306-R to A307-L). The *Seranin of Björkmann*, patented in 1867 consisted of NG 18.12, AN 72.46, sawdust or charcoal 8.70 & creosote 0.72 (Ref 2, p 713). It must not be confused with *Séranine of Horsley* which consisted of NG 27% absorbed by a mixture of pulverized K chlorate, gall nuts, alum & Mg sulfate (Ref 2, pp 226 & 713)

Although Nobel knew at the time of invention of his Dynamite with "active base" about advantages of Ammonium Nitrate as such a base, he could not use it in his Dynamites because AN based dynamites were already patented by Norrbin & Ohlsson. So he decided in 1870 to buy their patent covering the expl contg AN 80, NG 10-14 & charcoal 10-6% As this expl was hygroscopic, he coated particles of AN with paraffin (stearin, ozokerite or naphthaline), thus practically eliminating its hygroscopicity. For this expl Nobel was granted BritP 1570 (1873)

Previous to this, however, Nobel worked on expls consisting of NG partially absorbed by mixtures of combustible materials (such as woodmeal, charcoal, rosin, sugar, starch, etc) with oxidizers (such as K or Na nitrates) and was granted EnglPat 442 of 1869. These expls, as well as Ammoniakrut and Seranin, served as prototypes for expls known in the US as "Ammonia Dynamites" or "Ammonium Nitrate Dynamites". These Dynamites are used in the USA, Canada and Mexico (See also in Vol 1 of Encycl, under AMMONIUM NITRATE BLASTING EXPLOSIVES)(Ref 58, pp A341 to A356)

Meanwhile, ca 1870 an Amer chemist, James Howden of San Francisco, California, proposed a Dynamite consisting of 75 parts of NG absorbed by 25 parts of a mixture consisting of pulverized sugar, K nitrate and Mg carbonate. This **Howden's Dynamite** was stronger than corresponding Guhrdynamite. This Dynamite is described in DuPont's Blasters' Hdb (Ref 63, p 4) and we presume that it was manufd in San Francisco and used in 1872 on the first big Dynamite job, which was the driving of the Musconetcong tunnel, 1 mile long, near Easton, Pennsylvania on the Easton & Amboy Railroad, which later became (and still is) the Lehigh Valley Railroad

The next step in the Dynamite industry was replacement of straight NG with NG gelatinized with CC (Collodion Cotton), which is NC of ca 12% N (See Ref 60, pp C103 to C105 under CELLULOSE AND DERIVATIVES) rendering expls known as *Gelatin Dynamites* or simply *Gelatins*. The strongest of these expls were *Blasting Gelatins* (See Ref 60, pp B211-R to B212-L). BritP 4179 was granted in 1875 for both types of Dynamites. The last patent granted to Nobel on Dynamites was in 1879. That was for *Ammongelatin* or *Ammonium Nitrate Gelatin* (See Ref 58, p A295-R)

It must be noted that although Nobel believed at first that NG or Dynamite can easily be exploded by a spark or directly by a safety fuse, he found out later that it was difficult to do so. More convenient was to use MF (Mercuric Fulminate)(invented in 1815 as a good initiating explosive) and for this reason Nobel patented in 1867 (BritP 1345) the device called "fulminate blasting cap". This cap was crimped to one end of a length of safety fuse and the capped end of fuse inserted in Dynamite cartridge (Ref 35, p 19)

Freezing of Dynamites Containing NG. Since NG freezes at +13°C, it is natural that Dynamites contg NG freeze in cold weather. This is very undesirable because such Dynamites become so hard that a blasting cap cannot be inserted in cartridges. Moreover, such cartridges cannot be detonated, even when using the strongest cap (such as that of Corps of Engineers) but requires a booster. Although frozen cartridges can be detonated (with difficulty), they should not be used without defrosting them for the reason explained under Guhrdynamite listed here under Class I. Methods of defrosting are described in the same place

Since all the prescribed methods of defrosting are time-consuming, attempts to speed-up thawing such as by placing cartridges over open flames, or near stoves or hearth, or in contact with hot objects, such as steam pipes, were made, but they nearly always resulted in accidents

It seems that the safest method is to incorporate something in NG in order to lower (depress) its freezing point, thus producing a Dynamite which does not freeze in cold weather. Attempts to prepare such LF (Low-Freezing) Dynamites began ca 1860 when Rudberg proposed to add benzene or NBz to NG. These substances were not satisfactory, however, because the amt of additives required to render NG low-freezing was so great that the strength of resulting Dynamite was considerably lowered. A slightly better result was achieved in 1876 by A. Nobel who proposed to use methyl- or ethyl nitrate, but these substances were very volatile. He also proposed to use DNBz. Other substances proposed in 1876 were amyl- and isoamylnitrates. Rather better antifreezes were o-MNT, proposed in 1877 by Wohlenberg & Sundström, and polymerized glycerin proposed in 1890 by A. Wohl. All of the above listed additives reduced the strength and brisance of Dynamites, made them difficult to detonate, and affected adversely their oxygen balance

Since none of the above listed "antifreezes" was really good, many industrialists preferred not to use them at all, but just defrost the ordinary Dynamite in winter or store it in a warm magazine. This situation lasted until 1902 when a severe expln in Greisenau Mine, Germany, was caused by frozen Dynamite. This disaster produced the renewal of research on antifreezes and in 1903 the SA de Poudres et Dynamites of France introduced DNT & TNT as antifreezes. These compds did not reduce appreciably the strength of Dynamites and were satisfactory from this point of view. They, however, did not lower the fr p of NG sufficiently for use in very cold weather

In the same year, 1903, A. Mikolajczak proposed the use of Glycerin Dinitrate (GDN) in Gelatin Dynamites, such as in the formulation: GDN 38.4, NG 25.6, Collodion Cotton 1.7, Na nitrate 27.0 & woodmeal 7.3%. He described the method of prepn of GDN in journal Glückauf **1904**, p 629 (quoted from Ref 15, pp 15-16). This additive, however, was not very satisfactory because GDN contains only ca 85% of the energy of NG and possesses a high oxygen deficiency (OB to CO_2 = −17.6%), while OB of NG is +3.6%. In order to obtain a better antifreeze, the *Dynamit AG* of Germany patented in 1904 the use of Dinitrochlorohydrin (DNCH), described in this Encycl, Vol 3 (Ref 64, p C266-L) under the name of 2-Chloropropane-1,3-diol-1,3-dinitrate or β-Dinitrochlorohydrin, $(O_2NO).CH_2.CH(Cl).CH_2(ONO_2)$. This compd did not reduce appreciably the expl properties of Dynamites but lowered sufficiently the fr p of NG. As an example of such Dynamites may be cited a German LF Dynamite contg NG 44, DNCH 19, Collodion Cotton 2, Na nitrate 28 & woodmeal 7%. Its OB to CO_2 was +1.8%, Trauzl Test Value 398cc & Crusher Test Value 19mm, whereas the corresponding freezing Dynamite contg NG 63, Collod Cotton 2, Na nitrate 28 & woodmeal 7% had properties: Trauzl Test Value 415cc and Crusher Test Value 21mm (Ref 15, p 371). The disadvantage of these Dynamites was that they produced very noxious fumes contg chlorine

The problem of producing quite satisfactory LF Dynamite remained unsolved until S. Nauckhoff of Sweden published his classical work in ZAngewChem **18**, pp 11 & 53 (1905). In this work he calculated the molecular lowering of the fr p of NG by a great number of compds and formulated the requirements for a satisfactory antifreeze

After publication of Nauckhoff's paper, W. Will introduced in 1906, Diglycerin Tetranitrate (DGTeN), also known as Tetranitrodiglycerin (TeNDG), Polynitroglycerin (PNG) or Nitropolyglycerin (NPG), $(O_2NO).CH_2.-CH(ONO_2).CH_2-O-CH_2.CH(ONO_2).CH_2(ONO_2)$. Its prepn and properties are described in Naoúm (Ref 15, pp 199-202) and in Vol 5 of Encycl under Diglycerol, p D1261-L

Accdg to E.M. Symmes (translator of Naoúm's book), DGTeN was used in the USA to a very large extent beginning in 1912 until about 1926 (Ref 15, p 202, Footnote 5). It was not used, however, in Germany where it was invented. Its manuf was patented by the Zentrallstelle für wissenschaftlich-technische Untersuchungen, Neubabelsberg, near Potsdam (Ref 15, p 19)

In the years following 1906, other substances were proposed as antifreezes:

a) Mixed glycerin esters, such as Dinitroacetin with Dinitroformin [Vezio Vender, SS **2**, 21 & 195 (1907)]

b) 1,3-Butyleneglycol Dinitrate, patented in 1911 in the USA and described in Ref 15, p 375-76

c) Mixed Ethylene-propylene Glycol, prepd from "cracked" oil gases [USP's 1307032, -3 & 4(1919) and 1371215(1921)]. These mixtures were not expensive and proved to be rather satisfactory antifreezes (quoted from Ref 15, p 375, Footnote of Translator)

. The most important antifreezes proved to be **Nitroglycol** (NGc) or **Glycoldinitrate** (GcDN), also known as **Ethyleneglycol Dinitrate** or **Dinitroglycol**, $(O_2NO)CH_2.CH_2(ONO_2)$. Its prepn and props are described in Ref 15, pp 210-12 & 218-28 and also here

The first statements in the literature regarding NGc were in: GerP 179789(1904) and then later in EnglP 12770(1912) and in MémPoudres et Salpêtres **16**, 73(1912) and **17**, 175(1914). However, at that time there appeared to have been no commercial manuf of NGc because the cost of manuf of its parent compd, glycol (ethyleneglycol) was much higher than that of glycerin. This situation lasted until WWI when the scarcity of NG developed, especially in Germany. Since NGc is a very powerful expl (more so than NG), it was decided to use it in lieu of NG as an HE (and not as an antifreeze) in Dynamites, provided a reasonably inexpensive method of manuf of glycol will be developed. As a result of extensive investigations, the synthesis of glycol from alcohol via ethylene and ethylenechloride was developed by the firm Th. Goldschmidt of Essen, Germany and furnished for the explosives industries. Glycol was then nitrated to NGc partly as such and partly in mixtures with glycerin (to form NGc + NG) as a completely equivalent substitute for the pre-WWI mining expls (Ref 15, pp 211-12). When the large stocks of glycerin were released in Germany after WWI, the NG, being cheaper than NGc, started again to be used in mining explosives and the large scale production of NGc was reduced to the amount required for its use as an antifreeze in mixtures with NG. The cost of manuf of NGc was reduced, however, after WWI with the discovery of a cheap source of ethylene, independent from ethanol (such as from natural gas), and lowering of the price of chlorine used in manuf of NGc (Ref 15, p 20)

It was noted by Naoum (Ref 15, p 21), that NGc has an advantage over NG not only in its greater safety in handling, but also in its slightly greater brisance and detonation velocity. Explosives based on NGc such as *Gelatin-Astralits* developed after WWI by the Dynamit AG were allowed on German railways as ordinary freight, without restrictions

Gelatin-Astralits contg NGc were in no way inferior to Dynamites based on NG alone. They were slightly stronger than earlier (developed by Dynamit AG) Gelatin-Astralits contg large amt of DNCH, as for example: DNCH 25, NG 5, Collod Cotton 3, DNT+TNT 10, AN 45, Na nitrate 10 & woodmeal 2%. Its density was 1.45, OB to CO_2 +3.1%, Trauzl Test Value ca 400cc, Lead Block Crusher Test 18mm, detonation velocity 7300m/sec and Sensitivity to Initiation — minimum No 3 cap (Ref 15, p 379)

As an example of Gelatin-Astralit which was based on NGc may be given the one listed in Ref 15, p 381. It did not contain NG, but consisted of NGc (gelatinized with NC) 30, Aromatic Dinitrocompounds 8, Aromatic Trinitrocompounds 4 & AN+Vegetable Meal 58%. Its density was 1.45, Trauzl Test Value 415cc, Lead Block Crushing 19mm, Detonation Rate ca 6500m/sec, Temp of Explosion 2485°C, Heat of Explosion 1158.0cal/g and Sensitiveness to Initiation — can be detonated by No 1 cap

Nitroglycol was manufd in Germany before WWII by IG Farbenindustrie and has been manufd in the USA by Carbide & Carbon Chemical Corp. Gradually NGc replaced nearly all other antifreeze compds due to the following advantages:

a) It does not reduce the strengths of Dynamites because its strength is not lower than that of NG but even slightly higher
b) It is less sensitive to shock than is NG and has less tendency toward detonation by ignition
c) It can be prepd together with NG by nitrating a mixture of glycol with glycerine and no special equipment is required
d) Its density (ca 1.5) is practically the same as that of NG
e) Dynamites contg NGc are easier to detonate than those contg NG without it
f) Gelatinization of Collod Cotton is easier and faster than with NG alone and can be conducted at RT

The disadvantages of NGc, not considered serious, are as follows:

TABLE I

Component	Blasting Gelatin (Ger & Swiss)	French Gelatins		Vergé Gelatins		German Straight Dynamites		German Gelatin	Safety Explosives	
		Gomme B	Gelatine	I	II	I	II		Telsit-Gamsit (Swiss)	Grisou Dynamite (French)
NG	74.0	66.0	45.0	38.0	41.0	25.0	15.0	50.4	21.0	16.0
Diglycerin Tetranitrate	–	–	–	–	–	15.0	15.0	–	–	–
Nitroglycol	19.0	17.0	11.5	–	–	–	–	–	5.0	4.0
Dinitrochlorohydrin	–	–	–	–	–	–	–	12.6	–	–
Collodion Cotton	7.0	5.0	3.0	2.0	3.0	–	–	2.0	1.6	0.5
Liquid DNT (Drip Oil)	–	–	–	16.0	18.0	–	–	–	12.0	–
Liquid DNT + TNT	–	–	–	18.0	–	–	–	–	–	–
$NaNO_3$	–	–	32.0	–	–	50.0	55.0	26.0	–	–
KNO_3	–	9.0	–	–	–	–	–	–	–	–
NH_4NO_3	–	–	–	26.0(NH_4ClO_4)		–	–	–	–	–
$KClO_4$	–	–	–	–	38.0	–	–	–	60.0	55.0
Woodmeal (Woodpulp)	–	–	–	–	–	10.0	10.0	9.0	0.4	–
Cellulose	–	3.0	8.5	–	–	–	–	–	–	2.5
Sulfur	–	–	–	–	–	–	3.0	–	–	–
Rosin	–	–	–	–	–	–	2.0	–	–	–
NaCl	–	–	–	–	–	–	–	–	–	22.0
Properties										
OB to CO_2 %	± 0	–	–	+1.0	−1.1	–	–	–	–	–
Density – g/cc	1.55	–	–	1.52	1.57	–	–	–	–	–
Trauzl Test value – cc	555	–	–	450	405	–	–	–	–	–
Lead Block Crushing – mm	24	–	–	20.6	18.2	–	–	–	–	–
Velocity of Detn – m/sec	7600	–	–	6600	7300	–	–	–	–	–
Propagation (25mm cartridge in open air), mm	–	–	–	40	40	–	–	–	–	–

1) A higher volatility than NG, but not so high as to cause appreciable evaporation in the cartridged product
2) A slightly greater exudation in Dynamites contg NGc, than with Dynamites contg NG alone
3) A slightly more severe headache is caused by NGc contg Dynamites than with those contg NG alone (Ref 15, p380)

These disadvantages of NGc did not prevent its nearly universal adoption as an antifreeze in LF Dynamites. As was mentioned above, the US adopted it in 1926 (See also Ref 16, p1), but European countries did not adopt it until later

Table I gives examples of after WWI European LF Dynamites, some contg DNCH & DGTeN instead of NGc

Table II, which follows, gives a list of after WWI American LF Dynamites, half of them contg NG with Nitropolyglycerin, while the other half NG with NGc (Ref 16, p3)

Properties of these Dynamites are given in Ref 16, Tables 2 & 3, pp5 & 6

As is mentioned above, NGc is exclusively used now as an antifreeze, if one disregards Drip Oil used in very small amounts. The usual practice in prepn of LF Dynamites is to nitrate 25 or 20% of glycol in a mixture of 75-80% glycerin and mix the resulting blend with other ingredients (dopes) of Dynamites, such as woodpulp, AN, Na nitrate, sulfur & antacid. As NGc gelatinizes NC more rapidly than does NG, the time of gelatinization is cut to ca 5 minutes with NG-NGc mixture. Otherwise the manufacture is the same as in the case of Straight NG Dynamites

In case of a shortage of NG, as developed during WWII in Germany, the percentage of NGc could be increased and percentage of NG decreased

Accdg to the late G.D. Clift (Ref 59), it has been standard practice in the USA,

Table II
American LF (Low Freezing) Dynamites
Containing NPG (Nitropolyglycerin) or NGc (Nitroglycol)

Class and Grade of LF Dynamites	Composition, %								
	NG with NPG	NG with NGc	NC	$NaNO_3$	NH_4NO_3	Sulfur	$CaCO_3$	Carbonaceous Material	Moisture
25% Gelatin Dynamite	22.5	–	0.4	58.4	–	6.1	1.0	10.8	0.8
	–	22.3	0.5	57.8	–	5.8	1.3	11.8	0.8
40% Gelatin Dynamite	32.7	–	0.6	52.2	–	–	1.1	12.6	0.8
	–	32.8	0.6	52.4	–	–	1.1	12.6	0.5
60% Gelatin Dynamite	50.3	–	1.5	39.7	–	–	1.5	6.3	0.7
	–	50.1	1.4	38.9	–	–	1.5	7.5	0.6
60% Ammonia Gelatin	36.1	–	0.7	33.3	20.3	–	1.2	7.3	1.1
	–	36.1	0.7	32.2	21.1	–	0.9	7.7	1.3
40% Straight NG Dynamite	39.3	–	–	43.8	–	–	1.1	14.9	0.9
	–	39.2	–	43.8	–	–	1.1	15.0	0.9

since WWI, to manuf only LF Dynamites [at first with DGTeN (known also Nitropolyglycerin) and then with NGc]. Some Dynamites, however, manufd in Southern States, such as in Arizona and California did not contain any antifreeze if the Dynamite were intended for summer use, as, for example, the formulations of Apache Powder Co., Benson, Arizona, listed here in Table VIII. Formulations intended for winter use contained only half as much NGc as used in Northern or Middle States (See Apache Powder Co Tables IV & VII). Some Apache formulations contained Drip Oil as an antifreeze (Tables IX & X)

All the information about Dynamites manufd by the Apache Powder Co after WWI was obtd from the late Dr I.A. Grageroff. No info at our disposal about the current formulations of Apache Powder Co's explosives

Prior to the detailed description of current Dynamites, it is desirable to say a few words about the prepn and properties of NG and NGc, which are the principal ingredients of Dynamites. More detailed description of NG and NGc will be given under Glycerin and Glycol

Nitroglycerin (NG) or Glycerol Trinitrate, $C_3H_5(ONO_2)_3$, mw 227.09, N 18.50%, OB to CO_2 +3.5%, OB to CO +24.5%; colorless oil; sp gr 1.591 at 25°, n_D 1.4713 at 25°; fr p 13.2° (stable form) and 2.2° (labile form); bp decomp at 145–150°; hydrolysis 0.005% acid at 60° in 5 days, hygroscopicity 0.06% at 30° and 90% RH; specific heat 0.356 cal/g/°C for liquid and 0.315 for solid; viscosity 36.0 centipoises at 20°; volatility 0.11 mg/cm /hr at 60°; very sl sol in w; sol in alc or in ether; breathing of its vapors causes a headache

NG is prepd by nitration of so-called "dynamite glycerin" which is the glycerin of 99% purity, prepd as a by-product of soap manuf or by various synthetic methods. Nitration is usually conducted at 25° or below or by adding glycerin, very slowly, to a well agitated acid consisting, approximately, of nitric acid 40, sulfuric 59.5 & water 0.5%, using an acid/glycerin ratio of ca 6. Agitation of the reaction mixture is accomplished by means of compressed air. After all the glycerin has been added to the nitrator, agitation and cooling are continued until the temp drops to ca 15°. The charge is then run into a separator, where the NG rises to the top. From there NG is run off to the neutralizer where it is washed first with water, then with soda, and finally with water until the resulting washings stop producing color with phenolphthalein indicator and NG, itself, is neutral to litmus paper

NG is a very powerful and impact sensitive explosive. Following are its properties, as outlined in Refs 15 & 65

Brisance by Sand Test, Liquid Method – 51.5 g sand crushed vs 48.0 g for TNT

Detonation Rate – 1600-1900 m/sec in glass tube and 7700 in steel tube

Explosion Temperature – 222° in 5 secs

Flammability – Its explosives can be ignited by flame but not as easily as those with Nitroglycol (Ref 15, p225)

Friction Pendulum Test – 100% explns with steel shoe

Heat of Combustion – 1616 cal/g

Heat of Detonation – 1486 cal/g

Heat of Explosion – 1600 cal/g

Heat of Formation – 400 cal/g

Heat Test at 100° – % loss in first 48 hrs 3.6, in 2nd 48 hrs 3.5 and no expln in 100 hrs

Impact Sensitiveness with Kast Apparatus: 1 drop on filter paper exploded at 8 to 10 cm fall of a 2-kg weight, while Guhrdynamite exploded at 5 cm (Ref 15, p225)

Initiation Sensitiveness. It is slightly less sensitive than NGc but still can be exploded by No 1 cap (Ref 15, p226)

KI Test at 75° – less stable than Nitroglycol (Ref 15, p226)

KI Test at 81.1° – 10+ minutes

Power (Strength) by Ballistic Mortar – 140% of TNT

Power by Trauzl Test – Naoúm (Ref 15, p227) gives 590cc for 10g sample; TNT produces expansion ca 300cc

Rifle Bullet Impact Test – 100% explosions

Strength – See Power

Temperature of Explosion. Accdg to Brunswig (as quoted in Naoúm)–3470°C (Ref 15, p278)

Vacuum Stability Test at 90° – 1.6cc/g/6hrs; and at 100° – 11+cc/g/16hrs
Velocity of Detonation – See Detonation Rate

Nitroglycol (NGc), Glycol Dinitrate or Ethyleneglycol Dinitrate, $(CH_2)_2(ONO_2)_2$, mw 152.07, N 18.42%, OB to CO_2 0.0%, OB to CO +21.04%; nearly colorless, slightly syrupy liquid; sp gr 1.485 at 25°; n_D 1.4452 at 25°; fr p <–20°; bp expl at 114–116°; hygroscopicity 0.0% at 30° & 90% RH; viscosity 4.2 centipoises at 20°; volatility – much more volatile than NG; sl sol in w and sol in alc and ether; it causes headaches, just like NG

It may be prepd by nitration of ethylene glycol, $(CH_2)_2(OH)_2$ with a mixed nitric-sulfuric acid in the same apparatus that is used for prepn of NG. The glycol is prepd by synthesis from ethylene and ethylene chlorohydrin (See also Refs 12 & 13)

NGc is an explosive more powerful, but less sensitive than NG. Following are its properties, as outlined in Refs 15 & 65:
Brisance – No information
Detonation Rate: 2050 & 7300m/sec in glass tube of 10 inches diameter at RT
Explosion Temperature: 257° in 5 seconds
Flammability – Its explosives can be ignited by flame easier than those containing NG (Ref 15, p225)
Friction Pendulum Test – No information
Heat of Combustion: 1764 cal/g
Heat of Formation: 366 cal/g
Heat Test at 100° – No information
Impact Sensitiveness with Kast Apparatus – 1 drop on filter paper exploded at 20 to 25 cm fall of 2 kg weight (Ref 15, p225)
Initiation Sensitiveness – More sensitive than NG to initial impulse; can be exploded by cap No 1 (Ref 15, p226)
KI Test at 75° – More stable than NG (Ref 15, p226)
Power (Strength) by Trauzl Test – 650cc for 10g sample vs 590cc for NG (Ref 15, p227). TNT produces ca 300cc expansion
Rifle Bullet Impact Test – No information
Strength – See Power
Vacuum Stability Test – No information
Velocity of Detonation – See Detonation Rate

Classification of Dynamites

Following classification of Dynamites was given by Rinkenbach in Ref 62, pp637-44 and by others. This classification is according to the absorbent material (called *base* or *dope*) used for NG or for its combinations with other liquid nitric esters, such as NGc, etc. Dynamites contg NG combined with NGc or some other nitric esters are known as *nonfreezing*, or *low-freezing*. Practically the same classification was given by Naoum (Ref 15), Thorpe (Ref 32), Davis (Ref 35) and Perez Ara (Ref 37)

Class I. Nongelatinous (Powdery) Nitroglycerin Explosives with an Inactive (Inert) Base, such as *Gubrdynamite*, invented in 1867 by A. Nobel (Ref 62, p637)

Class II. Nongelatinous (Powdery) Nitroglycerin Explosives with an Active Base. These expls were invented by A. Nobel in 1869. The base can be either combustible or explosive. They are known as **Active Dope Dynamites** or **Straight Dynamites** (Ref 62, p639)

Class III. Gelatinous Nitroglycerin Explosives with an Explosive Base, invented by A. Nobel in 1875. They are known as **Gelatin Dynamites** and the strongest of them is **Blasting Gelatin** (Ref 62, p640)

Class IV. Nongelatinous Nitroglycerin-Ammonium Nitrate Explosives, known as **Ammonium Nitrate Dynamites** or **Ammonia Dynamites** (Ref 62, p640)

Class V. Gelatinous Nitroglycerin-Ammonium Nitrate Explosives, invented by A. Nobel in 1879 under the name of **Extra Dynamites.** They are now known as **Ammonium Nitrate Gelatin Dynamites or Ammonia Gelatin Dynamites** (Ref 62, p640)

Class VI. Semigelatinous Nitroglycerin-Ammonium Nitrate Explosives, known as **Semigelatin Dynamites** (Ref 62, p641)

Class VII. Permissible Dynamites, known in Great Britain as **Permitted Dynamites** (Ref 62, p641)

Class VIII. Nitrostarch Dynamites, which use NS in lieu of NG or NGc as a sensitizer (Ref 62, p641)

Class IX. Miscellaneous Commercial Explosives, not covered by Classes I thru VIII, are described in Chapters 16 thru 27 of DuPont's "Blasters' Handbook", listed here as Ref 63

Class X. Military Uses of Commercial Dynamites and **Military Dynamites** are described here as Class X, under "Description of Various Types of Dynamites"

Description of Various Types of Dynamites

Class I. Guhr Dynamite (GD) *(Kieselguhr Dynamite, Dynamite No 1 or Giant Powder No 1),* still used in Europe and some other countries, but its use in the USA was discontinued ca 1908 (Mr G.D. Clift claimed that it was 1907). It consists of NG 75 parts absorbed on 25 parts of guhr, which is known also as "diatomite" or "kieselguhr"

In *manufacture of Guhrdynamite,* the weighed quantities of NG and pulverized kieselguhr, placed in wooden (or lead) troughs, are kneaded carefully by hand (or by wooden shovel) and then rubbed by hand first thru a screen of 1½ meshes per cm, followed by a screen 3 meshes per cm. The resulting mix, which should have an appearance of fresh earth from the garden, is loaded by hand into a cartridge machine, which consists of a metal tube in which a wooden piston is worked by means of a lever. One end of the tube is terminated by a funnel-shaped piece with an outside diam equal to that of paper cartridge. The mass is then squeezed into cartridges (10 to 12 cm long and 20 to 30mm in diam), which are then removed and waterproofed by a paraffin coating. This description was given by the late Mr Clift (Ref 59), but a more detailed description of method of manufacture was given by Naoúm (Ref 15, pp266-73). Illustrations on pp267 thru 271 of Ref 15 show various Dynamite Cartridging Machines

GD is a plastic mass varying in color from pink or grey to dark brown, depending on the color of the guhr or whether or not ochre has been added, such as in Spanish Dynamite, known as "Dinamita tipo"

Properties of Guhr Dynamite with 75% NG are as follows:

Density – 1.4 to 1.6g/cc (1.5 is the highest practical)

Detonation Equation, accdg to Pérez Ara (Ref 37, p320): $2C_3H_5(ONO_2)_3+2.48SiO_2 \rightarrow 6CO+5H_2O+3N_2+0.5O_2+2.48SiO_2$

Detonation Rate – 5650m/sec at density 1.3 and 6650 to 6800 at 1.5g/cc

Explosion Temperature or Deflagration Point – 180–182°, when heated slowly and 240° when heated at the rate of 20° per minute (Ref 15, p 277)

Flammability. When touched with flame or incandescent body, a cartridge of GD burns without detonation. When a great mass of explosive is ignited, detonation might take place as soon as part of the mass heats to ca 180°. Explosion also occurs if the burning cartridge is placed in a tight container (Ref 62, p277)

Freezing Point – below 10° (Ref 15, p274)

Friction Sensitiveness – detonates when rubbed between iron or other metal plates (Ref 15, p276)

Gas Volume on Explosion – 536ℓ/kg (Ref 15, p276)

Heat of Explosion – ca 1200kcal/kg with water liquid (Ref 15, p276)

Impact Sensitiveness with 2kg weight ca 7cm and 20cm for frozen expl (Ref 15, p276)

Initiation Sensitiveness. GD can be detonated, when not frozen, with a cap contg 0.6g MF but requires at least 1.0g MF when in frozen state (Ref 15, p277)

Power. See Strength

Propagation of Detonation (Sensitivity by influence). Cartridges 30mm in diam can propagate detonation over a space of 30cm between them (Ref 15, p279)

Rifle Bullet Test – explodes when hit from a distance up to ca 60 paces. Less sensitive than straight NG (Ref 15, p277)

Stability by KI Test at 71°. Accdg to Engl regulations it must withstand this test for 15 mins. GD made from stable NG can be stored an unlimited time even in tropical climates (Ref 15, p275)

Strength (Power) by Trauzl Test. 10g GD under sand tamping gave a net expansion of 320cc vs 550cc for liquid NG. This means 55% NG (Ref 15, pp278-79). Rinkenbach gave 97% TNT (Ref 62, p637)

Temperature of Explosion. Accdg to Brunswig (as quoted by Naoúm) 3160°C (Ref 15, p278)

Uses of GD. It has been used extensively for blasting operations, but it proved to be not as efficient as straight NG or Dynamites with active bases

Accdg to the footnote of E.M. Symmes in the book of Naoúm (Ref 15, p266): "Although the translator entered the explosives industry in 1911 he has never yet seen any Guhr Dynamite in USA"

Guhrdynamite was proposed for use as a bursting charge in artillery projectiles but was found to be too sensitive for this purpose (See also further under Class X)

Kieselguhr was not the only inert absorbent used for prepn of Dynamites. The following materials were tried: magnesite in Rus expl called "Magnesialnyi" (See under Historical); magnesia alba, in "Dynamagnite" or "Nitromagnite"; magnesium sulfate, in "Nobel's Ardeer Powder"; pulverized mica, in "Mica Powder"; randanite in "Dynamite de Vonges"; and Na carbonate, crystallized, in "Wetterdynamit"

Frozen Guhrdynamite and other Dynamites. Guhrdynamite and other NG contg Dynamites freeze in winter to a very hard mass into which a blasting cap cannot be inserted, and which, moreover, cannot be detonated even by the strongest blasting cap. They can, however, be detonated by means of a booster, such as consisting of a small chge of unfrozen Dynamite initiated by "miner's safety fuse" (See Bickford Fuse in Vol 2 of Encycl, p B112). It is not advisable, however, to use frozen cartridges, because they lack the plasticity and cannot be inserted into boreholes without dangerous friction. This means that frozen cartridges should not be used unless they are previously softened by carefully thawing (melting) their NG. This can be done either at the place of manuf and storage or at the place of works (blasting). It must be noted that solidly frozen Dynamite can be safely transported, because such Dynamite is less sensitive to mechanical action than unfrozen or semifrozen material

Thawing (degelation or defrosting) of frozen cartridges can be accomplished by the following methods:

1) Storage of boxes contg cartridges for several days or overnight in a steam-heated magazine at temp of 15 to 30°C (Ref 15, p280 & Ref 52a, p116).

2) Placing the cartridges in a cylindrical vessel of non-rusting metal, closing the cover and immersing the ensemble in a larger diameter vessel filled with hot water at 60–70°. The exterior vessel was insulated with felt or was placed in a wooden box filled with sawdust, cotton, or asbestos. This method was used in the US (Ref 42a). In Russia they used essentially the same method, but the temp of water for thawing was 40°C (Ref 52a, p116). In an old DuPont Pamphlet (Addnl Ref 1) are described three methods. Samuel (Addnl Ref 2) described a "thaw-house" and in Engrg & MiningJ (Addnl Ref 3) is described an "electrical thawing box". Escales (Ref 4, p115) described "Nobel Cans" using hot water for thawing. Under no circumstances should the cartridges be placed near open fires, stoves, furnaces, etc. They should not be placed directly in hot w because w will displace part of NG thus lowering the strength of Dynamite and the separated NG can cause an explosion (See also Ref 15, pp280–82 & Ref 29, pp315–16)

Class II. Nongelatinous Dynamites with an Active Base, known as Straight Dynamites. In Guhrdynamite and in other Dynamites with inactive bases (dopes), the strength of NG is reduced, because these dopes do not take part in detonation. If, however, inert substances are replaced by materials which might contribute to the force of detonation, the strength of Dynamites increases. These substances, known as "active bases" or "active dopes", might include combustibles and their combinations, with Na nitrate. Small amounts of an antacid, such as Ca carbonate, are incorporated to increase the stability of such Dynamites. These expls are powdery and known in the USA as "Straight Dynamites", but this term does not include Dynamites contg AN or NC. Corresponding Ger expls are "Mischdynamite" (Ref 29, p89)

Among the dopes used may be cited the following combustibles: colophony (used by Nobel in his Dynamites of 1869 & 1873) (Vol 3 of Encycl, p C403R); woodmeal, woodpulp and sawdust in *Grisounite* and in some Amer Dynamites; cork, charcoal (in "*Carbodynamite*", described in Ref 60, p C52-L); naphthalene (in *Rheinischdynamit*) and cotton or other forms of cellulose (in "*Forcites*"). Less frequently have been used peat moss, ivory nut meal, unbaked corn flakes, starch, pulverized peanut hull, pulverized cottonseed hulls and sunflower-seed shells

In the *manufacture of Straight Dynamites*, the previously dried ingredients of dope are weighed out in a "dope house" for each batch (usually 1000 lbs of finished product) and put thru a rough screen over which a magnetic separator is placed in order to remove any iron which has entered the mass. A rough mixing of ingredients takes place during screening and the weighed quantity of dope for each batch of Dynamite is transferred to the "mixing house", where it is added to a wooden mixing bowl with two large wheels operating on a horizontal shaft and driven by an electric motor installed outside of the building. The base of the bowl is curved and kept well shellacked to prevent absorbsion by the wood of NG which is added next. After the dope has been placed in bowl, the wheels are run for ca 2 minutes (to further mix the ingredients), and the NG (which was previously weighed in a separate building called a "NG storage house") is added. Then the wheels are run an addnl 5–10 minutes to ensure a completely homogeneous mixture. During rotation, two small plows continuously keep the powder from clinging to the sides. The charge is then shovelled out of the mixing bowl into boxes which are transported by a buggy to the "pack house" where it is fed to a "packing machine" for cartridging. The cartridges (shells) are made of strong paper and are paraffined for waterproofing (Ref 59)

Straight Dynamites are very powerful expls. Their strength varies, accdg to Table 14 listed in Ref 62, p638, between 20% and 60% as expressed in percentage (approximate) of NG present. It is customary in the USA to express the density (count) in terms of the number of 1¼ by 8 inch cartridges contained in a 50 lb case. As a "standard", the USBurMines takes the *40% Straight Dynamite* of the following compn: NG 40, Na nitrate 44, woodpulp 15 & Ca carbonate 1% (Ref 11, p133). The British take as a "standard" the *60% Gelignite*, which contains NG 60, Collodion Cotton 4, woodmeal 8 & K nitrate 28% (Ref 10, p104). Its strength by ballistic pendulum swing is given as 3.27 inches (Ref 10, p184) as compared with 2.7–3.1 inches for the above Amer 40% Straight Dynamite (Ref 10, p183)

A slightly different compn for 40% Straight Dynamite is given in Table 14 of Ref 62, p638 & Ref 66, p7–83, which is reproduced here as Table III, together with count (density) of 1¼ by 8 inch cartridges per 50 lb case:

Uses: Due to the relatively high detonation rate, Straight Dynamites (especially 50 and 60% grades) are suitable for blasting hard rocks (boulders); they are also suitable for ditch blasting (by propagation) and mud-capping. As the fumes produced on their explosions are very toxic, they are unsuitable for use underground or in confined spaces. They are fairly resistant to water (Ref 63, p33)

Note: Since the above powdery expls contain Na nitrate, they have been sometimes called "Powdery Dynamites with Explosive Base". The same applies to expls contg K nitrate. There have been, however, powdery Dynamites which contained real expl ingredients (like NC) in addn to NG. To such expls belongs *Abel's Dynamite* contg NG 65.5, powdered Guncotton 30.0, K nitrate 3.50 & Na carbonate, patented in

Table III
Straight Dynamites (USA)

Components, % and Properties	Strength, %				
	20	30	40	50	60
Nitroglycerin	20.2	29.0	39.0	49.0	56.8
Na nitrate	59.3	53.3	45.5	34.4	22.6
Carbonaceous combustible material	15.4	13.7	13.8	14.6	18.2
Sulfur	2.9	2.0	–	–	–
Antacid	1.3	1.0	0.8	1.1	1.2
Moisture	0.9	1.0	0.9	0.9	1.2
Count (Density)	102	102	104	104	106
Ballistic pendulum test, % TNT	83	90	95	103	114
Detonation rate, m/sec	3600	4300	4800	5150	5900

England in 1867 (Ref 2, p1). *Trauzl's Dynamite,* proposed in 1867, consisted of NG 73, Guncotton 25.0, and charcoal 2.0%. It was impregnated with 15% of w to make it less sensitive (Ref 2, p772 & Ref 15, p282). Another of Abel's expl, known as *Glyoxylin,* consisted of Guncotton & Na nitrate saturated with NG (Ref 15, p282). An expl called *Glyoxyline* was patented in 1867 by Brodersen. It is listed in Daniel (Ref 2, p355) without giving its compn. Another Dynamite contg nitrated cellulosic substances was *Meganit* which consisted of NG 60.0, nitrated woodpulp 10.0, nitrated ivory nut meal 10.0 and Na nitrate 20.0% (Ref 15, p282). There were Dynamites contg NG and aromatic nitrocompounds. The earliest of them was *Castellanos Powder,* described in Vol 2 of Encycl, p C83). Accdg to the late G. Clift (Ref 59), none of the above expls is used now

It might be of interest to include here a list of powdery explosives, developed under the name of "Standard Dynamites" between WWI and WWII at the laboratory of Apache Powder Company, Benson, Arizona, leading manufacturers of explosives for the Western USA. This was obtained from the late Dr I.A. Grageroff, former employee of Apache Powder Co and later of Keystone Ordnance Works, Meadville, Pennsylvania

These powdery Dynamites were low-freezing and contained, besides NG/NGc and large amt of Na nitrate, a substantial amt of coated AN. One of the formulations contained less than 0.1% Collodion Cotton for reasons unknown to us. The quantity was not sufficient to serve as a gelatinizer. Oxygen balance of these explosives was near zero

Table IV lists these explosives

Table IV

Components, % and Properties	Strength, %					
	25	30	35	40	50	60
NG/NGc–90/10	15.0	16.5	19.0	22.0	24.5	26.93
Collodion Cotton	–	–	–	–	–	0.07
Na Nitrate	60.0	55.5	51.5	49.3	42.5	38.30
AN (coated)	14.0	17.0	20.0	20.0	25.0	30.00
Sulfur	4.5	4.5	3.0	2.0	1.0	–
Marble Dust	1.0	1.0	1.0	1.0	1.0	0.50
"A" Pulp *	1.5	–	1.0	1.5	2.0	–
"B" Pulp **	–	–	3.0	3.5	4.0	4.20
Oat Hulls	4.0	5.5	1.5	0.7	–	–
Detonation Rates in ft/sec	–	–	–	–	11860	13380

* "A" Pulp was a woodpulp with a low absorptive value

** "B" Pulp was a woodpulp with a high absorptive value

Note: Compare with *Cochise No 1* of Apache Powder Co, listed in Vol 3, pC388-L

Class III. Gelatinous Dynamites with Explosive Bases (Dopes). These expls contain sufficient amount of NC (Nitrocellulose) of ca 12% N (Collodion Cotton abbreviated as CC) to form a tough gel when warmed with NG and then cooled. If a mixture containing 92–03% NG is so colloided, it is termed *Blasting Gelatin (BG)* or *Nitrogelatin* and is called by the DuPont Co *100% Gelatin.* Its preparation and properties are given in Vol 2 of Encycl (Ref 60, pp B211-R to B212-L). Comparison of props of Blasting Gelatin with those contg BG 67.05 & Al 32.95% and with some other HE's is given in Vol 1 of Encycl (Ref 58, p A147, Table). The so-called *Antifume Blasting Gelatin* is described on p A466

Blasting Gelatin manufd by the DuPont Co has a texture like para rubber and is absolutely waterproof. It has been used in deep well shooting, cutting or blasting steel and sometimes for tunneling or shaft sinking in exceptionally hard materials. Since its fumes are poor, good ventilation must be provided when used underground or in confined places (Ref 63, p38)

Accdg to Stettbacher (Ref 27, p533), a low-freezing Blasting Gelatin may be obtd by substituting a nitro-aromatic compd for part of NG. Such an expl under the name of *Spezialsprengelatine* was used in the construction of the Jungfrau tunnel in Switzerland

If an expl mixture contains 90% (or less) NG, colloided with NC of ca 12% N (Collodion Cotton), it is known as *Gelatin Dynamite* or *Gelatin*, and if there is only NG and NC in such expls, DuPont Co named them *Straight Gelatins* (Ref 63, p37)

There are also DuPont's *Special Gelatins* which contain AN (Ammonium Nitrate). They are described here under Item V, entitled "Gelatinous Nitroglycerin-Ammonium Nitrate Explosives" (Ref 63, p40)

In the manufacture of Blasting Gelatin and Gelatin Dynamites, Collodion Cotton which is prepd in the manner described in Vol 2 of Encycl (Ref 60, p C103-R), is used. Before using stabilized material, known as "Dynamite Cotton", it is dried at 40° and tested for its gelatinizing properties, as described in Ref 37, p325. For this a small specimen (ca 0.2g) of NC is placed in a crucible contg ca 10g of NG which is heated on a water bath for 13mins with constant stirring of the contents with a wooden spatula. Then the crucible is cooled for 15mins by immersing it in water at 15° and contents examined. The resulting gel shall easily be separated from crucible and retain its shape afterwards. If NC proves to be suitable, a weighed portion of it is gradually run into a weighed portion of glycerin placed in a "mechanical mixer" ("kneading machine") usually of the "Figure Eight Type", which is provided with a hot water jacket. Stirring is continued until a uniform gel is obtained. Then the pulverized dope is added and stirring is continued until blending is complete. Finally, material is transferred to the "cartridging house", where it is extruded into paper cartridges (Ref 15, Footnote on p299 and Ref 59).

Properties of Blasting Gelatin are discussed in Vol 2 of Encycl (Ref 60, pp B211-R to B212-L). Its military uses are described here under Class X

Properties of Gelatin Dynamites. Since these expls contain less NG than Blasting Gelatin, they are less powerful and less brisant. They are, however, easier to initiate and more sensitive in the explosion by influence test. In the unfrozen state, Gelatin Dynamites are cohesive plastic masses which can be kneaded and shaped. They freeze easily and in a frozen condition are less sensitive to impact and more difficult to detonate. The DuPont Straight Dynamites have excellent fumes in 20 to 60% grades, but in the strengths from 70 to 90% their fumes are poor. Their plasticity makes it possible to load solidly in boreholes in order to obtain maximum loading density. They are adapted to all varieties of wet work. The higher strengths are recommended for blasting very hard, tough rock or ore as encountered in tunnel driving and in both underground and open pit mining (Ref 63, pp37–8)

DuPont Co developed also the so-called *"High-Velocity" Gelatins* of 40 to 90% strengths in order to combat the inherent characteristics of the conventional types of Gelatin Dynamites to become less sensitive and to detonate at lower velocities when aged or subjected to high hydrostatic pressures. The DuPont's "High-Velocity" Gelatings are claimed to retain their high velocities even under extremely adverse conditions. Their densities are about 10% lower

Table V
Gelatin Dynamites (USA)

Components, % and Properties	Strength, %						
	20	30	40	50	60	80	100
NG	20.2	25.4	32.0	40.1	49.6	65.4	91.0
Na nitrate	60.3	56.4	51.8	45.6	38.9	19.5	–
NC, soluble	0.4	0.5	0.7	0.8	1.2	2.6	7.9
Carbonaceous combustible material	8.5	9.4	11.2	10.0	8.3	10.1	–
Sulfur	8.2	6.1	2.2	1.3	–	–	–
Antacid	1.5	1.2	1.2	1.2	1.1	1.7	0.9
Moisture	0.9	1.0	0.9	1.0	0.9	0.7	0.2
Ballistic pendulum test, %TNT	70	78	86	93	101	112	143
Rate of detonation, m/sec	4000	4600	5150	5600	6200	–	7400

than the regular DuPont Gelatins, but they are comparable in sensitiveness to shock and friction (Ref 63, p38)

Ageing (aging) of Dynamites is discussed in detail in Vol 1 of Encycl (Ref 58, pp A110-R to A112-L)

The so-called *Toval* is the densest, most economical Gelatin Dynamite developed recently by the DuPont Co. Its composition is not given in Ref 63, p40

The foregoing Table V, given by Rinkenbach in Ref 62, p638, lists typical US "Gelatin Dynamites" in strengths ranging from 20 to 80%. It also lists "Blasting Gelatin" as 100% Gelatin Dynamite (See also Ref 66, p7-84

Compositions and properties of several German and Swiss Gelatin Dynamites are listed in PATR**2510** (Ref 56, p Ger 67). Some of them are listed here in Table VI

Accdg to Stettbacher (Ref 41, p85 and Ref 46, p111), the "Dynamit 1" (by German classification) was also known as the "65% Gelatinedynamit". It usually contained ca 0.5% of antacid (such as pulverized chalk) and could have K nitrate or K perchlorate in lieu of Na nitrate. Its NC (Collodion Cotton) content could be as high as 4% and the woodmeal could be replaced by ryemeal. This explosive was extensively used before WWII and probably later

It should be of interest to list here some "Gelatin Dynamites" developed at the laboratory of the Apache Powder Company, Benson, Arizona between WWI and WWII (See Table VII)

The "Gelatin Dynamites" of Table VII were prepd by mixing the ingredients at ca 40°(110°F) during 5–8 minutes

As the weather in Arizona and neighboring states using expls of Apache Powder Co is very hot in summer, special formulations of Gelatins were developed for summer use. Some of them are listed in Table VIII

Table VI
Gelatin Dynamites (German & Swiss)

Components, % and Properties	Designation		
	No 1	No 3	No 5
NG (or NG + NGc)	62.5	40.0	18 to 20
NC	2.5	–	–
Vegetable meal	8.0	6.0	2 to 4
TNT & DNT	–	10.0	12.0
Na nitrate	27.0	44.0	–
K perchlorate	–	–	54.0
Alkali chloride	–	–	12.0
Oxygen Balance to CO_2, %	+4.4	+7.0	+11.0
Density	**1.55**	1.6	1.8
Trauzl Test, cc	400	290	250
Pb Block Crushing, mm	20.0	18.0	18.0
Detonation Rate, m/sec	7000	6500	6500
Heat of Explosion, kcal/kg (H_2O vapor)	1235	1030	800
Temperature of Explosion, °C	2950	2800	2650

Table VII
Gelatin Dynamites of Apache Powder Co

Components, % and Properties	Strength, %					
	30	35	40	50	60	80
NG/NGc – 90/10	26.0	29.0	32.5	41.0	49.0	63.0
Collodion Cotton	0.2	0.4	0.5	0.65	0.8	1.3
$NaNO_3$	57.3	56.0	53.0	44.35	41.4	26.7
"A" Pulp *	–	0.1	0.5	–	–	–
"B" Pulp **	–	–	–	1.5	5.6	8.0
Sulfur	8.0	4.5	3.0	2.0	–	–
Oat Hulls (dry)	–	3.5	4.5	3.0	–	–
Starch (dry)	7.5	5.5	5.0	3.0	–	–
Flour	–	–	–	3.5	2.2	–
Marble Dust	1.0	1.0	1.0	1.0	1.0	1.0
Detonation Rate, m/sec	2210	2275	2500	2605	–	2735
Detonation Rate, ft/sec	7245	7515	8200	8545	–	8965

* "A" Pulp was a woodpulp with a low absorptive value

** "B" Pulp was a woodpulp with a high absorptive value

Table VIII
Gelatins (Summer Formulas) of Apache Powder Co of Arizona

Components, %	Strength, %						
	25	30	35	40	50	60	80
NG	22.5	26.0	29.0	33.0	41.0	49.0	63.0
Collodion Cotton	0.3	0.35	0.4	0.45	0.6	0.75	1.3
$NaNO_3$	62.0	60.65	57.0	53.85	48.8	41.65	26.7
"A" Pulp *	–	–	–	1.0	1.0	–	–
"B" Pulp *	–	–	–	–	1.0	3.0	8.0
"C" Pulp **	–	–	–	–	1.5	2.5	–
Flour (hard wheat)	6.2	4.0	5.0	2.0	–	–	–
Wheat Middlings	–	2.0	4.6	7.0	5.1	2.1	–
Corn Meal	4.0	2.5	–	–	–	–	–
Sawdust	–	–	1.0	–	–	–	–
Sulfur	4.0	3.5	2.0	1.7	–	–	–
Marble Dust	1.0	1.0	1.0	1.0	1.0	1.0	1.0

* "A" Pulp and "B" Pulp – See under Tables V & VII

** "C" Pulp – No information available

Class IV. Nongelatinous (Powdery) Nitroglycerin-Ammonium Nitrate Explosives, known as **Ammonium Nitrate Dynamites** or **Ammonia Dynamites.** They contain substantial amounts of AN which usually replaces part of NG and of Na nitrate. The replacement of part of Na nitrate by AN increases the volume of gas produced on detonation. They have approximately the same explosive-strength values as do the Gelatin Dynamites, but have lower rate of detonation and brisance values (Ref 62, p640)

In the *manufacture of Ammonia Dynamites*, the same procedure is followed as described here under "Nongelatinous Dynamites with an Active Base", known as "Straight Dynamites"

A list of typical American "Ammonia Dynamites" is given in Table, p A355 of Vol 1 of Encycl of Expls (Ref 58). Some of these expls are listed also in Table 14 of Ref 62, p638 and a list of several European "Ammonia Dynamites" is given in Ref 58, p A356

One of the German "Ammondynamites", which was also used in France before WWII, is listed in PATR **2510** (Ref 56, p Ger 5-L). Its compn was: NG 63.0, NC 2.0, AN 30.0 & woodmeal 5.0%. Its OB to CO_2 +1.5%, density 1.44, Trauzl test value 485 cc, Pb block crushing 21.0mm, detonation rate 7000m/sec, heat of expln (H_2O vapor) 1300kcal/kg and temp of expln 2770°C
Note: This expl, called "Ammonia Dynamite" (Forcite) by Naoúm (Ref 15, p349), seems to belong to the "Ammonia Gelatin" type

In Table IX are listed some "Ammonia Dynamites" developed at Apache Powder Co, Benson, Arizona between WWI and WWII. Composition of these expls, which are "permissible" was communicated to us in 1950 by Dr I.A. Grageroff

Table IX
Nongelatinous Ammonia Dynamites (Permissible)
of Apache Powder Co, Benson, Arizona

Components, %	Designation						
	A	B	DLF	ELF	FLF	HLF	S
NG	10.0	10.0	9.2	9.2	9.2	10.0	7.0
Drip Oil	–	–	0.8	0.8	0.8	–	0.7
AN (fine)	77.0	–	66.0	68.75	70.75	66.75	–
AN (coarse)	–	70.0	–	–	–	–	40.0
Soda (screened)	3.0	–	–	–	–	10.0	18.0
Soda (pulverized)	–	–	11.0	8.0	7.0	–	–
Pulp	10.0	5.0	8.0	8.0	8.0	8.0	–
Corn Meal	–	5.0	–	–	–	–	20.0
NaCl	–	10.0	–	–	–	–	–
NH_4Cl	–	–	5.0	5.0	4.0	5.0	–
$(NH_4)_2SO_4$	–	–	–	–	–	–	14.0
Marble Dust	–	–	–	0.25	0.25	0.25	0.3

Class V. Gelatinous Nitroglycerin-Ammonium Nitrate Explosives, known as **Ammonia Gelatin Dynamites** or **Ammonia Gelatins.** The so-called *Special Gelatins* manufd by the DuPont Co differ from "DuPont Straight Gelatins" (See Class III) in that a portion of the strength is derived from AN; however, the products still maintain most of the gelatinous characteristics of "Straight Gelatins", but they are somewhat lower in velocity of detonation and slightly less water-resistant. They are manufd by the DuPont Co in a series of grades from 25 to 80% strength and have very good fumes in all grades. They are economical and can replace "Straight Gelatins" for most uses, but are particularly suited for outside construction work. They are also widely used in quarrying and mining, where the high density and blasting action of such expls are advantageous (Ref 63, p40)

In the *manufacture of Ammonia Gelatins*, the same procedure is followed as described under "Gelatinous Dynamites with an Explosive Base". AN and other "dope" ingredients are added in pulverized form to the NG–NC gel and the ensemble stirred until complete blending

A Table, listing composition and properties of non-permissible American, Belgian, British and French Gelatin Dynamites is given in Vol 1 of Encycl (Ref 58, p A368). American Ammonia Gelatins are also listed in Table 14 of Ref 62 and in Ref 66, p7-84. Germans used before WWII several types of Ammonia Gelatins contg large proportion of DNCH (Dinitrochlorohydrin), $(O_2NO)CH_2.CH(ONO_2).CH_2Cl$. A mixture of DNCH with 15–20% NG was known as "Nitrochlorin". Following is an example of such low-freezing Dynamites:

Deutsche Ammongelatine: Dinitrochlorohydrin 30, NC 3, DNT with TNT 10, AN 45, Na nitrate 10 & woodmeal 2%; its density was 1.45, brisance by Kast formula 82×10^6 vs 86×10^6 for TNT, detonation rate 6900m/sec, Trauzl test value 400cc, heat of explosion 1100kcal/kg and temperature of explosion 2570°C (Ref 56, p Ger 5)

Class VI. Semigelatinous Nitroglycerin-Ammonium Nitrate Explosives, known as **Semigelatin Dynamites.** These expls are intermediate in composition between Ammonia Gelatins and Ammonia Dynamites, and are essentially Ammonia Dynamites to which insufficient NC has been added to form a stiff gel. They are more plastic than Ammonia Dynamites and somewhat less sensitive to shock and friction than Gelatin Dynamites, though more sensitive to initiation (Ref 62, p641). The DuPont Co has been manufacturing these expls under the name of *"Gelex"* in strengths ranging from 30% to 60%. They have found wide application in a variety of mining, quarrying and construction operations. Where applicable, the "Gelex" expls give lower costs than the Gelatin Dynamites. A special grade, known as *"Gelex" D* is designed for use in submarine blasting (Ref 63, pp 39–41)

Class VII. Permissible Dynamites, known in Great Britain as **Permitted Dynamites** are expls so formulated as to be suitable for use under conditions where explosive gas mixtures or dust-air mixtures may be present, as in coal mines (Ref 62, p641)

A detailed description of permissible coal mining expls is given in Vol 3 of Encycl (Ref 64, pp C444-R to C456-R). German permissible expls, known as *Wettersprengstoffe* are described in PATR **2510** (Ref 56, pp Ger 260–61). A list of some Permissible Dynamites developed at Apache Powder Co, Benson, Arizona is given in Table IX

The DuPont Co's permissibles comprise three series of non-gelatinous ammonia types: (1) high velocity grades, or the *"Duobel"* Dynamites; (2) low velocity grades, or the *"Monobel"* Dynamites and (3) very low velocity grades called *"Lump Coal"*. In addn there is a series of gelatinous and semi-gelatinous permissibles designated as *"Gelobel"* (Ref 63, pp43–4). Properties of these expls, without revealing their compns are given on pp 44–46 of Ref 63

Class VIII. Nitrostarch Dynamites. These powdery expls began to be manufd in the USA shortly after turn of this century, and they are made nowhere else. The name of the concern which manufactures them nearly exclusively is the Trojan Powder Company of Allentown, Pennsylvania. In these expls Nitrostarch (NS) is used as the sensitizing agent instead of NG, NGc or other liquid organic nitrates of ordinary Dynamites, or instead of TNT, RDX, etc of Military Dynamites, described here as Class X

NS Dynamites are formulated so as to be comparable with the various types of NG Dynamites with respect to conditions of use and explosive effect. NS, being a powder, does not freeze at atmospheric temperature (as does NG); is not volatile and does not cause any headaches when it comes in contact with the skin, or when small amounts of muck-pile gases (those remaining after expln in rock, etc) are inhaled. NS Dynamites do not undergo exudation as do some NG expls under adverse storage conditions. In general, NS Dynamites are less sensitive to shock, friction and heat than are corresponding NG Dynamites, and, therefore, are less hazardous when subjected to transportation and handling. Truckloads of NS Dynamites have undergone accidental burning without detonation, while NG Dynamites usually detonate under such conditions (Ref 62, p641)

Accdg to Davis (Ref 35, p276), three US patents were granted in 1916 to Bronstein & Waller of Trojan Powder Co. Following are examples: a) NS 30.0, AN-TNT mixt 15.0, Na nitrate 46.8, carbonaceous material 3.0, paraffin oil 0.7, sulfur 3.0 & Ca carbonate 1.5% b) NS 39.0, AN-TNT 20.0, Na nitrate 37.25, paraffin oil 0.75, sulfur 2.0 & Ca carbonate 1.0%; c) NS 30.0, Na nitrate 58.0, carbonaceous material 5.0, paraffin oil 0.5, sulfur 5.0 & Ca carbonate 1.5%; d) NS 40.0, Na nitrate 37.7, Ba nitrate 20.0, paraffin oil 0.8 & Na carbonate 1.5%; e) NS 40.0, Na nitrate 34.7, Ba nitrate 20.0, paraffin oil 0.8, sulfur 3.0 & Ca carbonate 1.5%; f) NS 40.0, AN-TNT 20.0, Na nitrate 17.7, Ba nitrate 20.0, paraffin oil 0.8 & Ca carbonate 1.5%. They also contained ca 0.2% of diphenylamine or urea added as stabilizers

Military uses of NS or its expls are described here under Class X. Many commercial NS expls are listed under Additional References

Class IX. Miscellaneous Commercial Dynamites, listed in alphabetical order:

A. Agriculture and Forestry Uses of Explosives are described in Vol 1 of Encycl (Ref 58, pp A112 to A114)

In Chapter 24 of Ref 63, entitled **Agricultural Blasting,** are described: a) *Ditching*, pp 367 to 378, with recommended explosive "DuPont Ditching Dynamite" which is a 50% "Straight Nitroglycerin Dynamite" (p369); b) *Field Clearing with Dynamite*, which includes *Stump Blasting* (pp378–83) with recommended expls "40% DuPont Red Cross Extra Dynamite" as well as 20% and 60% (p380); and c) *Boulder and Ledge Blasting* (pp383–84) with recommended expls: "DuPont Ditching Dynamite" or "40% DuPont Red Cross Extra Dynamite" (p383)

The following **Stump Dynamite** was developed at the Apache Powder Co, Benson, Arizona, between WWI and WWII: NG 15.2, Drip Oil 1.8, soda (pulverized) 40.0, soda (screened) 28.0, "A" Pulp (woodpulp with low absorption value) 2.5, sulfur (pulverized) 3.5, coal (pulverized) 8.0 & marble dust 1.0%. Its strength by Ballistic Pendulum Test was 73.4% of TNT

B. Construction, Uses of Explosives in are described in Chapter 25 of Ref 63. These include: a) *Excavation for highway construction* (pp385–93) with recommended expls: DuPont 40% "Special Gelatin" or "Toval", as well as "Red Cross Extra" or a grade of "Hi-Cap" (p390); and b) *Ditch Blasting for Pipeline Construction* (pp394–98) with recommended expls: DuPont "Hi-Cap", 40% "Red Cross Extra" and 40% "Special Gelatin" or "Gelex" (p394)

C. Controlled Blasting, intended to reduce and better distribute the explosive charges to minimize stressing and fracturing of rock beyond the next excavation line, is described in Chap 26 of Ref 63, pp390–420. The expls recommended by DuPont Co are: "Special Gelatin C" and "Red Cross Extra C" (p420)

D. Coyote Tunnels, Blasting of is briefly described in Vol 3 of Encycl (Ref 64, pC551) and in Ref 63, pp240–44. It is noted on p244 that when Dynamite is used in tunnel shots great care is necessary in loading, especially if it is removed from the original boxes

E. Development of Underground Mines is the title of Chap 16 in Ref 63, pp245–64. The expls recommended by DuPont Co for this work are: "Nilite 303" and standard Dynamites, such as "Gelex", "Red Cross Extra", "Hi-Cap" or, under special conditions, high strength DuPont "Special Gelatin" and "Hi-Velocity Gelatin" (Ref 63, p263)

In Chap 18 of Ref 63, pp279–84, there is described the development of "Anthracite Coal Mines", with recommended expls: DuPont "Monobel D", "Monobel E", "Monobel C" and "Monobel AA" (p282)

In Chap 19 of Ref 63, pp285–98, is described the development of "Bituminous Coal Mines", with recommended expls for blasting rock faces: DuPont "Gelobel AA", while "Monobel AA" or even lower density AN permissibles are strong enough to produce adequate breakage and displacement in soft rock or coal (p297)

In Chap 20 of Ref 63, pp299–312, is described the development of "Strip Mining of Coal" with recommended expls: "ANFO-P", "Nilite" and "Hi-Cap" (p305) for bituminous coal, while for anthracite coal "Nitramon", "Nitramite" or "Tovex" is used. If a Dynamite is preferred, "Hi-Cap" is usually most economical, although a stronger expl such as 50% "Red Cross Extra" may be needed (p310)

In Chap 21 of Ref 63, pp313–26, is described the development of "Underground Mining Other Than Coal", with recommended expls: DuPont "Nilite 303" and various grades of DuPont "Extra", "Gelex" or "Hi-Cap" Dynamites for use in "stoping methods" (p319), and the same Dynamites can be used for the "caving method" (p321) and for "room and pillar mines" (p324)

F. Quarries and Open Pits, Operations in are described in Chap 22 of Ref 63, pp327–50. The recommended expls by DuPont Co for

Table X
Quarry Powders of the Apache Powder Co, Benson, Arizona

Components, % and Properties	Designation			
	1LF	2LF	4LF	6LF
NG	7.2	8.3	7.5	4.5
Drip Oil	0.8	1.7	1.5	0.5
$NaNO_3$ (pulverized)	–	–	22.0	–
$NaNO_3$ (screened)	11.0	24.0	22.0	66.0
Sawdust	10.0	–	–	–
NH_4NO_3 (coarse)	65.0	50.0	–	–
NH_4NO_3 (fine)	–	–	27.0	–
"A" Pulp (low absorptive value)	–	5.0	6.0	–
Sulfur (pulverized)	5.0	–	8.0	–
Sulfur (screened)	–	–	–	14.0
Corn Meal	–	–	5.0	–
Coal (pulverized)	–	10.0	–	14.0
Marble Dust	1.0	1.0	1.0	1.0
Ballistic Pendulum, % TNT	98.0	88.0	85.0	69.0
Lead Block Crushing, mm	4.1	5.0	8.75	4.8

quarries are: Blasting Agents (which are described in Chap 5 of Ref 63) and Dynamites, such as 60% "Special Gelatin", 60% "Red Cross Extra", "Gelex", 40% "Red Cross Extra" and "Hi-Cap" (p333). For secondary blasting in quarries are listed expls: 20 to 40% "Red Cross Extra", "Gelex" 2, or 30 to 40% "Special Gelatin" (p347). For open pits are recommended "Nitramex 2H" and "Pelletol" (p346)

Quarry Dynamites developed at the Apache Powder Co are listed in Table X

G. Seismic or Seismographic Explosives. Accdg to Ref 63, Chap 23, pp351–66, this prospecting depends upon the fact that the earth has been segregated by geological processes into layers of varying density and elastic behavior. The purpose of seismic prospecting is to detect irregularities in the layering, such as faults and folds, which may serve as reservoirs for the accumulation of oil and gas or of substances like salt. A sudden shock such as that caused by an expln, sends out seismic energy in all directions. This energy is divided when it reaches boundaries of geological formations of different accoustic impedances (the density multiplied by the sound velocity). Some of the energy returns to the surface where it is detected by sensitive instruments. When these instruments are placed near the source, so as to record the energy traveling in an essentially vertical path, the technique is known as the *reflection method*. When the instruments are extended over large distances from the source, as compared to the depths of interest, the technique is known as the *refraction method* (pp351–52)

The seismic prospectors first utilized the refraction method, and it had spectacular success in locating salt domes along the Gulf Coast during the period 1923 to 1928. Since 1936, the reflection method has dominated seismic prospecting (pp353–54)

Accdg to Ref 62, p645, seismographic explorations are carried out chiefly in the location of petroleum deposits, both on land and below the surfaces of bodies of water

The following "Seismograph Type Dynamites" manufd by the DuPont Co are briefly described without revealing their compns (See Ref 63, pp41–3):

(1) *Seismograph 60 Percent "Hi-Velocity Gelatin"* is a Straight Dynamite which has been specially formulated to provide maximum resistance to desensitization by high hydrostatic heads (See also Ref 63, p359)
(2) *"Seismogel"* is a Straight Gelatin which is lower in cost than above Gelatin, but which generally will give an equivalent seismic energy return. It is, however, not as resistant to pressure desensitization and its use should be limited to less severe conditions (See also Ref 63, p362)
(3) *"Seismex"* is a high strength Ammonia Dynamite which provides the same seismic return as the gelatins but is not as water resistant and its detonation velocity is lower. It should not be exposed to severe conditions (See also Ref 63, p362)
(4) *"Seismic PW"* is designed for shallow-hole pattern shooting where high water resistance is not required. Its detonation velocity is low (See also Ref 63, p362)
(5) *"Hi-Cap S"* has a lower density and slightly less water resistance than "Seismex" and "Seismex PW". It is offered as an economy seismograph grade
(6) *Sausage Powder.* A series of three Ammonia Dynamites, designated as A, B & C, are intended for use in areas where the sequential shooting is matched to reduce "ghosting" and ground roll

Note 1: "Ghosting" is a false image seen in an optical instrument, or a foggy appearance produced in one by some defect, as internal reflection in a lens

Note 2: Accdg to p365 of Ref 63 the DuPont Sausage Powder is supplied in four velocities: 5300, 7000, 8100 and 10200 feet per second, while only three velocities are listed on pp43 and 358 of Ref 68

H. Submarine Blasting, described in Chap 27 of Ref 63, pp421–28, includes: a) *Harbor and Channel Works*, with recommended expls: 60 to 80% "Hi-Velocity" Gelatin and "Nitramon WW" (p421), as well as "Ditching Dynamite" (p422), 40 or 50% "Hi-Velocity" Gelatin, "Gelex D", certain grades of "Tovex", "Nitramon S" and "Pelletol" 1 (p423); for canals and levees are recommended "Nitramite" 1 & 2, "Nitramex 2H", 40 to 60% "Red Cross Extra", some "Special

Gelatins", such as 75%, 60% "Hi-Velocity" Gelatin and "Nitramon S" (pp426–27); and

b) *Blasting of Wrecked Ships* for which the 60,75 & 80% "Hi-Velocity" Gelatins (p428) are recommended by DuPont Co

I. Controlled Blasting, described in Chap 28 of Ref 63, includes *Line Drilling, Cushion Blasting* and *Pre-shearing*. One can use "Trimtex" and "Trimtex Z", which are modified DuPont Ammonia Dynamites (Ref 63, p43)

J. Well Shooting. It is occasionally required to shoot either oil, gas or water wells to fracture and loosen the producing formations, with the object of increasing the flow. Such shooting may also provide a cavity in the well, resulting in additional drainage area from the fluid bearing strata into the well. Shooting of water input wells used for "water flooding" of old oil fields is also done, and here the purpose is to increase the flow of water thru the oil bearing strata from the input wells to the producing well and thus flush out the remaining oil (Ref 63, pp477–78)

Accdg to Rinkenbach (Ref 62, p645), straight liquid Nitroglycerin (LNG) has been used for many years for "bringing in" oil wells after drilling operation. This was done despite many disastrous episodes in transportation and handling of such a sensitive explosive as LNG

In the book of VanGelder & Schlatter (Ref 14, p396) is described the use of LNG in Pennsylvania oil fields and its transportation in special "buggies" from places of manuf to oil fields

In modern practice expls less hazardous than LNG are used for enlarging well bottoms, cracking strata to rejuvenate wells and for the bringing in of dry wells. Most of these expls are solids, similar in compn to 75% Gelatin Dynamite or 70% Nitrostarch Dynamite, which have high explosive strength and detonation rate values. In some cases, a "Special Dynamite" contg only 2.5% NG and as much as 87% AN is used (Ref 62, p645)

In Blasters' Hdb (Ref 63, pp477–79) for oil or gas well shooting, the following expls are recommended: "SNG" (DuPont *Solidified NG*) and 60 & 80% "High Velocity" Gelatin. Other grades, such as the "Special Gelatin" or even "Red Cross Extra", may be substituted if water depth is not excessive. Another expl recommended by DuPont Co is *EL-389-B*. It is a desensitized NG and has less shattering effect than SMG or LNG and in some cases greater radius of blasting

K. Contract Tunneling. In Chapter 17 of Blasters' Hdb (Ref 63, pp265–78) is described tunneling used in contract construction work, such as railroad, vehicular, water, sewer and utility tunnels. Tunnels vary in size according to their purposes. Railroad and vehicular tunnels may be as great as 60 feet in diameter, while water supply, sewer and utility tunnels may be as small as 4 ft in diam

The following explosives are recommended by the DuPont Co for tunnel driving: "Gelex" 1 & 2, 40 to 60% "Special Gelatin" and "Nilite 303". For initiation, delay electric blasting caps such as DuPont "Acudet Mark V" delay is recommended (Ref 63, pp271–72)

Stettbacher in the Chapter entitled "Tunnelsprengungen" (Ref 27, pp383–86) describes boring of tunnels thru the Alps in construction of some railways connecting Switzerland with Italy, and Italy with France. He gave on p385 a list of the longest European railway tunnels. A similar list of tunnels is given in the book of Vivas, Feigenspan & Ladreda (Ref 38, p401). Following is a combination of both lists:

a) Construction of *Mont Cenis Tunnel,* 12233 meters long, connecting Italy with France, commenced in 1857 using Black Powder. It was estimated that it would take 24 years to complete it, but construction was completed in 13 years (1870) because, beginning in 1865, BlkPdr was replaced by Dynamite. It required 1000 tons of Dynamite

b) *San Gotthard Tunnel,* 14920 meters long, connecting Italy with Switzerland, used low-freezing Dynamite and construction was completed in 6¾ years between 1873 & 1880

c) *Arlberg Tunnel,* 10240 meters long, in Tyrol (Austria), used Dynamite and was constructed in 3 years, betw 1880 & 1883

d) *Simplon Tunnel,* 19770 meters long, connecting Italy and France, used Blasting Gelatin and Dynamite, and was constructed in 6¾ years, betw 1898 & 1905

e) *Loetschberg Tunnel,* 14535 meters long, connecting valleys of Rhein & Rhône, used Dynamite

and small quantity of Blasting Gelatin. It was constructed in 6½ years, betw 1906 & 1913
f) *Appennin Tunnel*, 18510 meters long, traversing the Appennin mountains in Italy, used Blasting Gelatin and Dynamite. It was constructed in 9½ years, betw 1920 & 1929

We have no information on construction of railroad tunnels in the USA

L. Miscellaneous Uses. This is the title of Chap 31 of Ref 63, pp 473–86 and it includes the following: a) *Excavating in Hard Ground* (pp473–74), with recommended expl DuPont 40% "Red Cross Extra"; b) *Blasting Old Foundations* (pp474–75), with recommended expl "Gelatin Dynamite"; c) *Demolishing Bridge Piers* (pp475–76), with recommended expls: "Nitramon" S and 40% "Red Cross Extra"; d) *Trenching in Hard Materials* (p477), with recommended expls: 40% "Red Cross Extra", "Gelex" 1 or 2 or 40 to 60% "Special Gelatin"; e) *Well Shooting* (pp477–79). See item H above; f) *Digging Pole Holes* (pp479–80), with recommended expls: "Gelex" 2, 40% "Special Gelatin" or "Red Cross Extra" and for hard rock 60% "Special Gelatin" may be used; g) *Cutting Timber* (pp480–81), with recommended expls: 60 or 80% "Hi-Velocity Gelatin"; h) *Blasting Log Jams* (p481), with recommended expls: heavy charges of Dynamite (the strength is not indicated); i) *Blasting Ice Jams* (pp481–82), with expls recommended by DuPont Co – Medium Strength Dynamites; j) *Breaking and Cutting Steel* (pp482–84), with expls recommended by DuPont Co – 60 or 80% "Hi-Velocity Gelatin" and "Blasting Gelatin"; k) *Blasting in Steel Mills* (pp484–86), with the expl recommended by DuPont Co – "Nitramon S"

Class X. Military Uses of Commercial Dynamites and Military Dynamites

Ever since the invention of NC and NG, attempts were made to apply them either by themselves or in mixtures for military purposes. Under the term "uses for military purposes" may be understood: propellants in rifle, cannon, rocket and pyrotechnic ammunition; booster and bursting charges in bombs, grenades, mines (land and sea), artillery projectiles, warheads of torpedoes and of rockets, etc; military blasting operations, military cratering, military demolition, military excavation and production of antitank ditches

Since NC's, which include Collodion Cotton, Pyrocotton and Guncotton, are not dangerous to handle, they easily found application, not only as propellants, but also as HE's for loading various kinds of ammunition. Their mixtures with "dopes" used in Dynamites can be used for blasting. Historical discussion of military application of NC is given in Vol 2 of Encycl (Ref 60, p C101). Its application as single-base "Cannon Propellants" is discussed in Ref 60, pp C29-R to C38-L and a list of some US rifle proplnts is given in Table VI, p C35 of Vol 2. Application of NC gelatinized by NG is discussed under the name of "Double-Base Cannon Propellants" on pp C33 to C36 and as a "Triple-Base Cannon Propellant" on p C37 of Vol 2

To the above discussion may be added that the Russians used compressed Guncotton, contg ca 17% of moisture since about the 1860's for loading ammunition but during the Russo-Japanese War (1904-1905), Russian shells loaded with Guncotton proved to be much inferior to Japanese shells loaded with **Shimose** (cast Picric Acid). Some Whitehead torpedoes of the Russian Navy were loaded with Guncotton as late as WWI. One of the older Russian expls, known as **Slonit** (Elephantit) consisted of Guncotton hydraulically compressed to the hardness of elephant's bone

Russian demolition charges, known as *Piroksilinovyie Shashki* consisted of compressed NC and are probably still used

Because NG and its mixtures (such as Dynamites) are very sensitive to friction and impact (especially by rifle bullet), they found no satisfactory use as bursting charges in ammunition, although many attempts were made to do so. One of the earliest attempts was by a Russian officer Petrushevskii, who made a mixture of NG with magnesite and proposed to use it for loading artillery shells during the Crimean War (1854–1856). This expl, known as "Magnesial'nyi", is discussed at the beginning of this section on DYNAMITE, under Historical (See also Ref 54, pp9–10)

When Guhrdynamite was invented in 1867, attempt was made to use it for loading artillery shells instead of BkPdr used at that time. This idea was, however, abandoned after several explns of such shells inside the barrels. These prematures were due to the high initial impulse created by propellants

A liquid expl consisting of NG & alcohol was proposed in 1890 by Smolianninoff for loading shells up to 203mm. No prematures took place at muzzle velocity up to 654m/sec (See "Americanite" in Vol 1 of Encycl, p A168-L)

When 7 to 10% of CC (Collodion Cotton, NC of ca 12%N) was incorporated with NG, the resulting hard gel was less sensitive than Guhrdynamite, and for this reason it was tried as a bursting charge in artillery shells. As this expl, known as "Blasting Gelatin", proved to be not sufficiently insensitive, it was proposed in 1878 by Siersch & Hess, both of Austria, to incorporate ca 4% of camphor. An expl consisting of NG 90 & CC 10%, with 4% camphor incorporated was used for a while by the Austrians and also by the Spanish Army under the name "Gelatina explosiva de guerra". As this expl was not sufficiently insensitive, especially to impact of rifle bullet, it was replaced in 1892 in Austria by *Ekrasit* (Ecrasite). Its compn was kept secret

A Blasting Gelatin contg NG 90, NC 7 & camphor 3% was used by the Russians as late as WWI for loading trench mortar shells. The Italians used a similar expl contg 5% camphor (Ref 15, p 11 & Ref 27, pp233–34). Stettbacher proposed later, under the name of *Kriegs-Gelatine-Pentrinit*, the military expl contg NG, PETN & NC

The use of Dynamites as bursting charges in artillery shells became possible when pneumatic guns were invented in the US. As there was no shock on firing from such guns by means of compressed air, there was no danger of explosion of Dynamites inside the barrel, as often happens even on shooting from ordinary guns. The disadvantage of pneumatic guns was their short range

Accdg to VanGelder & Schlatter (Ref 14, p934), the first gun of this type was built in 1883 by Mefford of Ohio. It was cal 2 inches. Then later guns of 4 and 8 inches were built, and in 1895 a battery of several 8 inch guns was installed at Fort Winfield Scott in California. Other batteries of that kind were installed as late as 1900. Some guns were of 15 inch caliber, firing 600 pound shells at a muzzle velocity of 600ft/sec. Some field guns of that caliber were built, but proved unsatisfactory because their range was only 1950 yards

Another variant was *Hiram Maxim Gun* (Ref 14, p937), in which the shell was started with compressed air and then its velocity was brought to the desired level by exploding a charge of volatile hydrocarbon, which was placed previously in the gun barrel at the breech

There was also a "gun" designed by W. Hicks for throwing Dynamite shells by means of centrifugal force

Pérez Ara reported (Ref 37, pp333–34) that Americans used large caliber pneumatic guns, "canones de dinamita" in their coastal defenses and also during the Spanish-American War (1898). One of the American warships "Vesubius", equipped with such guns, bombarded Cuban coastal towns at short range, but not very successfully. The shells for these guns contained "Gelatina explosiva de guerra", listed above (Ref 37, p327)

Although Dynamites proved to be unsuitable as bursting charges of artillery shells fired by propellants from ordinary guns, they have been used for the military purposes listed at the beginning of this item X, such as blasting, cratering, demolition, etc

The expls used for these purposes are described in this Encycl, under the following headings:

a) *Agriculture and Forestry Uses of Explosives* in Vol 1 (Ref 58), pp A112-R to A116-R. Some of these expls may be used for military excavations and for production of antitank ditches

b) *Blasting Explosives* in Vol 2 (Ref 60), pp B202-L to B211-R. Blasting Gelatin, pp B211-R & B212-L

c) *Cratering Charge or Cratering Explosive* in Vol 3 (Ref 64), p C553-R & p D57 and in Picatinny Arsenal Tech Repts listed as Refs 2, 3, 4, 6 & 7 on p C553-R

d) *Demolition Explosives* in Vol 3 (Ref 64), pp D56-R to D61-L and in PicArsnTechRepts, listed as Refs 3 & 9a on p D60-L

A "Special Demolition Explosive" developed by Trojan Powder Co of Allentown, Pennsylvania and standardized shortly before WWII was based on NS (Nitrostarch) instead of NG. Its compn and properties are given in Ref 52, p207; Ref 66, p7–85 and in Vol 3 of Encycl under Ref 12, listed on p D60-R. Trojan demolition explosives were tested at Picatinny Arsenal by J.D. Hopper and covered by PATR's **782**(1936), **848**(1937), **864** (1937) and **957**(1939)

Straight NS in 1/4-pound blocks was used as a US military demolition charge. Its properties are as follows: detonation rate 4570m/sec and relative effectiveness 0.86 of TNT block with velocity 6400m/sec (Ref 64, p D57, Table)

Accdg to Davis (Ref 35, pp275–76), a grenade expl of WWI, under the name of *Grenite*, consisted of dry NS grains (97%) which were sprayed with a mixture of petroleum and gum arabic (ca 1.5% each), while they were stirred in a rotary mixer. The resulting granules were dried and screened, yielding a free-running expl which could be loaded by machine

Another NS explosive of Trojan Powder Co was used during WWI for loading trench mortar shells and hand & rifle grenades. Its compn was: NS 23.0–27.0, AN 31.0–35.0, Na nitrate 36.0–40.0, charcoal 1.5–2.5, heavy hydrocarbons 0.5–1.5, antacid 0.5–1.5, DPhA 0.2–0.4 & moisture up to 0.2%. In manuf of this expl, all the dope materials were first ground to the desired fineness and dried; then turned over in a large mixing barrel while the dry NS was added. Trench mortar shells were loaded by "stemming", while grenades were loaded by "jarring" NS thru small funnel-shaped openings (Ref 35, p275)

Commercial NS Dynamites are briefly described under Class VIII of this section

Accdg to All & En Expls (Ref 38a, pp151–52), two types of military demolition expls were used during WWII. They were **50% Dynamite** and **Gelignite**. The first was a brownish powder consisting essentially of NG (ca 50%) and a mixture of Na nitrate and woodmeal, whereas the second was a brownish putty-like material consisting of NG (gelatinized with 2–7% Collodion Cotton) ca 50% and a mixture of Na nitrate and woodmeal. Explosive properties of both expls were practically the same, but the first expl was more hygroscopic and some NG exuded in storage

Properties of Gelignite are: Ballistic Strength – comparable to TNT; Brisance by Sand Test – ca 35g sand crushed vs 43g for TNT; Detonation Rate 6000m/sec vs 6900 for TNT; Friction and Impact Sensitivity – very sensitive; Rifle Bullet Sensitivity – detonate from impact of cal .30 bullet fired from 90 ft.

Gelignites were used by the British not only as demolition charges but also in A/T (antitank) mines and Stickey grenades. Another Dynamite used by the British for the same purposes as Gelignite was *Saxonite*, which, accdg to Naoum (Ref 15, p407), had the following compn: NG (with NGc) 42.5 to 62.0, NC 2.5 to 5.0, K nitrate 16.0 to 27.5, woodmeal 3.5 to 8.0, Amm oxalate 9.0 to 27.0 & chalk 0.5% (Ref 38a, pp151-52)

In addition to the above listed Dynamites, several others were developed in the US during and after WWII. Considerable work was done at Picatinny Arsenal, Dover, New Jersey, and some of its Technical Reports are listed in Vol 3 of Encycl (Ref 64) on pp C553-L and D60-L

Following are PATR's (PicArsnTechReports), Hercules Powder Co Report and a GLR (General Laboratory Rept) dealing with Military Dynamites developed at Picatinny Arsenal, Dover, New Jersey and by Hercules Powder Co, Wilmington, Delaware

PATR **1611** (1946) by L.H. Eriksen deals with development of military equivalent of 20% Gelatin Dynamite. A plastic expl designated as *PAB–1* was developed at PicArsn at the request of Corps of Engineers which needed a general blasting expl for use in both demolition and construction work around coral formations in the tropical Pacific theater. It was required that this expl should have a low rate of deton and sufficient phys & chem stability to withstand extended storage, and exposure under adverse conditions of temp and humidity. The expl developed consisted of NG 23.0, DNT oil 4.0, TNT 3.0, K perchlorate 20.0, K nitrate 47.0 & NC (13.15N) 3.0% *Properties:* Density 1.79g/cc; Brisance 22.3g sand crushed; Detonation Rate 2060m/sec in paper cylinder & 2150 in steel cylinder; Friction, by steel shoe 7 cracklings out of 10 trials and unaffected by fibre shoe; Heat Test at 100° – % Loss in wt in first 48 hrs 2.37, in 2nd 48hrs 0.17 and no expln in 100 hrs; Hygroscopicity, 90% RH, 30° gained in wt 2.25% in 26 days; Impact Test with 2kg wt, PicArsn App 7 inches; Rifle Bullet Impact Test in std cast iron bomb 100% explns; Vacuum Stability at 100° 3.66cc of gas in 40 hrs

PATR **1760** (1950) by K.G. Ottoson deals with development of Military Dynamite equivalent to Commercial Dynamites. Its composition: RDX 70, talc 15, di(2-ethylhexyl) sebacate 9, polyisobutylene 3 & SAE 10W motor oil 3%. It was equivalent in strength to 47% Straight Dynamite and its properties are: Brisance 1/3 to 1/2 that of Composition C-3, when initiated with a Corps of Engineers Blasting Cap and Detonation Rate 6625 m/sec (22000 ft/sec) at max density 1.40 g/cc

It was intended for use as a demolition charge

A more powerful explosive, also intended for demolition charges, was Comp C-4 or Harrisite, also developed by Ottoson. Its detailed description is given in Vol 3 of Encycl (Ref 64, p C485

GLR **51-H1-1826** (1951) by A. Rubin & G. Silvestro deals with a Dynamite known as *Tooth Paste Explosive.* It consists of RDX 84, di (2-ethylhexyl) sebacate 6, polyisobutylene 1 & SAE 10W motor oil 10%. Accdg to A.L. Forchielli, it can be extruded in a width of 1/4 inch and adhere to horizontal metal surface to form a ribbon up to one inch wide and 50 or more inches long. When such a ribbon is initiated by the Corps of Engineers Special Blasting Cap, the resulting detonation cuts the metal into two pieces

Hercules Powder Co, Formal Progress **RI 2086** (1952) by W.R. Baldwyn, Jr; Army Contract DA-36-034-ORD-110 deals with Blasting Explosives (Dynamite Substitutes) (See also Ref 66, pp125–27). The following **MVD (Medium Velocity Dynamite)** compn is listed in Spec MIL-D-45413A (MU), 15 Aug 1962: RDX 75±1.0, TNT 15±1.0, Grade SAE No 10 Engine Oil plus Polyisobutylene 5±0.5 & Cornstarch 5±0.5%

In a more recent formula, cornstarch is replaced by Guar gum and Engine Oil by "Process Oil"

Properties of MVD (prior to replacement of cornstarch by Guar gum and use of "Process Oil"): Buff-colored plastic, tamped by Hall Packer machine in the same size cartridges as for LVD at density 1.1; it remains plastic to as low as –70°F.

Brisance by 200 Gram Bomb – 52.6 g sand crushed vs 48.0 g for TNT

Detonation Rate – 6000 to 6600 m/sec or 20000 ft/sec for machine-tamped, unconfined cartridges at density 1.1

Explosion Temperature – not listed

Friction Sensitivity – unaffected by fiber shoe and crackles with steel shoe

Gap Propagation in Air – max dist 1 inch for expln

Gas Volume – 945 cc/g

Heat of Explosion – 935 cal/g

Heat Test at 100°C – 0.62% loss after first 48 hrs, 0.12% loss after 2nd 48 hrs and no expln after 100 hrs

Hygroscopicity – satisfactory after 30 days at 71°C and 95% RH

Impact Sensitivity – PicArsn App 25 inches with 2 kg weight

Initiation Sensitivity – dry material explodes by No 6 Electric Cap and Corps of Engineers Cap; wet material gave 50% explns with Corps of Engineers Cap; minimum detonation chge 0.20 LA & 0.10 g Tetryl

Low Temperature Usage – satisfactory after storing for 1 day at –65°F

Power by Ballistic Mortar – 122% of TNT

Rifle Bullet Impact – burned in 10% trials and was unaffected in 90%

Strength – NG Equivalent, 60%

Vacuum Stability Test at 120°C – 0.94 cc/40 hrs

Uses of MVD: Excavation, demolition and cratering

Note: We presume that the properties of new formulation contg Guar Gum and Process Oil are similar to the above

PATR **2056** (1954) by J. Veliky & M. Epton deals with development of Nipolit–Type Explosives. One compn selected as the best of the experimental types studied on a lab & semi-plant scale, and designated as CP–1B consisted of:.

	%
RDX	35.9
Nitrocellulose (12.6% N) /graphite glaze	24.5
Nitroglycerin	22.8
Dinitroethylbenzene	10.0
Dibutylphthalate	6.6
Diphenylamine	0.2

It has favorable characteristics for use as an unconfined expl charge in military items. Comp CP-1B is thermally stable, relatively nonhygroscopic, and when unconfined is insensitive to friction, impact and rifle fire. It is superior to TNT in rate of detonation & brisance. The material is fairly rigid in large diameters but somewhat flexible in small diameters

The method of prepg CP-1B consisted of vibrating casting powder into a mold, introducing the nonvolatile, liq expl solvent by gravity thru the base of mold, and curing from 24–48 hrs at approx 65°C

PATR **2239** (1956) by C.E. Jacobson & B.J. Zlotucha deals with the development of Military Dynamite equivalent to Commercial Dynamite

The requirement for a military dynamite, a dynamite substitute containing no nitroglycerin and suitable for quarry and general construction work, was established at the end of World War II. Picramite I (70/15/9/3/3 RDX/talc/di-(2-ethylhexyl) sebacate (SAE 10 engine oil/polyisobutylene a composition developed by Picatinny Arsenal, met the performance requirements but was not acceptable because it could not be loaded on commercial dynamite packing machines. Picramite II (66/32/0.466/1.178/0.356 RDX/barium nitrate/polyisobutylene/di-(2-ethyl-hexyl) sebacate/SAE 10 engine oil), a modification of picramite I, was loaded into 1¼-inch by 8-inch dynamite cartridges by Company A on a Kimber Packing machine. Subsequent tests at Engineer Research and Development Laboratories (ERDL), Fort Belvoir, Virginia, revealed that the composition was unacceptable because it was too sensitive and did not function satisfactorily in underwater tests

About this time Company B developed on an Ordnance Corps contract a medium velocity blasting explosive to replace 60% commercial dynamite. The new composition, consisting of 75/15/5/4.85/. RDX/TNT/cornstarch/SAE 10 engine oil/polyisobutylene, was loaded without difficulty on a Hall dynamite packing machine. This composition, loaded in paraffin-sprayed paper cartridges (designated M1) was standardized as medium velocity (20000 fps) military dynamite after successfully passing field tests at ERDL. Later, when tests were being developed at Picatinny Arsenal for inclusion in the specification for medium velocity military dynamite, it was found that wet M1 cartridges (1¼-inch by 8-inch) could not be reliably initiated by a Type II special blasting cap. A dry cartridge, however, always initiated the wet cartridges that were tested

Desensitized RDX, one of the constituents in the explosive composition of the standardized military dynamite, was manufactured by coating the SAE 10 engine oil on the RDX in a water slurry at ambient temperatures without a wetting agent. No significant separation at ambient temperature or in 11 months at 65°C

PATR **2374** (1957) by H.W. Voigt dealt with development of Low-Velocity Military Explosives equivalent to Commercial Dynamites. The following Low-Velocity Dynamite (LVD) is described in AMCP 706-177 (Ref 66), p122–24

Composition of LVD: TNT 67.8, TPE (Tripentaerythritol) 8.6, (99.5/0.5 – RDX/1-MA dye) 17.5, (68/32 – Vistac No 1/DOS) binder 4.1 & Cellulose acetate (Grade LH-1, thru 40 mesh) 2.0%

Note: 1-MA dye is 96% pure 1-methylaminoanthraquinone; Vistac No 1 is low mol wt polybutene; DOS is dioctylsebacate

It is suitable for use with the Hall Dynamite Packer

Properties of LVD: Pink-colored plastic material loaded by tamping, using the Hall Packer machine, into cartridges 1½-inch diameter & 8-inches long to density 0.9 g/cc; it remains plastic to as low as –65°F

Brisance by 200 g Bomb – 40.5g sand crushed 48.0 g for TNT

Detonation Rate – 4397 m/sec or 14400 ft/sec for hand-tamped at density 0.9 unconfined cartridge

Explosion Temperature – ignites in 5 secs at 480°C

Friction Sensitivity – unaffected by steel or fiber shoe

Gap Propagation in Air – max distance 2.5 inches for expln

Gas Volume – 611 cc/g

Heat of Explosion – 625 cal/g

Hygroscopicity – 0.31% gain in 30 days at 71° & 95% RH

Impact Sensitivity – PicArsn App 22 inches with 2 kg weight

Initiation Sensitivity – dry material explodes by No 6 Electric Cap or by Corps of Engineers Cap; wet material explodes by Corps of Engineers Cap; minimum detonating chge 0.20 g LA and 0.15 g Tetryl

Low Temperature Usage – satisfactory after storing for 1 day at –65°F

Power by Ballistic Mortar – 92% of TNT

Strength – NG Equivalent-not given

Vacuum Stability Test at 135° – 0.90 cc/40 hrs

Uses of LVD: Excavation, demolition and cratering

PATR **2576** (1958) by J. Zlotucha deals with preparation of Low-Velocity Military Dynamite described in PATR2374 on a semiplant scale. It was loaded without difficulty into standard commercial paper cartridges on a Hall dynamite-packing machine. Test results indicate that its rate of detonation is 4660 m/sec for loaded dry cartridges and 4550 m/sec for loaded wet cartridges (24 hours in 10 feet of water). The cartridges have been reliably initiated by Type II special blasting caps after immersion in 10 feet of water for 24 hours, thereby meeting the minimum underwater initiation and propagation requirements established by USCONARC and the Corps of Engineers. Cartridges immersed in 4 feet of water for 72 hours were initiated by a type II special blasting cap in about 80% of the tests. Cartridges stored at elevated temperature for 60 days did not exude, and the friction sensitivity of the material was unaffected by such storage. The dynamite cartridges and bulk material were unaffected in rifle bullet impact tests

Note: LVD was never standardized or manufactured and there was not issued any Military Specification for this Dynamite, although a "Proposed" Specification was drafted at Picatinny Arsenal in 1961

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This is insufficient for low-NG Dynamites and it is necessary to use special waterproofing agents, such as Ca stearate, phlegmatized starch products, rye flour, cereal meals & PRP (petroleum, rosin, petrolatum); pp 12–13 (Advantages of Dynamites are: flexibility in composition, density, strength, pressure, sensitivity, water resistance, etc. Another unique property of Dynamites is their texture which allows easy cutting, loading, and handling in all kinds of operations. These properties are of special importance in commercial blasting of very small diameter boreholes. Disadvantages are: toxicity of NG & NGc, danger in handling, not as good fume characteristics as for some blasting expls (e.g. Nitrostarch expls), higher cost than for BkPdr, and high sensitiveness (which sometimes is desirable) 57) M. Giua, "Trattato di Chimica Industriale", UTET, Torino, **6**(I)(1959), pp338–45 (Dinamiti) 57a) S. Nauckhoff & O. Bergström, "Nitroglycerin och Dynamit. 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Note 1: Additional References on Dynamites and Their Substitutes are given after Analytical Procedures

Note 2: Explosives with individual names not beginning with "Dynamite" are listed under corresponding letters

DYNAMITES AND THEIR SUBSTITUTES; EXAMINATION, INCLUDING ANALYTICAL PROCEDURES

(This section was reviewed by Mr N. Liszt of Picatinny Arsenal)

The composition of Blasting Explosives may vary from that of a simple BkPdr (Black Powder) mixture of three ingredients to very complex mixts, such as Dynamites or their substitutes, contg as many as 20 ingredients

Their examination procedures may be divided into two parts: physical testing and chemical analysis. The former determines its value as an explosive from a purely physical standpoint, whereas the latter includes determination of composition and of certain physical characteristics having a direct relation to one of the ingredients of the explosive mixture

The knowledge of compn and properties of an explosive is required not only from the point of view of its use for certain work, but also for the purpose of its handling, storage and transportation

Regulations for transportation of explosives and of other dangerous materials were prescribed for US railroads by the ICC (Interstate Commerce Commission), under the authority of the federal law, Act of Congress, approved June 25, 1948. The ICC may utilize the services of "The Bureau of Explosives", organized by the American Railway Association in 1906 to secure safety in the transportation of expls

Regulations for transportation of dangerous articles by water are prescribed by the US Coast Guard, whereas rulings as to what products, and in what quantities, may be transported by air, are made by the US Civil Aeronautics Board. Packaging and labeling of air shipments are subject to the regulations of the ICC, as interpreted by the Bureau of Explosives

The current ICC regulations are published under the title: "Regulations for Transportation of Explosives and Other Dangerous Articles by Land and Water, in Rail Freight Service, and by Motor Vehicle (Highway), Including Specifications for Shipping Containers". Copies of these regulations are available thru The Bureau of Explosives, 63 Vesey St, New York, NY, 10007 (Ref 9a, pp XIII to XV)

Many explosives suitable for blasting and for other industrial purposes are already described in Vols 1, 2 & 3 (Refs 9, 10 & 13) of Encyclopedia, but for some of them, such as for "Blasting Explosives" (Vol 2, pp B211 to B212), no examination procedures were given, but will be included here. They were described, however, for the following explosives: Ammonals (Vol 1, pp A292 & A293), Ammonium Nitrate (Vol 1, pp A369 to A379), Black Powders (Vol 2, pp B176 & B177), Cheddites (Vol 2, pp C161 to C164) and Coal Mining Explosives (Vol 3, pp C368 to C378 – includes chemical tests, physical tests, gallery tests (for permissibility) and thermochemical calculations (Vol 3, pp C447 to C449)

The methods of testing industrial Dynamites described here are based on procedures used at the Bureau of Mines (Refs 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 12, 13 & 15) and on those by R.H. Pierson (Ref 11). We have no copies of the tests used by American Dynamite firms (such as DuPont & Hercules) but, accdg to Dr Van Dolah (Ref 15), they are similar to those used at the BurMines and to those described in Ref 11. The tests described in Ref 7, are the same BurMines procedures as given by Dr Storm in Ref 6. In Ref 8 are described by G.D. Clift changes (as of 1949) in BurMines procedures on analysis of Dynamites, as outlined to him by J.E. Crawshaw of Bruceton, Pa. These changes are included in Standard Methods mailed to us by Dr Van Dolah (Ref 15)

Analytical procedures for military types of Dynamites are described in Military Specifications listed as Ref 16

Analytical procedures developed at the Research Center of Hercules Powder Co, Wilmington, Delaware are listed as Ref 17

Caution

Before proceeding with the description of testing explosives contg NG and NGc, it is necessary to warn that both of these substances (especially NGc) cause headaches on inhaling their vapors and more severe headaches when handling them with bare hands. For this reason, a good ventilation system is recommended in buildings manufg and storing these expls and rubber (or plastic) gloves must be used in handling them

Preliminary Examination

Upon receiving a sample of expl for testing, it is desirable to record full information in regard to the size and wt of each cartridge, with a complete copy of any lettering that may appear on the wrapper. It is also advisable to record whether the cartridge has been re-dipped in a paraffin bath after being filled. This can usually be detd by carefully opening the wrapper and examining it. If there is a greater thickness of paraffin near the edge where the sheet overlaps, or if the overlapping edge is attached to the adjacent portions of the paper by means of an adhering deposit of paraffin, it may be assumed that the cartridge has been dipped

The next step is the detn of **gravimetric density (apparent specific gravity)** of a cartridge. A rapid, approximate, method is to calculate the volume by multiplying the circumference of the cartridge by its length and then to divide the weight of the cartridge by its volume. This method, however, is not accurate and not recommended when exact results are desired. If a cartridge is waterproof (such as when re-dipped in paraffin), its volume can be detd by displacement of water, but displacement of sand is more convenient because the method can be used for all types of cartridges. For this a glass cylinder (ca 30 cm high and 5 cm ID) is weighed empty (W_c) then full of distd water (W_w) and finally full of sea sand, passing a 60-mesh screen (W_s). The sand is leveled off even with the top by drawing across it a straight edge. By dividing the wt of sand (W_s-W_c) by the wt of water (W_w-W_c), the sp gr of sand is obtd. After drying the cylinder, the cartridge (previously weighed separately (W) is placed in it and sand is poured to fill the space around the cartridge and flush with the top edge of the cylinder. If the total wt of the ensemble is equal to W_t, then the wt of sand displaced (W_{sd}) is equal to:

$$W_{sd} = W_t - (W_c + W)$$

Volume of the cartridge, $V = W_{sd}/\text{sp gr of sand}$ and Gravimetric density of cartridge $\doteq W/V$

In making this test care should be taken that the cylinder is filled each time in exactly the same manner, the sand being poured in slowly and not packed by jolting or shaking. In practice this method has been found to be both rapid and exact (Ref 1, pp7–8 and Ref 4, p116). This test is mentioned in Vol 3 (Ref 13, pC369) under Coal Mining Explosives, Physical Tests

If visual examination of boxes contg cartridges indicates that there was some leakage of liquid components (such as of NG or NGc), the following qualitative **forty degree exudation test** can be used on cartridges without unwrapping them. Make small perforations in the wrappers at the ends of cartridges, place them vertically on a small wire tripod located in a small beaker and transfer the ensemble in an oven maintained at const temp of 40°C. See at the end of a 6-day period if any liquid, in the form of drops, leaked from the cartridges. If the test is positive, prepare the sample as described below and use 8 g of it, employing the **centrifuge test for exudation**, which is quantitative. This test is briefly described in Vol 2 of Encycl (Ref 10, pp C143-R & C144-L), but a detailed description, including the drawing of the centrifuge, is given in Ref 1, pp9–10

Note: Before the centrifuge test was developed by Dr W.O. Snelling, the Bur of Mines used the **pressure test for exudation**, which consisted in exposing a sample of explosive to a definite pressure produced by the weight of a lever arm, and determining the amt of liquid forced out of the expl. Many modifications of this test have been tried, in which absorbent materials like cotton or blotting paper have been used to hold the exudant, but none of them made the test reliable (Ref 1, p9)

Sample Preparation

The usual procedure for obtg an average sample of a lot is to take one cartridge from each box. Then the wrapper is carefully unrolled and each unbroken stick transferred to a sheet of parchment paper or dynamite shell paper. Any residual materials adhering to wrappers must be carefully removed and discarded and the cleaned wrappers saved for analysis. Care must be taken not to allow any paraffin to be mixed with the sticks. The next step is to cut off and discard, by means of a spark-proof knife, ca 1 inch of material from each end of the stick and after thoroughly mixing the remaining portion with a horn or a hard rubber spatula, a portion of material is transferred to wide-mouthed, rubber-stoppered (but no glass-stoppered) bottles Usually two 8-oz (or 16-oz) bottles are filled and the contents of one bottle is used for immediate analysis, while the other bottle is retained for checking purposes. No glass-stoppered bottles are used to avoid grinding of expl particles and no polyethylene containers are used to avoid producing static electricity (Ref 11, p1353 and Ref 15, Method No 28)

Note 1: Gelatin Dynamites, after removal of the wrapper or shell, should be quartered lengthwise, cut into ¼-inch sections and then blended. BurMines Method 28 recommends cutting the gelatin stick crosswise into 5–6 sections and then cutting each section into 1/8-inch discs, which in turn, are cut into 1/8-inch chunks. Then the chunks are blended

Note 2: If portions of the Dynamite for analysis are to be weighed out soon after bottling (say 1 hour), the bottle may be kept on its side and the portion withdrawn without remixing, otherwise it is necessary to remix the sample just before taking any of it because of the settling of NG. In general, however, frequent remixing is to be avoided because of possible loss of NG (Ref 11, p1353)

Note 3: In Ref 4, p117 it is stated that, if the analysis is not made at once, the whole sample is removed from the bottle and remixed before each use, as in many Dynamites there is considerable segregation on standing

PHYSICAL TESTS FOR PREPARED SAMPLES

Besides the *gravimetric density test* which is conducted with unwrapped cartridges, and the *exudation test* which can be conducted either with unwrapped cartridges or with prepd samples, the following stability tests are recommended before proceeding with complete chemical analysis

Abel or KI-Starch Test at 65.5°C. A brief description is given in Vol 1 of Encycl, p A2-L, while a detailed description including the drawing of apparatus and prepn of KI-starch paper, is in BurMinesBull 51 (Ref 1, pp10–12) (See also Ref 14, pp5–14 & 5–15)

International Test at 75°C. This test, recommended by the International Commission which reported to the 8th International Congress of Applied Chemistry, 1912, on "Methods for Testing the Stability of Explosives" is conducted as a supplement to the Abel Test. The test is described in Vol 1 of Encycl (Ref 9, p XVIII) and in Ref 1, p12

Another test which might be recommended is the Vacuum Stability Test described in Ref 9, p XXVI

If a sample fails stability tests, it shall be rejected and no further testing or chemical analyses are advisable

If the expl submitted to the BurMines is intended to be used in gaseous and/or dusty coal mines, it shall be tested (in addition to stability) for permissibility in galleries which are located at Bruceton, Pennsylvania. A brief description of these galleries and tests is given in Vol 3 of Encycl (Ref 13, pp C375 to C377), whereas a detailed description is found in BurMinesBull 346 (Ref 5, pp49–59)

Foreign galleries are described in Ref 13, pp C370 to C375

Other physical tests include:

Strength of Explosives can be detd by the *Ballistic Mortar Test*, which is briefly described in Vol 1 of Encycl (Ref 9, pVII) and in Vol 3 (Ref 13, p C370-L). A detailed description of the mortar and of the test as conducted at the BurMines is given in Ref 5, pp40–9. The so-called UDT (Unit Deflection Test) with the Ballistic Pendulum is, accdg to Dr Van Dolah, no longer used at the BurMines (See Ref 13, p C370-L)

Note: Trauzl Lead Block Test, such as described in Vol 1 (Ref 9, ppXXV & XXVI) is used in foreign countries (See also Ref 14, p 5–23)

Detonation Velocity can be detd by Dautriche or Chronograph Methods described in Vol 3 of Encycl, pp C311 to C318. Mettegang- and Dautriche Methods have been used at the BurMines and their description is given in Bull 346 (Ref 5, pp60–66)

Friction Sensitivity can be detd by *Pendulum Friction Apparatus*, as briefly described in Vol 1 (Ref 9, pp XIII & XIV) and given in detail in BurMines Bull 346 (Ref 5), pp79–84

Explosion-by-Influence Sensitivity or Air-Gap-Sensitivity has been detd at the BurMines by the *halved-cartridge gap method*, described in Vol 1 (Ref 9, pXIV) under Gap Tests. The test is mentioned in Vol 3 (Ref 13, p C370) and described in detail in Ref 5, pp59–60 (See also Ref 14, pp5-20 & 5-21)

If other tests are required, such as, for example, brisance, see Vol 2 of Encycl, pp B299 & B300

CHEMICAL ANALYSES OF DYNAMITES

These can be separated into qualitative and quantitative analyses or can be combined as was done by R.H. Pierson (Ref 11) or in Ref 7, Chap XI

The following preliminary test is described in Ref 4, p118

Dope-Size Test. It has been recognized that the granulation of the solid ingredients of an expl has an appreciable effect on its expl characteristics; a considerable variation might have as much effect as a change in chem compn, and so may cause failure in gallery tests for permissible expls. The "dope-size test" consists of treating a sample of expl (under a hood) with carbon bisulfide, in order to extract any NG, NGc, oils, sulfur and the nitro-substituted products. The sample must be large enough to leave at least 10 g of "dope" residue after its drying. The residue is kept in a desiccator and care must be taken to prevent its caking, which might take place if handling of dry material is done in the presence of moisture

Ten grams of this "dope" residue is weighed out, when atm humidity is low, and brushed thru a set of US Std 20 to 200 mesh-screens (See Table 1 in Vol 1 of Encycl, p A674). The amt caught on each is weighed and the percentage recorded. There is usually a little dust lost in the operation, so that the total sum is slightly less than 10 g. The difference is usually reported as part of the finest material. As a check against this detn, and so that the condition of the dope may be seen at a glance, the BurMines lab used to make a photomicrograph of each sample of dope. This would not be practical in a plant where there are hundreds of samples, nor would it be necessary if the ingredients themselves were tested with screens before use (Ref 4, pp118–19)

GENERAL METHODS OF ANALYSIS APPLICABLE TO COMMERCIAL BLASTING EXPLOSIVES INCLUDING DYNAMITES

I. MOISTURE DETERMINATION

Moisture can be approximately detd by drying in a desiccator over Ca chloride for 2–3 days an accurately weighed sample of ca 5 g spread in a thin layer on a watch glass. This method can be used only for samples contg small amts of NG and NGc and no other volatiles

For precise analysis of ordinary Dynamites, the "azeotropic distillation method with carbon tetrachloride" is used, while for Dynamites contg high AN content and low NG or those contg Al, the "Karl Fischer method" is used

IA. Moisture by Distillation Method

In this method the moisture is carried over as an azeotrope with carbon tetrachloride into a special trap (burette). Here the water separates into a layer on top of the CCl_4 and the amt distilled over is read in a graduated portion of the trap. Two types of traps, (also called "distillation traps") are described by Pierson (Ref 11, pp1289–90). The Hercules Powder Co type (shown here as Fig 1), known as "6-ml type", has a 6.0-ml graduated portion with the smallest intervals 0.05 ml and is numbered at each 1.0 ml division, whereas the E.I. DuPont de Nemours & Co type has a 1.0-ml portion with smallest intervals 0.02 ml (See Fig 1a)

The Hercules type tube is used by the US Bureau of Mines as described in "Standard Method No 28", issued on May 12, 1970 (Ref 15)

Procedure:

1) Obtain two cartridges of the explosive to be analyzed and place one of them on a 2-ft sheet of heavy dynamite paper (or waterproof tracing paper or parchment paper)

2) Cut and discard a 1-inch segment from each end of the cartridge, using a spark-proof knife or hard rubber spatula

Note A: Dynamites are subject to changes in compn while in storage, and in cartridges standing on end NG separates into the lower section

Note B: As moisture is absorbed in more exposed portions, such as the ends of cartridges, they are discarded in order not to obtain too high a moisture content

3) Remove the wrapper from the cartridge, taking care that no paraffin from the wrapper becomes mixed with the sample. Retain the explosive on the paper and either discard the wrapper or place it in an envelope if it is to be analyzed later

4) Combine the explosive removed from two cartridges and mix it thoroughly on paper by means of a hard rubber spatula

5) Weigh rapidly on a rough balance to 0.1 g a 50-g portion of explosive and transfer it into 250-ml round-bottom flask for moisture determination. Transfer the remainder of the sample into a 16-oz glass bottle and seal it with a rubber stopper

Note: For storage do not use glass-stoppered bottles or polyethylene containers for reasons explained above under "Sample Preparation"

6) Add 150 ml of dry CCl_4 to the flask contg the 50-g sample, attach the moisture burette to the top of the flask and install the reflux condenser above the burette (See Fig 2)

7) Clamp the apparatus on a hot water bath (such as modified Arthur H. Thomas Co Nos 9864 or 9865) maintained at 80° and reflux for 1.5 hours. The reflux should be at a rate which causes distillate to fall from the tip of the condenser at 2 or 3 drops per sec

Note: Pierson (Ref 11, pp1353–54) recommends 1 hr for Hercules tube (6.0-ml) and 30 mins to 1 hr for DuPont tube (1.0-ml)

8) Record the moisture burette readings at the lower part of the upper meniscus (A), and the upper part of the lower meniscus (B)

Calculation:

$$\% \text{ Moisture} = [(A-B)+(0.11)^*] \times 100 / \text{Wt of sample}$$

*(0.11) is constant correction in grams compensates for water present in CCl_4 at equilibrium plus the water forming the sides of the meniscus when the reading is taken

9) Filter the sample into a large bottle and dump the solids and filter paper into a paper sack

Note: In the description given by Pierson (Ref 1, p1289), it is recommended, for Dynamites and Propellants contg not more than 40% NG, that a 100-g sample be used for the 6.0-ml trap (Hercules) and a 50-g sample for the 1.0-ml trap (DuPont). For 100-g samples a 500-ml flask and the 200-ml CCl_4 are used in distillation in lieu of a 250-ml flask and the 150-ml CCl_4 used for 50-g samples. For samples contg more than 40% NG reduce the sample size one-

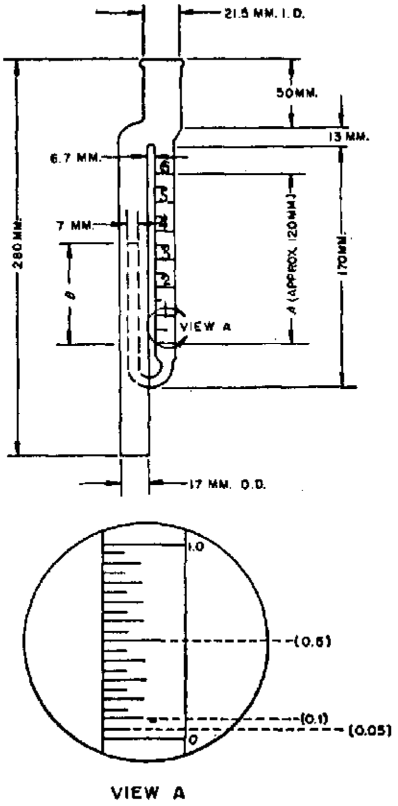


Fig 1. HERCULES TUBE
Length A must be 1.6 times as long as B

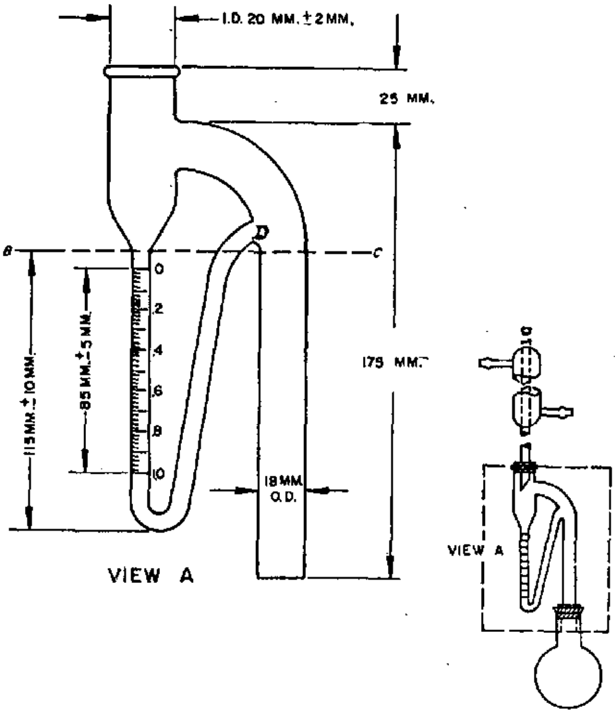


Fig 1a. DU PONT TUBE
Overflow point D must be at least 5mm above line BC

half. In case a sample contains a larger than usual amt of moisture, use less than 50 g samples

IB. Moisture by Karl Fischer Method

Accdg to Pierson (Ref 11, p1353), the Karl Fischer Method, as described on pp1290–93, should be used for Dynamites that contain aluminum, or those that are high in AN and low in NG

It can also be used for rapid detn of small amts of water in miscellaneous materials. The KF reagent consists of sulfur dioxide-iodine in pyridine-methanol solution. It is available in commerce in strength equivalent to 5–6 mg water per 1 ml of reagent. It can also be prepd as described below

Karl Fischer Reagent used in Method 101.4 of MIL-STD-650 (Ref 10a, p2) is prepd in the following manner: Turn on the hood (to avoid inhaling pyridine vapor which is odorous and toxic). Tare a clean and dry 1000-ml Florence flask, and, while leaving the flask on a balance pan, add 264 g of highest purity pyridine. Then invert the cylinder with liquid sulfur dioxide and run 61 g of it thru a glass tube into the flask. Finally, add a 664-ml portion of anhydrous methanol (not more than 0.02% moisture). Attach a Ca chloride drying tube and chill the flask in an ice-bath. When thoroughly chilled, add 65 g of CP resublimed iodine all at once. Swirl the flask, while still chilling it, until complete dissolution of iodine. Due to the rapid initial deterioration of reagent, it should not be standardized for 24 hours. After this one ml of reagent shall be equivalent to ca 0.0025 g water

The KF reagent described by Pierson (Ref 11, p436) for use in microtitrations contains methyl cellosolve instead of methanol

Karl Fischer reagent reacts immediately and quantitatively with water, accdg to the equation derived in 1939 by Smith, Bryant & Mitchell and later described by R.W. Freedman in AnalChem **28**, 247 (1956):

$$SO_2.C_5H_5N+I_2.C_5H_5N+C_5H_5N+CH_3OH+H_2O$$
$$\rightarrow C_5H_5N(H)SO_4.CH_3+2C_5H_5N.HI$$

Excess pyridine, iodine, and methanol are required for quantitative consumption of water. Sulfur dioxide reduces iodine quantitatively in the presence of excess water. The excess iodine present in the mixt makes a visual detn of the end point difficult. Therefore, the endpoint can be detnd electrometrically using the transistorized "dead-stop" end-point detector, described by R.W. Freedman in AnalChem **31**, 1287 (1959). Fig 1 of BurMines Method No 21 (Ref 15) shows the "Transistorized Dead-Stop End Point Detector". It is not reproduced here because there is no explanation of its wiring

It seems that Foulk & Bawden dead-stop end point apparatus is similar to the device used at BurMines. Foulk & Bawden app shown and described in Bofors Manual (Ref 8, p15) and here in Fig 2 consists of two bright platinum wire electrodes which are dipped into the soln to be titrated. They are joined in series with a galvanometer, resistance and a source of current ensuring a constant voltage of 0.2V between the electrodes. As long as the addition of KFR is continued and the end point is not reached, only a very small current flows between the electrodes because the cathode is polarized. At the equivalence point of the titration, however, free iodine appears in the soln with the result that a depolarization occurs, and a galvanometer makes a large deflection. The Pt electrodes should be placed right below the buret tip. This makes it easier to see when when the endpoint is approaching, because the local excess of KFR's cause momentary deflections of the galvanometer

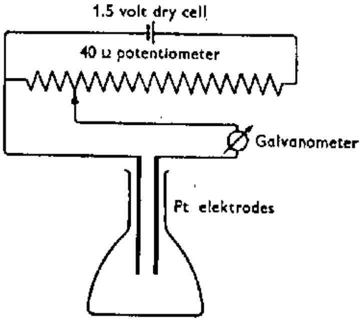


Fig 2. WIRING DIAGRAM

A similar wiring arrangement is shown in Method 101.4 of MIL-STD-650 (Ref 10a) and is reproduced here as Fig 3. A titration control arrangement which is an integral part of the "Aquatrator" of Precision Scientific Co is shown here on the left hand side of Fig 5

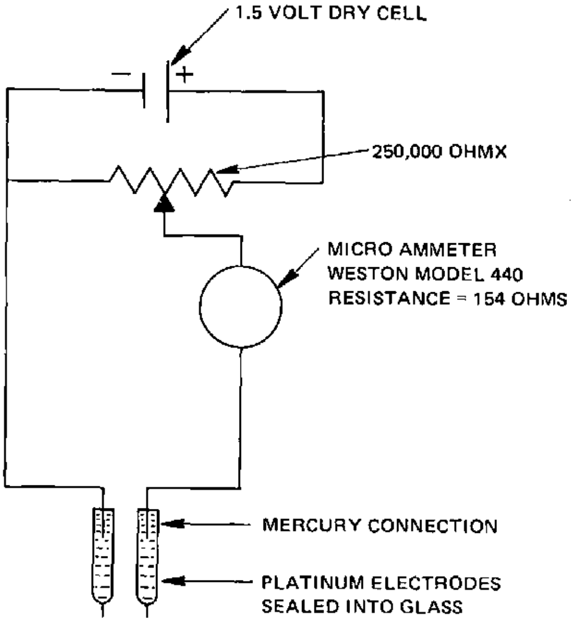


Fig 3. WIRING DIAGRAM

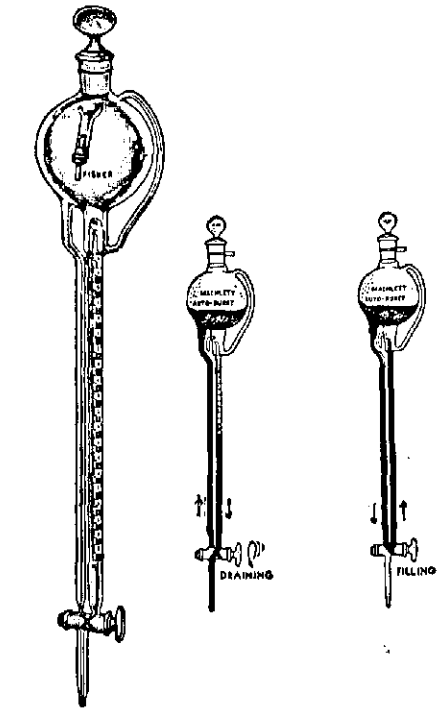


Fig 4. MACHLETT AUTOMATIC BURET

Apparatus for Karl Fischer Method

The apparatus used at the BurMines consists of one 10-ml Machlett buret which is automatic filling, self-zeroing and made of Pyrex glass. Its 1000-ml reservoir bulb is covered by black paper or black paint to protect the standardized KF reagent from white light. Fisher Scientific Co manufactures Machlett burets of sizes 2 to 50 ml. Fig 4 is a copy of Fisher's Fig 3-847. Their burets intended for KF reagent are of Pyrex amberized glass, while those for methanol-water soln are of clear Pyrex glass. Method 101.4 of MIL-STD-650 (Ref 10a) recommends the use of apparatus with amber and clear burets on the same stand. The same arrangement is an assembly manfd by the Precision Scientific Co, Chicago, Ill, under the name of Aquatrator (See Fig 5). It is provided with two Pyrex 25-ml Machlett burets (one of amberized glass), attached to 1000-ml bulb reservoirs and assembled on one support. Both burets are of 25 ml capacity, graduated in 1/10-ml divisions. The assembly is provided with a Mag-Mix Stirrer, a sample beaker 40 x 100 mm, its bakelite cover with holes for capillary tubes and for double Pt electrodes. Its control box has a receptacle for the Mag-Mix power cord, pilot lamp, on-off switch, calibration & run switch, a full scale voltage adjusting knob and a 0.25 microamp ammeter. Electrode outlets are located on the side panel, one red and the other black. Its line cord is three conductor with polarized plug. The price of the assembly includes a glass drying tube with a 4-hole stopper. The amberized glass reservoir with buret is for KF reagent, while the clear glass reservoir with buret is for standard water-in-methanol solution

Note: Accdg to Mr N. Liszt of PicArsn, the disadvantage of buret shown in Fig 5 is that the thin tubing located outside the reservoir is very easy to break during handling. This defect is absent in the buret made by Eck & Krebs of Long Island, NY (their catalog No 10105), because the corresponding tube is located inside the reservoir (See Fig 5a). The buret is automatic, self-filling by gravity and has a modified drying tube secured by small ground joints. It can be easily cleaned because its tip is detachable with 12/2 ball and socket joint

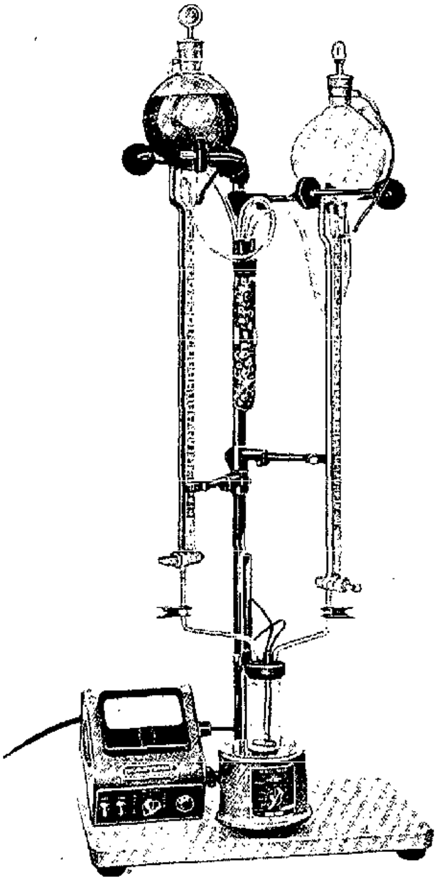


Fig 5. AQUATRATOR

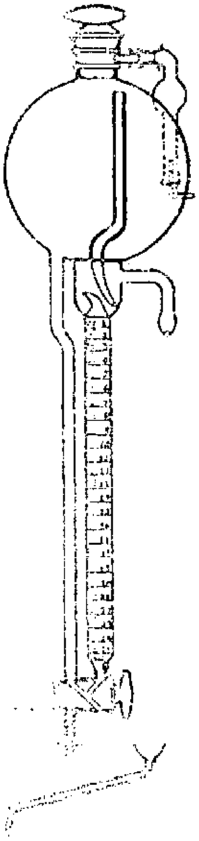


Fig 5a.

Reagents:

Standard Water-in-Methanol Solution. Pipet to a previously dried 1000-ml volumetric flask 2 ml of distd water, and immediately dilute to the mark with "anhydrous" methanol. Stopper, mix thoroughly and transfer to a reservoir protected with a drying tube. This soln has a water factor of ca 2.2 mg per ml

"Anhydrous" Methanol can be prepd as described in Bofors Manual (Ref 8, p17-L) in the following manner: Place 15g of Mg chips or clippings in a 5-liter flat-bottomed flask equipped with a long condenser, add ca 200 ml of methanol (with water content less than 1%) and 1.5 g of iodine. Heat the flask cautiously on a hot plate until the iodine has disappeared. If a heavy evolution of hydrogen does not occur add ca 1 g iodine and continue heating until almost all Mg has been dissolved. Then add ca 2800 ml of methanol, boil the soln under reflux condenser for 30 mins and, without cooling, quickly replace the reflux condenser with a Liebig cooler connected by ground joints with a receiver and, at the air outlet, with a drying tube. Distill off the methanol to the receiver under conditions such that any entry of air is prevented

Sodium Tartrate Dihydrate($Na_2C_4H_4O_6 \cdot 2H_2O$) shall have a water content of 15.66±0.05%. This can be established by heating a weighed sample at 105°C for 3 hours, cooling and weighing. If the water content is above or below the prescribed value, discard the bottle of material and replace it with another bottle (Ref 10a, p2)

Drying Tube. For its prepn, insert a glass wool plug into the bottom of the tube (tubing end). Pour (with tamping) 5A molecular sieve into the tube to a depth of ca 3.5 inches, and

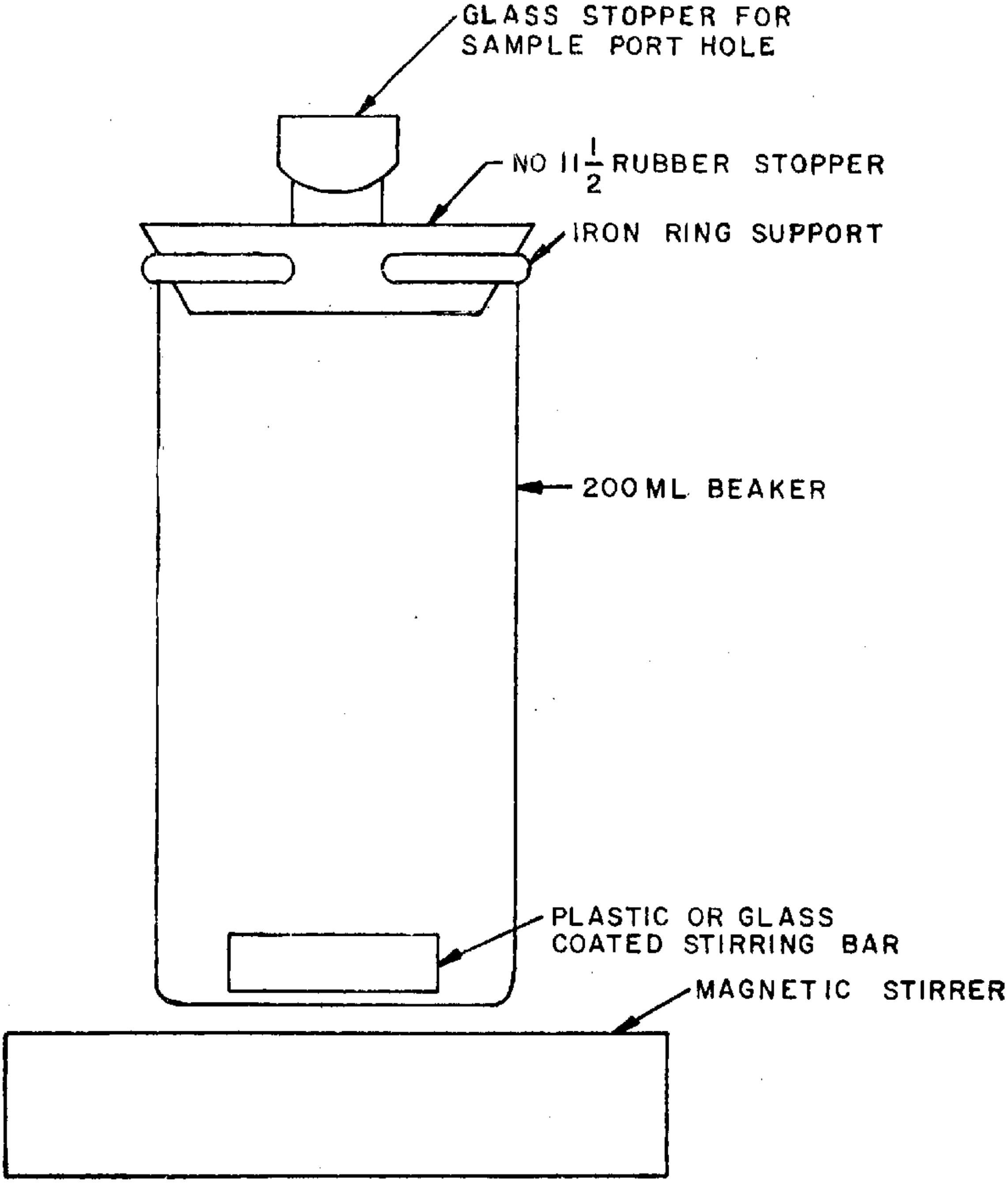


Fig 6. TITRATION ASSEMBLY

add a thin layer of glass wool followed by ca 1 inch of 10–20 mesh Drierite. Finally place a glass wool plug on top of Drierite and seal the open end of the tube with a rubber stopper fitted with a glass elbow (Ref 15, Method No21)

Titration Assembly for Karl Fischer Method

Description given in this write-up and Figs 3, 6 & 7 are from MIL-STD-650 (Ref 10a, Method 101.4). The method described by Pierson (Ref 11, pp1291ff), without including any illustrations, is very similar

The apparatus consists of two 25-ml Machlett burets (such as in Fig 4), one for KF reagent, another for water-in-methanol solution, both attached to the same stand as shown in Fig 5. The tips of burets are arranged to dispense into a titration cell (such as a tall-form beaker, shown in Fig 6) equipped with a rubber or Teflon gas-tight cover. This cover (See Fig 7) has coverings for extended buret tips, electrodes, drying tube and sample-inlet plug. When assembled all the openings in the lid are tightly sealed to protect the cell from atmospheric moisture

Pierson (Ref 11, p1291) uses oil-pumped cylinder nitrogen which is dried by passage thru a 3-ft tube of ca 1-inch diam filled with Drierite or Mg perchlorate, followed by a 1-ft tube contg P_2O_5 or Linde molecular sieve No 4A. The dry nitrogen is supplied to the titration cell, the burets, and the storage reservoirs for KF and water-in-methanol reagents. When

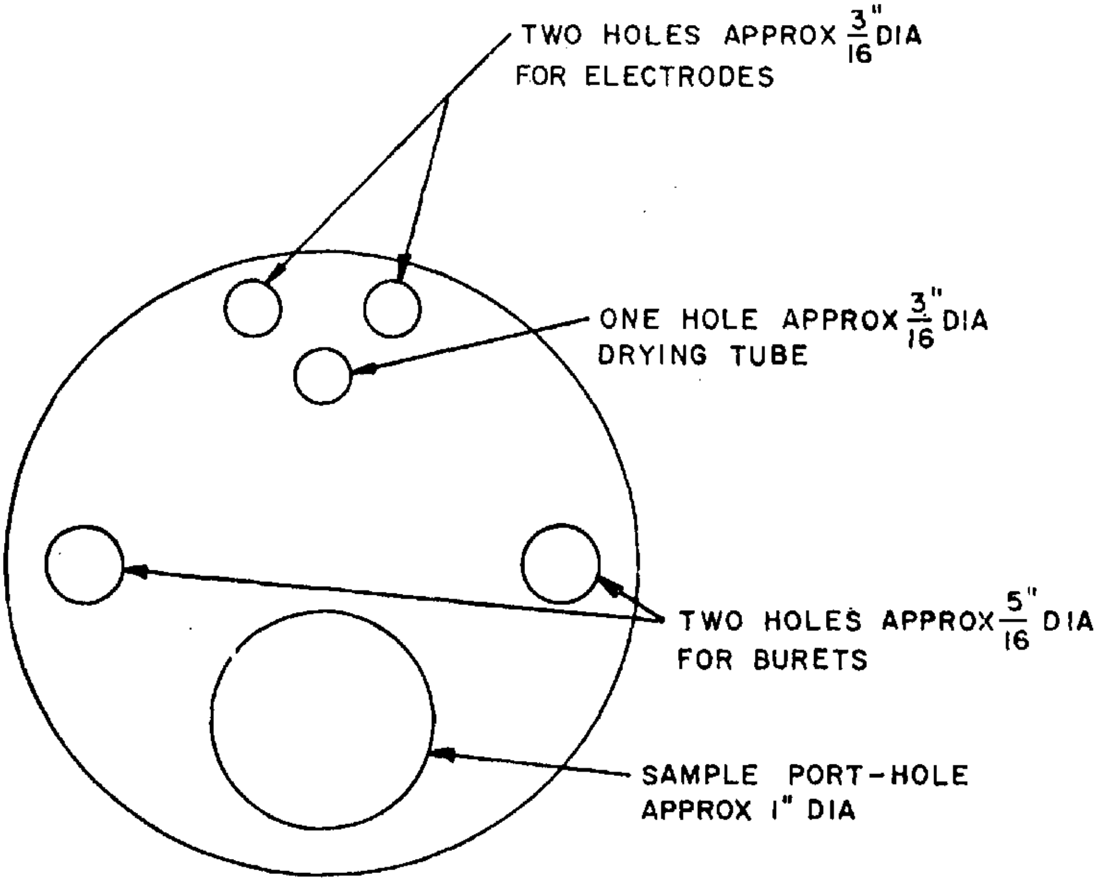


Fig 7. RUBBER STOPPER
Top View

not in use the nitrogen supply is blocked from the storage vessels by suitable stopcocks. During sample introduction, a very slow, steady stream of the dry nitrogen emerges from the sample inlet opening of the stopper. During titration, however, the cell is preferably under very slight pressure of dry nitrogen and, if the cell is not quite gas-tight, a very slow stream of dry nitrogen must be passed into the upper part of the cell to prevent the ingress of atmospheric moisture

Platinum electrodes used in Method 101.4 (Ref 10a, p1) are wires 0.016-inch diam, sealed-in-glass, with mercury connections. A magnetic stirrer with plastic- or glass-coated stirring bar is used (See Fig 6) for stirring the liquid in the cell. The cell is emptied by means of a glass suction tube and a suitable trap connected to a vacuum line

Detection of the end-point by the dead-stop electrometric procedures is described in Ref 8, p15 and in Ref 15, Method No 21. In Method 101.4 of MIL-STD-650 (Ref 10a, p3), the arrangement is shown in Fig 3. Here the end-point is defined as the point at which one drop of the standard water-in-methanol soln causes the micro-ammeter needle to waver and then slowly drift from the upper end of the scale toward the lower end in not less than 15 seconds. In using this method a stop-watch is required

Standardization of KF Reagent. As KF reagent is not stable but continuously changes its water equivalent towards lower values, the reagent must be standardized on the same day the titration of moisture is to be run. Bofors Manual (Ref 8, p17-L) recommends placing 10 ml of standard water-in-methanol soln in the titrating cell (beaker) and titrating with KF reagent to the change of color specified

Calculation:

Water Equivalent of KF Reagent in mg/ml =

$$\frac{\text{mg of } H_2O \text{ in } 10\,\text{ml water-in-methanol soln}}{\text{ml of KF reagent used}}$$

Pierson (Ref 11, p1292) recommends placing 25 ml of dry methanol in the titration cell, adding KF reagent in moderate excess, and back-titrating with water-in-methanol soln. Then make a series of titrations of 10-ml portions of KF reagent with water-in-methanol soln

Repeat the titrations until the ratios from 3 successive titrations agree within 5 ppt. Then standardize the KF reagent against water or sodium tartrate reagent

Titration Against Weighed Water is described by Pierson (Ref 11, p1292)

Titration Against Sodium Tartrate Dihydrate. Pierson (Ref 11, p1292) recommends placing 50 ml of dry methanol in the titration flask, adding an excess of KF reagent and back-titrating with water-in-methanol soln. Transfer 0.40 to 0.44 g of $Na_2C_4H_4O_6.2H_2O$ (weighed by difference from a small vial) to the titration cell, stir the mixture until the salt is nearly dissolved, and then add KF reagent in small increments until soln is complete. Continue to add KF reagent until a moderate excess is reached, and back-titrate with water-in-methanol soln. Convert the weight of dihydrate used to milligrams of water (each mg of dihydrate contains 0.1566 mg H_2O) and calculate the water equivalent (E) of the KF reagent in mg/ml as follows:

$$E = \frac{\text{mg of water required}}{[\text{ml of KF reagent} - (r \times \text{ml of water-in-methanol soln})]}$$

r = ratio of volumes, KF reagent per water-in-methanol

Determination of Moisture in Dynamites

Arrange the apparatus as described under "Titration Assembly for Karl Fischer Method" and proceed by the method of direct titration as described in Ref 8, p17-R

Procedure:

a) Transfer a sufficient amount of "anhydrous" methanol (prepd as described above) to cover platinum electrodes and stirring bar in the 200-ml beaker, shown in Fig 6

b) Cover the beaker with stopper (shown in Fig 7), pass the leading wires of the electrodes thru two holes, insert drying tube in its hole and insert the tips of burets in their holes

c) Consume the water in anhydrous methanol by titrating with KF reagent exactly to the end point, indicated by the "dead-stop" method, using an apparatus shown in Fig 2. Disregard the buret reading

d) Transfer into beaker thru the one-inch hole in stopper an accurately weighed quantity of sample contg 0.01 to 0.10 g of water (Ref 8, p17), or 0.030 to 0.060 g, as recommended by Pierson (Ref 11, p1293)

e) Immediately close the hole and start the magnetic stirrer. Continue stirring until complete dissolution of the sample

f) Titrate with KF reagent to the "dead-stop" end point and calculate from the equation:

$$\% \text{ Water content} = \frac{A \times E}{10 \times W}$$

where: a = ml of KF reagent used for the titration (after introducing the sample in the flask)

E = Water equivalent of KF reagent in mg H_2O/ml, determined as described above

W = Weight of sample in g

The above method of direct titration is described in Ref 8, p17-R, while Pierson (Ref 11, p1293) recommends the method of "back-titration". He continues to add KF reagent until a moderate excess is reached after passing the direct end point. Then he back-titrates with water-in-methanol soln contd in the 2nd (clear glass) buret

Microdetermination of Water by Titration with Karl Fischer Reagent is described by Pierson (Ref 11, pp436–38)

II. ETHER SOLUBLE CONSTITUENTS DETERMINATION IN COMMERCIAL BLASTING EXPLOSIVES INCLUDING DYNAMITES

(Using Bureau of Mines Method No 30, Issued 8 June, 1970)

This procedure is used for the quantitative determination of organic nitrates [NG (Nitroglycerin), NGc (Nitroglycol or Ethyleneglycol Dinitrate) and DEGDN (Diethyleneglycol Dinitrate)], aromatic nitrocompounds [MNT (Mononitrotoluene), DNT (Dinitrotoluene) and TNT (Trinitrotoluene)], paraffin oil, phthalates, coatings from AN (Ammonium Nitrate), such as paraffin, grease (oils & resins naturally occurring in carbonaceous filler) and sulfur (of which only part is soluble)

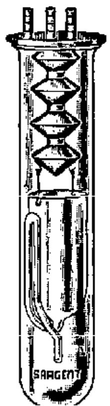
Ether (diethyl) soluble materials are extracted from explosive mixtures using a Wiley-Richardson Extraction Assembly, ASTM, represented in Fig S-31505, p486 of E.H. Sargent & Co catalog (See our Fig 1). An alundum crucible is used in place of extraction cup S-31615 or S-31625. If elemental sulfur is present, a 2nd extraction is made with CS_2 The extracts are combined, the ether evaporated, and the residue is weighed to obtain a value for total ether solubles, sulfur and moisture. If nitro compds are absent, the residue is treated with acetic acid to remove NG ("explosive oil"). The grease is then separated from sulfur by treating with acetone. If nitrocompounds are present, only half of the ether-CS_2 extract is used for detn of grease and sulfur, while the other half is treated, after evaporation of solvents, with methyl sulfuric acid to remove NG. The ether and acid layers are separated and the ether is evaporated. The residue is extracted with acetone to remove the grease and sulfur and the nitrocompds weighed

A flow diagram for the analysis is shown in Figure 2

All operations, especially with CS_2, must be conducted under a hood with good ventilation

I. Extraction with Ether and Carbon Bisulfide

Note A: Diethyl ether must be free from peroxides in order to avoid possible expln during extraction or subsequent evaporation of ether extract. The presence or absence of peroxides may be detected by adding to 10 ml ether in a bottle provided with a glass stopper: 1 ml of soln prepd by dissolving in 90 ml of distd w, 5 g CdI_2 and



S-31505

EXTRACTION ASSEMBLY
Wiley-Richardson, ASTM
Figure 1

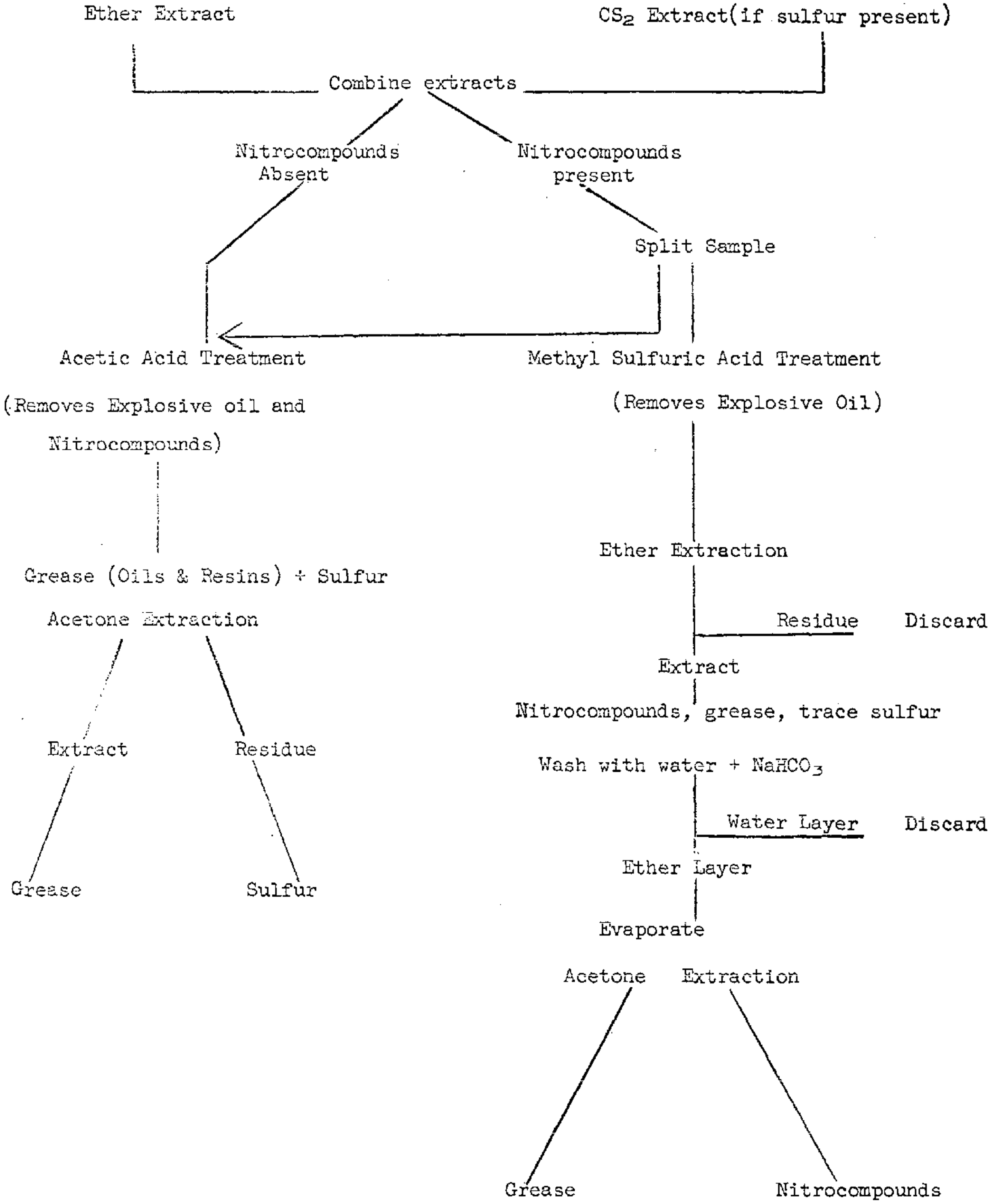


Figure 2 Determination of Ether Solubles

5 g KI. Then the bottle is stoppered and shaken several times during one hour, while keeping the bottle in the dark. Absence of orange color indicate that the ether is free of peroxides. Such ether is usually ACS grade, anhydrous (Ref 11, p1354)

Extraction Procedure:

a) Clean the 35 ml alundum crucible by boiling it in dilute HCl (1:1), followed by washing with tap water and then distd w. Dry the crucible in an oven at 110°C and then heat in a muffle furnace at 1500°F. Finally cool it and tare

b) Fill the tare crucible with original sample contg moisture, to ca 3/8 inch from the top and weigh (Pierson recommends taking exactly 10 g of sample and 40 ml of ether)

c) Place the crucible in the holder of the extraction apparatus (See Fig 1) and lower the condenser so that the crucible is just within the flask

d) Wet the outside of the crucible and then fill it with ether from a wash bottle

e) Add ether to the flask until the total volume used is 50 ml

f) Lower the condenser and crucible until the top of the condenser rests on the upper (ground) surface of the glass tube

g) Start to circulate an adequate amount of tap water thru the condenser and adjust the position of the tube in the hot water bath so that the ether boils steadily but gently

Note B: Too rapid boiling may cause the solvent to overflow the crucible and carry some solid material with it

h) Reflux for 2–6 hours

Note C: A 2-hour extraction period is allowed for Permissible, Straight, Semigelatin and Ammonia Dynamites. Four to six hours are required for Gelatin Dynamites and Blasting Gelatin

i) Remove the Wiley-Richardson apparatus from the water bath and remove the alundum crucible with residue. Place it on the edge of the steam bath to drive off residual ether

j) Dry in an oven for 2 hours at 95°C or overnight at 70°, cool in a desiccator and weigh. Save the crucible with residue for water (or water-alcohol) extraction described in Standard Method No 31

k) The loss of weight in the crucible is considered as total ether solubles, plus moisture

Calculation 1:

$$\% \text{ Ether solubles} + \text{Moisture} = \frac{(\text{Wt loss of cruc}) \times 100}{\text{Wt of sample}}$$

l) Transfer the ether extract from extraction tube of the Wiley-Richardson apparatus to a tared 100-ml beaker

m) If much sulfur is present, as shown by crystallization in the extraction tube, replace the alundum crucible (with residue) into the Wiley-Richardson extractor, add ca 30 ml CS_2, place in a water bath and extract for 2 hours

Caution: Avoid breathing the CS_2 vapors

n) Remove the extractor from the water bath, cool, and place the crucible on an edge of a steam bath to evaporate residual CS_2

o) Dry in an oven for 2 hours at 95° or overnight at 70°, cool in a desiccator and weigh

p) *Calculation 2:*

$$\% \text{ Ether} - CS_2 \text{ solubles} + H_2O =$$

$$\% \text{ Ether solubles} + S + H_2O = \frac{(\text{Wt loss of crucible}) \times 100}{\text{Wt of sample}}$$

Save the crucible with residue

q) Transfer the CS_2 extract from tube of Wiley-Richardson apparatus to the tared 100-ml beaker contg ether extract (See step l above)

r) If nitrocompounds are supposed to be present, make up the above ether-CS_2 extract to 200 ml with ether and withdraw 100 ml into a beaker for the detemination of nitrocompds (See step 3a). Transfer the remaining 100 ml into another beaker and treat the solution as described below

2. Explosive Oil, Grease and Sulfur Determinations

a) Evaporate the solvents of the 2nd 100-ml portion of steps 1q & 1r on a steam bath but remove the beaker as soon as boiling ceases

Note D: The bath should be of such a temperature as to produce slight boiling of the ether-CS_2 soln and the air-jet should produce only a perceptible ripple on the surface. Alternately, the solvent can be evaporated under the hood overnight

b) Wash the residue in the beaker 5 or 6 times with 70% acetic acid using 5-ml portions until all the explosive oils (NG + NGc) and nitrobodies are removed from the grease and sulfur. Decant each time into a 7-cm No 4 Whatman filter paper held in a small funnel. Use a glass rod with flattened end to break up any lumps of sulfur and to scrub the sides of beaker. Do not use a rubber policeman because it would be slightly sol in acetic acid

c) Wash the filter several times with acetic acid, allow it to drain thoroughly, and then wash with distd w to remove the acid

d) Remove the contents of the filter paper into a tared 100-ml beaker using the flattened end of a glass rod, followed by ether to dissolve any grease and sulfur adhering to the paper

e) Evaporate the ether, dry the beaker and contents at 75°C for 1 hour, cool in a desiccator and weigh

Note E: Examine the residue. Any sulfur present will appear as brittle and crystalline yel lumps. Grease can be recognized by its characteristic feel

f) *Calculations 3 and 4:*

$$\% \text{ Grease(oils \& resins)} + S = \frac{(\text{Wt gain of beaker}) \times 100}{\text{Wt of sample}}$$

% Explosive Oil (NG+NGc) = Calculation 2(Step 1p) minus the sum of: Calculations 3 & 4(Step 2f), Nitrobodies(Calculation 7, step 3) and % Moisture (as determined by Standard Method No 28, described here as the first item)

g) Add ca 5 ml of ether to the beaker of step 2e, agitate, and decant the soln into a tared 50-ml beaker. Rinse the first beaker with three 5-ml portions of ether, decanting each time into the 2nd beaker. Evaporate the ether, dry for 1 hr at 75°C and weigh

h) *Calculations 5 and 6:*

$$\% \text{ Grease} = \frac{(\text{Wt gain of 50-ml beaker}) \times 100}{\text{Wt of sample}}$$

% Sulfur = Calculation 3(Step 2f) minus % Grease

Note F: Sulfur can also be detd by oxidizing it with bromine water, followed by pptg with Ba chloride soln and weighing as $BaSO_4$

3. Nitrocompounds Determination

a) Take the 100-ml ether-CS_2 extract retained for the detn of nitrocompounds in the first beaker of step 1r, evaporate the solvents under the hood in the manner described in step 2a and in Note D. Remove the last traces with a current of dry air

b) Add slowly to the residue at RT 10 ml of methyl sulfuric acid and warm the beaker gently until the explosive oil (NG+NGc) dissolves and for an addnl 15 mins

c) Cool the soln and pour into a small separatory funnel contg distd w cooled in ice-water

d) Add to the cold liquid in the funnel 10 ml of ether, close the top of the funnel and shake gently

e) Allow to separate into two layers and draw off the acid (bottom) layer into a small beaker and save

f) Draw off the ether layer into a 2nd beaker (ca 100-ml) and save

g) Return the acid layer from the beaker of step 3e to funnel and repeat the extraction four times using a 10-ml portion of ether each time in the manner described in steps 3d, 3e & 3f

h) Discard the acid layer and save the beaker with the combined five ether extracts

i) Transfer the combined ether extract into a small, clean separatory funnel and wash by shaking with 10 ml of distd w. Draw off the aq layer, add a 2nd portion of w and neutralize the acid (to litmus paper), by adding $NaHCO_3$ soln, a few drops at a time. Draw off the neutral water and discard

j) Draw off the ether extract into a 50-ml beaker and rinse the funnel into the beaker with two 5-ml portions of ether

k) Evaporate the ether on a steam bath and remove the last traces with a current of dry air

l) Treat the residue (which consists of nitrocompounds with small amts of grease and sulfur) with three or four 5-ml portions of acetone and filter thru a 5.5-cm filter paper into a small tared beaker

Note G: Most of the grease and sulfur remains on the filter or on the walls of the beaker, but a little dissolves in acetone

m) Evaporate the filtrate of step 3l on the steam bath to dryness, but heat only 5 mins after the odor of acetone is gone to avoid loss of volatile compds

n) Cool and weigh the beaker

o) Repeat the extractions with acetone until all grease and sulfur are removed and constant wt is attained

p) *Calculation 7:*

$$\% \text{ Nitrocompounds} = \frac{(\text{Wt gain of beaker}) \times 100}{\text{Wt of sample}}$$

III. WATER (OR WATER-ALCOHOL) SOLUBLE CONSTITUENTS. DETERMINATION IN COMMERCIAL BLASTING EXPLOSIVES

(Using Bureau of Mines Standard Method No 32, Issued 17 July 1970)

To this group of constituents usually belong the following substances: AN, Na nitrate, Na chloride, water soluble portions of carbonaceous materials and traces of Ca and Zn salts. A small amt of Amm chloride which may be present as an impurity, is not determined separately

Extraction Procedure:

1) Transfer the contents of an alundum crucible containing the residue from the ether extraction of the sample (step i of Method II) to a 250-ml beaker

2) Place the crucible in a funnel using a filter adapter and insert the funnel into a suction flask

3) Extract with a total of 200 ml of 1:1-ethanol-water soln (by volume) making extractions in 10-ml portions, as follows:

a) Add each portion to the residue in the beaker and stir. Macerate any large prills with a flattened rod and allow to settle

b) Decant the supernatant liquid into the crucible while using gentle aspiration to filter the liquid

c) Finally, transfer the undissolved material quantitatively to the crucible and give it a 10-ml rinse. A drop of final rinse evaporated on a glazed surface should not leave any deposit

Note A: Extract with water, alone, if starch is not present (indicated by a cloudy film, not crystals, in the ether extraction tube of Standard Method No 30)

Note B: Extract NS (Nitrostarch) expls with water alone, because NS is somewhat sol in alc-water mixture

4) Transfer extract from the suction flask to a 250-ml volumetric flask, rinse the suction flask several times with w (transferring the rinses into the volumetric flask) and dilute to the mark. Remove any foam rising in the neck of the flask with a drop of ether

Note C: Since expls contg flour are difficult to extract with water, mix the entire ether insol residue with water and transfer quantitatively to a 250-ml volumetric flask. Dilute to the mark, mix, and let settle. Save an aliquot portion of the clear, supernatant liquid to det the salts present

5) Dry the crucible and contents, after extraction (Step 3), at 95°C or overnight at 70°C, cool in a desiccator and weigh. Calculate:

% Water (or water-alcohol) solubles =

$$\frac{\text{Loss of weight x 100}}{\text{Sample wt (50 g)}}$$

and save the crucible with residue for the acid extraction, using "Standard Method No 33"

Note D: Water-sol constituents that may be present are listed at the beginning of Standard Method No 32. The total amt of salts present in soln of step 4 should equal the loss of water due to the water extraction, or show an "unidentified" value of not more than 1.0%

6) Transfer the extract of step 4 to a clean, dry bottle with a screw cap, and use the liquid for taking aliquots in detn of AN and of other salts

7) *Ammonium Nitrate by Formaldehyde Method.* It is described in a paper by R.W. Williams et al, AnalChem **29**, 1356–57 (Sept 1957). It is based on the reaction:

$$4NH_4NO_3 + 6HCHO \rightarrow (CH_2)_6N_4 + 6H_2O + 4HNO_3$$

Prepare reagents for this method as follows:

Formaldehyde-water solution. Mix 50 ml of 38% formaldehyde with 50 ml of distd w in a 400-ml beaker, add 4 drops of 1% phenolphthalein or thymolphthalein indicator (0.8g per 100 ml ethanol) and neutralize with carbonate-free 0.5N NaOH soln dropwise to a pink color (pH 8.6) on glass-calomel electrode system

Standard sodium hydroxide, 0.5N. Dissolve 20 g of carbonate-free NaOH in 500 ml CO_2-free water, add enough Ba chloride to more than 2 liters distd w to precipitate carbonate present, let the Ba carbonate settle, and filter into a 1 liter volumetric flask. Standardize the NaOH soln by adding it from a buret into a soln of 2 g of potassium acid phthalate (weighed accurately on an analytical balance), and four drops of phenolphthalein indicator in 75–100 ml CO_2-free water contained in a 150-ml beaker until the first appearance of a pink end point

$$\text{Normality of NaOH soln} = \frac{1000 \text{ x (Wt } KHC_8H_4O_4)}{204.2 \text{ x (ml NaOH)}}$$

Standard sodium hydroxide, 0.1N. Dilute 50 ml of 0.5N NaOH to 250 ml with CO_2-free distd w

Standard hydrochloric acid, 0.1N. Dilute 8.6 ml of concentrated acid (11.7N) to one liter, transfer to a 50-ml buret and run the acid into 25 ml of 0.1N NaOH, diluted with 75 ml dist w and contg 4 drops of 1% phenolphthalein in a 250-ml beaker. Continue titrating to the disappearance of the pink color

$$\text{Normality of HCl} = \frac{(\text{ml NaOH})(\text{N NaOH})}{\text{ml HCl}} = \frac{25.00(\text{N NaOH})}{\text{ml HCl}}$$

Procedure for Ammonium Nitrate. Transfer a 25-ml aliquot from the bottle of step 6 to 100 ml of the neutralized formaldehyde-water soln in a 400-ml beaker, add 4 drops of 1% phenolphthalein (or thymolphthalein) indicator and titrate to a pink color with 0.5N NaOH. Add 2 ml of NaOH in excess, cover the beaker, and let stand for 5 mins. Back-titrate with 0.1N HCl to pH 8.5 (glass-calomel electrode system)

$$\%\ \text{AN} = [(\text{ml NaOH} \times \text{N NaOH}) - (\text{ml HCl} \times \text{N HCl})] \times \frac{0.08005 \times (\text{Tot extract}) \times 100}{[(\text{ml Aliquot}) \times (\text{Sample wt})]}$$

where Tot extract = 250 ml, Aliquot 25 ml & Sample wt 50 g

8) *Sodium Chloride (Adsorption Indicator Method).* It is described in the book of H.A. Laitinen, "Chemical Analysis", McGraw-Hill, NY (1960), pp212–14

Reagents:

Standard silver nitrate, 0.1N. Dissolve 16.989 g of the salt in 1 liter of chloride-free water and standardize against pure NaCl using the adsorption indicator method

Fluorescein indicator. Dissolve 0.2 g in 250 ml ethanol. It is not satisfactory for solns of chloride content of less than 0.005 (such as in drinking water). Solutions 0.025N in chloride may be titrated with dichlorofluorescein as indicator

Dichlorofluorescein indicator. Dissolve 0.1 g in 125 ml of ethanol. It is preferred to fluorescein because it can be used even in acid solns (pH>4), whereas fluorescein is used in neutral or slightly basic solns (to pH 10)

Procedure for NaCl:

Pipet a 20-ml aliquot from the bottle of step 6 into a 125-ml Erlenmeyer flask. Add 6 drops of dichlorofluorescein indicator and titrate with std 0.1N Ag nitrate, with vigorous shaking, until the ppt suddenly turns reddish

$$\%\ \text{NaCl} = (\text{ml AgNO}_3) \times (\text{N AgNO}_3) \times (0.05845) \times (\text{Tot extract}) \times 100\ /\ [(\text{ml Aliquot}) \times (\text{Sample wt})]$$

where ml of $AgNO_3$ is detd by titration, Tot extract = 250 ml, Aliquot = 20 ml and Sample wt = 50 g

Note E: The color change and coagulation coincide in dil solns. With higher concns of chlorides, the ppt tends to flocculate about 1% before the end point

Note F: A disadvantage of adsorption indicators is that Ag halides are sensitized to the action of light by a layer of adsorbed dye. For best results, the titration should be carried out with minimum exposure to light

9) *Total Nitrates*

a) Transfer the 50-ml aliquot of step 6 to a tared porcelain or glazed silica evaporating dish and evaporate to dryness on a steam bath

b) Dissolve the residue in distd w, add 5 ml of concd nitric acid, and again evaporate to dryness

c) Repeat step 9b when NaCl is present

d) Place the evaporating dish on an electric hot plate and heat carefully until ammonium salts are volatilized. Do not heat too rapidly to avoid loss of salts by spattering

e) Heat carefully with a Mekker burner to remove the last trace of ammonium salts and to burn off the carbonaceous material. A distinct yellow in the residue, while hot, indicates the presence of ZnO

f) Cool the dish and repeat step 9b to insure complete conversion of Na and Zn to nitrates

g) Dry at 105–110°C for 3 hrs or overnight

h) Cool in a desiccator and weigh as total nitrates

$$\%\ \text{Total Nitrates} = \frac{(\text{Wt of residue in dish}) \times (\text{Tot extract}) \times 100}{(\text{ml Aliquot}) \times (\text{Sample wt})}$$

where Tot extract = 250 ml, Aliquot = 50 ml and Sample wt = 50 g

10) *Zinc Oxide.* If ZnO is present (see step 9e), separate the Zn nitrate from the Na nitrate as follows:

a) Dissolve the residue of step 9h in distd w and precipitate the Zn with Na carbonate soln

b) Filter on a Gooch crucible, rinse ppt with w, ignite and weigh as ZnO

$$\%\ \text{ZnO} = \frac{(\text{Wt of residue after ignition}) \times (\text{Tot extract}) \times 100}{(\text{ml Aliquot}) \times (\text{Sample wt})}$$

where Tot extract = 250 ml, Aliquot = 50 ml and Sample wt = 50 g

Note: Practically all of the Zn salt present in the explosive will be extracted by water. The amt found in acid extraction (Standard Method No 33) is negligible. ZnO can also be detd as follows:

c) Gently heat the residue of step 9h over a burner until the evolution of nitrogen oxides from the decompn of Zn nitrate has ceased

d) Cool and weigh the remaining material as Na nitrate and Zn oxide

e) Treat with w, filter the insol ZnO on a Gooch crucible, and wash

f) Dry the crucible, ignite, cool and weigh

$$\% \text{ ZnO} = \frac{(\text{Wt after ignition}) \times (\text{Tot extract}) \times 100}{(\text{ml Aliquot}) \times (\text{Sample wt})}$$

where Tot extract = 250 ml, Aliquot = 50 ml and Sample wt = 50 g

11) *Sodium Nitrate.* Calculate it from equation:

$$\% \text{ NaNO}_3 = \% \text{ Total nitrates} - [(\% \text{ZnO} \times 2.33) + (\% \text{NaCl} \times 1.494)]$$

where % Tot Nitrates is obtd in step 9h, % ZnO in step 10f and % NaCl in step 8

12) *Sodium Salts as Sodium Sulfate.*

a) Transfer a 50-ml aliquot from the bottle of step 6 to a tared porcelain or glazed silica evaporating dish and evaporate to dryness on a steam bath

b) Dissolve the residue in distd w, add 6 ml of 1:1 (by volume) sulfuric acid and evaporate nearly to dryness

c) Transfer the dish to an electric hot plate and continue heating until the ammonium salts are volatilized and the excess of sulfuric acid driven off

d) When white fumes are no longer visible, heat the dish carefully over a free flame for a few minutes and then at the full heat of a Mekker burner

e) Transfer the dish to a muffle furnace at 900°C for 30 mins, cool in a desiccator and weigh

f) Repeat the muffle furnace heatings to const wt and calculate as follows:

$$\% \text{ Na}_2\text{SO}_4 = \text{Evaporated residue} - [(\% \text{ZnO} \times 1.984) + (\% \text{NaCl} \times 1.215)],$$

where % ZnO is obtd from step 10f and % NaCl from step 8

Calculate: $\% \text{ NaNO}_3 = \% \text{ Na}_2\text{SO}_4 \times 1.197$ and compare the value with that obtd in step 11

IV. ACID SOLUBLE CONSTITUENTS DETERMINATION IN COMMERCIAL BLASTING EXPLOSIVES

(Using BurMines Standard Method No 33, Issued 21 August 1970)

The purpose of this procedure is to determine the acid-soluble constituents of commercial Blasting Explosives. To this group of constituents belong starch and antacid Ca carbonate. Infrequently Zn oxide is present, but it appears to a large extent in the water extract when ammonium salts are present. Metallic aluminum powder may also be present

For the tests described below use for acid extraction the residue in the alundum crucible from the water (or water-alcohol) extraction, as described in step 6 of "Standard Method No 32"

Before proceeding with the extraction, test for the presence of starch in the above residue by placing a small portion in a 50-ml beaker, adding 10 ml of distd w and bringing it to a boil. Then cool and add a drop of iodine (1% in methanol) soln. Appearance of a blue color indicates the presence of starch

Note A: Rice hulls may give a faint starch test from small rice particles, and corn meal will give a positive test also. Determine the presence of these materials by examination of the sample to be extracted

1. Extraction and Determination of Acid Solubles in Absence of Starch

a) Transfer residue from the alundum crucible (step 6 of Standard Method No 32) to a 250-ml beaker and add to this the material used for the starch test

b) Extract the material in a beaker with 100–150 ml of dilute (1:10) hydrochloric acid, adding it in small portions, and stirring with a policeman. Usually six increments of acid are sufficient. Pour the supernatant liquid after each addition into the crucible and apply suction

c) Quantitatively transfer the material remaining in the beaker to the alundum crucible, using a policeman and a stream of water

d) Rinse the material in the crucible with water to remove all excess acid

e) Dry the crucible in an oven at 100° for 4 hours (or overnight at 85°C), cool in a desiccator and weigh

f) Calculate the loss in wt as percentage acid soluble

$$\% \text{ Acid Soluble} = \frac{(\text{Wt loss of crucible}) \times 100}{\text{Wt of original sample (50 g)}}$$

g) Retain the alundum crucible with residue for the carbon tetrachloride extraction if sulfur is present and for the acetone extraction if NC is present

2. Extraction and Determination of Acid Solubles in the Presence of Starch

a) Transfer the residue from the alundum crucible (step 6 of Standard Method No 32) to a 400-ml beaker and add to this the material used for the qualitative starch test

b) Add 150 ml water and 25 ml of 1:10 HCl and digest for 2.5–3 hrs at a temp between 80 & 90°C. The beaker may be either suspended in a constant temp bath, or in a larger beaker filled with hot water and placed on a steam bath or hot plate

Note B: The starch which is present in an expl in either powdered or flaked form is hydrolyzed by this operation to a soluble dextrin. The correct strength acid should be used and the specified temp and time of heating must be strictly adhered to in order to prevent the removal of other ingredients of dope, such as bagasse, corn meal, rice hulls, woodpulp, etc

c) Filter thru the original alundum crucible, wash the residue with hot water and dry at 100° for 4 hrs (or overnight at 85°)

d) Cool in a desiccator, weigh and calculate as in step 1f

e) Save the crucible with residue for further operations as outlined in step 1g

3. Extraction and Determination of Acid Solubles in Presence of Starch and Aluminum

a) Transfer the residue from the alundum crucible (step 6 of Standard Method No 32) to a 400-ml beaker and add 100 ml of distd w

b) Add concd hydrochloric acid, using 5 ml of acid for samples contg up to 2% of aluminum with no antacid, and using 10 ml of acid for samples contg up to 3.5% aluminum and 0.5% antacid

c) Stir the mixture, breaking up all lumps, cover with a watch glass and heat almost to boiling on the steam bath

d) Continue heating with occasional stirring until all the aluminum is dissolved and filter, while hot, thru the above alundum crucible

e) Rinse the contents of crucible with hot dist w, while applying suction

f) Cool the filtrate to RT and transfer it to a 250-ml volumetric flask. Make up to the mark and save for further operations

g) Dry the crucible with residue at 100° for 4 hrs (or at 85° overnight), cool in a desicc tor and weigh

h) Calculate % acid solubles as in step 1f

4. Determination of Aluminum as Oxide

a) If aluminum is present, pipet a 50-ml aliquot of 250-ml acid filtrate (step 3f) into a 250-ml beaker, make the soln slightly ammoniacal and heat to boiling. Add several drops of phenol red to see that the color of the mixtures becomes red (pH 8) and is not yellow (pH 6.4)

b) Filter the hot slurry thru S&S 589 Black Ribbon or Whatman 41-H paper and save it, together with ppt for step 4c. Transfer the feebly ammoniacal filtrate from suction flask to a 400-ml beaker and save for antacid determinations of step 6a or step 7a

Note C: The compd precipitated from a hot soln is crystalline or granular and filtrate goes thru filter readily, whereas pptn from a cold soln gives a flocculant aluminum hydrous oxide which clogs the filter

c) Dissolve the ppt from the paper (See step 4b), in the smallest amt of 1:1 hydrochloric acid and collect the chlorides in a 250-ml beaker

d) Make this soln slightly ammoniacal, heat to boiling and add a few drops of phenol red to see that the color of mixture becomes red

e) Filter the hot mixture thru S&S 589 Black Ribbon or Whatman 41-H paper, fold the paper with the ppt and place it in a tared porcelain or platinum crucible. Discard the filtrate

f) Burn off the paper carefully and ignite

g) Cool the crucible in a desiccator and weigh it together with the aluminum oxide

$$\% \text{ Aluminum} = \frac{(\text{Wt } Al_2O_3) \times (0.52923) \times (\text{Tot extract}) \times 100}{(\text{Sample wt}) \times (\text{Aliquot})}$$

where Tot extract = 250 ml, Aliquot = 50 ml and Sample wt = 50 g

5. Determination of Aluminum as 8-Hydroxyquinoline

a) Repeat steps 4a thru 4e, incl and dissolve aluminum hydroxide ppt in the smallest amt of 1:1 hydrochloric acid

b) Warm the soln and add a slight excess of 8-hydroxyquinoline, $Al(C_9H_6ON)_3$ (ca 1 ml for each 3 mg of aluminum)

c) Slowly add to the warm soln 2N ammonium acetate soln (15.42 g per liter) until a ppt forms (some ppt may have formed before the addn). Add 25 ml for each 100 ml soln to insure complete pptn

d) Allow the mixture to stand for an hour without further heating and if the supernatant liquid is yellow, enough reagent has been added

e) Filter thru a medium porosity filtering crucible and wash thoroughly with cold distd w

f) Dry at 120 to 140°C, cool and weigh. The ppt is $Al(C_9H_6ON)_3$ and contains 5.87% Al

% Aluminum =

$$\frac{[\text{Wt } Al(C_9H_6ON)_3] \times (0.0587) \times (\text{Tot extract}) \times 100}{(\text{Sample wt}) \times (\text{Aliquot})}$$

where Tot extract = 250ml, Aliquot = 50ml and Sample = 50 g

6. Determination of Antacid (Calcium Carbonate)

a) Take the filtrate in 400-ml beaker (See step 4b) and heat to boiling

b) Add 7ml of saturated ammonium oxalate (soly 11.8 g per 100 g water at 50°), stir well and place on the steam bath for an hour or more. This will ppt Ca as oxalate, CaC_2O_4

c) After allowing to stand overnight, filter the slurry thru a paper, collecting the filtrate into a 2nd 400-ml beaker. Wash the 1st beaker and the filter with hot w to remove excess Amm oxalate. It is not necessary to remove at this time the CaC_2O_4 which adheres to the 1st beaker

d) Save the filter paper with adhering CaC_2O_4 and also the filtrate in beaker No 2 for further determinations

f) Add 100ml distd w and 5ml concd sulfuric acid to the 1st beaker and scrub the walls to dissolve any adhering CaC_2O_4

g) Heat to boiling and titrate to a pink color with 0.1N $KMnO_4$ (See Note D below)

h) Transfer the filter of step 6d contg the remainder of CaC_2O_4 to the 1st beaker, stir and continue titration until a faint pink color persists for one min

i) Calculate as follows:

$\% CaCO_3$ =

$$\frac{(\text{ml } KMnO_4) \times (\text{N } KMnO_4) \times (0.05005) \times (\text{Tot extract}) \times 100}{(\text{Aliquot}) \times (\text{Sample wt})}$$

where Tot extract = 250ml, Aliquot = 50ml and Sample = 50 g

Note D: For *preparation* of standard 0.1N potassium permanganate, dissolve 3.250 g$KMnO_4$ in 1 liter of hot distd w and allow the soln to stand 12 hrs or longer. Filter thru a fine porosity, sintered glass crucible to remove MnO_2 and store in a dark bottle

Note E: For *standardization* of $KMnO_4$ soln, transfer 0.3g of dried (at 105°) Na oxalate to a 600-ml beaker and add 250ml dilute (5:95) sulfuric acid, previously boiled for 10–15 mins and cooled to 27°. Stir until oxalate has dissolved and gradually add $KMnO_4$ soln, with stirring, at the rate of 25–35ml per minute until the appearance of a pink color. Usually 39–40ml are required. Heat the soln to 55–60° and complete the titration until a faint pink color persists for 1 min. Add the last 0.5–1ml dropwise, allowing each drop to become decolorized before the next is introduced

$$2KMnO_4 + 5Na_2C_2O_4 + 8H_2SO_4 =$$
$$K_2SO_4 + 2MnSO_4 + H_2O + 10CO_2$$

$$\text{Normality of } KMnO_4 = \frac{\text{Wt } Na_2C_2O_4}{(\text{ml } KMnO_4) \times (0.067)}$$

7. Determination of Antacid (Zinc Oxide)

a) Before proceeding with this test, see if there is any zinc present in the acid extract: Take 10ml of acid extract (See steps 1a & 1b or 2a & 2b), neutralize it with dilute NaOH soln and then add 1ml of glac AcOH. Pass in H_2S and if a white ppt forms, Zn is present. When ammonium salts are present, a portion, or nearly all (99%), of the Zn will be present in the water (or water-alcohol) extract (See Standard Method No 32, step 10) and none might be found in acid extract

b) Take the filtrate of step 4b located in 400ml beaker No 2 and add 50ml of water (water-alcohol) extract (See Standard Method No 32, step 10)

c) Acidify with hydrochloric acid, add 25ml of mercuric ammonium thiocyanate precipitating reagent and allow the precipitation to take place overnight

Note: For prepn of precipitating reagent dissolve 32 g Amm thiocyanate and 27 g mercuric chloride in 500ml distd w. Remove any residue by filtering and store the reagent

d) Collect the ppt of step 7c on a tared Gooch crucible and wash it with a soln prepd by dissolving 10ml of precipitating reagent in a liter of distd w

e) Burn the crucible with ppt to ZnO and calculate:

$$\% ZnO = \frac{(\text{Wt of ppt in crucible}) \times (\text{Tot extract}) \times 100}{(\text{Aliquot}) \times (\text{Sample wt})}$$

where Tot extract = 250ml, Aliquot = 50ml and Sample = 50 g

V. CARBON TETRACHLORIDE AND ACETONE SOLUBLE CONSTITUENTS DETERMINATION IN COMMERCIAL BLASTING EXPLOSIVES

(Using Bureau of Mines Standard Method No 34, Issued 15 September, 1970)

In this method sulfur is determined by carbon tetrachloride extraction of the residue remaining after the ether, water, and acid extractions of the sample. The sum of the sulfur found here and by the ether extraction procedure (Standard Method No 30) represents the total sulfur content. The sulfur removal is followed by acetone extraction for the determination of NC (Nitrocellulose)

Note A: If NS (Nitrostarch) is present it is extracted by the acetone and may be identified by slowly evaporating a drop of acetone extract on a microscope slide and examining the ppt as described in Bull 96 (Ref 2, pp74–5). It is unlikely, however, that both NC & NS are present in the same sample

1. Carbon Tetrachloride Extraction (for Sulfur)

a) Stand in a 100-ml beaker the alundum filtering crucible which contains the residue from the acid extraction (See steps 1e or 2e in this description of procedures of Standard Method No 32)

b) Add enough carbon tetrachloride to come within 1/8 inch of the top of the crucible both inside of it and outside in the beaker

c) Heat on a steam bath placed under the hood (because the fumes of CCl_4 are very toxic), until the solvent in the beaker and in the crucible evaporates to a vol of ca 5 ml

d) Remove the beaker from the steam bath and place the crucible contg the wet residue in a crucible holder attached to a filter flask

e) Apply suction, remove the crucible, stand it in a 2nd 100-ml beaker, add CCl_4 as in step 1b and repeat operations of steps 1c and 1d

f) Wash the residue in the crucible attached to suction flask with 20 ml hot CCl_4 and test for completeness of extraction by evaporating the last portion of the rinsing filtrate on a watch glass. A residue indicates incomplete extraction and steps 1c, 1d & 1f must be repeated

g) Heat the crucible contg the residue in an oven (place under a hood) for 2 hrs under 70°C, cool in a desiccator and weigh

h) Calculate wt loss of residue in the crucible and:

$$\% \text{ Sulfur} = \frac{(\text{Wt loss in crucible}) \times 100}{(\text{Original sample wt} = 50\,\text{g})} + \% \text{ sulfur (found by Standard Method No 30)}$$

2. Acetone Extraction (for Nitrocellulose)

a) If sulfur is absent use the alundum crucible after acid extraction (See steps 1e or 2e of Standard Method No 30), whereas if sulfur were present and removed by extraction with CCl_4, use the crucible after step 1g of Standard Method No 33

b) In each case, transfer as much as possible of the residue from the crucible to a 400-ml beaker, add 100 ml acetone and stir to break up any agglomerates

c) Heat to boiling on a steam bath and continue heating until all the NC is dissolved. This will be evidenced by the disappearance of all lumps and free dispersion of all insol material. It might take six hours for dispersing the gels

d) Remove from the steam bath and add enough acetone to fill the beaker within 1/2 inch of the top. Cover with a watch glass and let stand until the solids have settled to the bottom

e) Decant slowly, in a running stream, the clear supernatant acetone soln from the 400-ml beaker into an 800-ml beaker contg 200 ml distd w, while stirring it rapidly. Save the 400-ml beaker with residue

f) Discontinue stirring, allow to stand, and observe if there are any white stringy fibers of NC floating on the surface of the aqueous acetone

g) If NC is present, make addnl digestions and decantations until a negative test is obtd

h) Rinse the alundum crucible (See step 2b) with 100 ml of warm acetone and quantitatively transfer the residue from the 400-ml beaker (See step 2e) to the above alundum crucible

i) Rinse the residue in the crucible with 100 ml of warm acetone, dry the ensemble at 100° for 2 hours, cool and weigh

j) Calculate:

$$\% \text{ NC} = \frac{(\text{Wt loss of acetone extraction} \times 100}{(\text{Original sample wt} = 50\,\text{g})}$$

k) Save the alundum crucible with residue for identification of adsorbents and determination of ash

VI. EXAMINATION OF ABSORBENTS AND DETERMINATION OF COMBUSTIBLE MATERIALS AND ASH IN COMMERCIAL BLASTING EXPLOSIVES

(Using Bureau of Mines Standard Method No 35)

(Issued 10 October 1970)

These procedures discuss the examinations of residue after all the required extractions and tests of Standard Methods Nos 30, 32, 33 & 34 are completed. Identification of absorbents in the sample before extraction is usually difficult because of the presence of explosive oils. If addnl sample is required for examination, extract 10 g of the original material for 2 hours with ether in Wiley (or equivalent) apparatus

1. Determination of Absorbents

a) Transfer the residue remaining in the alundum crucible after acetone extraction (See steps 2a to 2k of Standard Method No 34) to a 4-inch Petri dish

b) Pick it apart with a dissecting needle or small metal spatula and examine the material with a powerful hand lens or a low power microscope (25–50X)

c) Compare the material to a set of sample absorbents (or to their photomicrographs) commonly used in Dynamites

d) Estimate the proportion of the constituents of absorbents found by microscopic examination

e) The percent absorbents present is calcd after the value for the % combustibles is obtained (See steps 2a to 2d, incl)

Note A: Following are absorbents which might be found in US current permissibles: apricot meal; apricot shell pulp (hard structure and light brown in color); bagasse(Fibrous residue from cane sugar; it is pithy and can range from small to large irregular chunks); clay; coal, ground (Black or brownish with the coarse particles having lustrous faces); corn flakes, corn flour & corn meal; crude fiber, Fuller's earth, guar flour & guar meal; guhr; ivory nut meal (Hard structure when pressed under a spatula. Appears under magnification as white chunks, some of which retain the brown outer layer of the original nut. Fine, irregular striations can be distinguished on the larger particles); kaolin; oat hulls, ground (Lengthwise sections of the hull are readily distinguished by their flexible character; they are usually light buff in color); rice hulls; sawdust (granular and free-flowing); tamarin seed, vegetable ivory & vegetable meal; woodpulp (Has long, thin, cohesive fibers)

Note B: Microscopic examination of absorbents is described in BurMinesBull 96 (Ref 2, pp74–5). For prepn of glass microscopic slides, it is sufficient to spread a small quantity of dry residue uniformly over the surface of slide in a thin layer so that individual particles are separated from each other. Then the slide is placed directly under the objective, without a cover glass. The most suitable magnification is found by trial. A number of samples of different materials that may be found in the insoluble residue are shown in Plates I, II and III of Ref 2 under magnifications of 10 to 150 diameters. The plates are not reproduced here

2. Determination of Combustible Material and Ash

a) After the examination of the absorbents, return all the material from the slides and Petri dish to the alundum crucible of step 1a

b) Ignite with a burner or in a muffle furnace just to a red heat, taking care to prevent loss from too rapid combustion

Note C: If the carbonaceous material is consumed slowly, stir it occasionally with a Pt wire. The presence of coal may be confirmed during this operation, as coal usually burns with a yellow flame and with a good deal of smoke of characteristic odor

c) When no more particles of carbon are visible, cool the crucible in a desiccator and weigh

d) Calculate as follows:

$$\%\ \text{Combustibles} = \frac{(\text{Loss on ignition}) \times 100}{(\text{Sample wt} = 50\,\text{g})}$$

% Total absorbents = A+(B−C), where A = % combustibles, B = % loss on water-alcohol extraction (See step 5 in Standard Method No 32); C = sum of the percentages of the determinations in water-alcohol extractions

Note D: If the total of "C" equals "B", which it should, then "A" is not changed. If "C" is less than "B", then "A" is higher and may have to be rechecked for incomplete water extraction

3. Determination and Examination of Ash

a) Calculate the amount of residue (ash) in the crucible and determine

$$\%\ \text{Ash} = \frac{(\text{Wt of residue in the crucible}) \times 100}{(\text{Sample wt} = 50\,\text{g})}$$

b) If % ash is above 1.5%, a microscopic examination must be made. The ash usually consists of kieselguhr, Fuller's earth, bentonite, ferrosilicon, iron oxide and metals insol in hydrochloric acid. When a Dynamite contains coal, the ash is relatively high and is a red color due to the presence of ferric oxide. A high ash may indicate that extraction with water-alcohol (Standard Method No 32) or with acid (Standard Method No 33) was *not* complete

EXAMINATION OF COMMERCIAL DYNAMITES BY THE METHODS OF HERCULES POWDER COMPANY AND BY THE METHODS DESCRIBED BY R. PIERSON

The description given below is based on Methods D90-0 to D90-11 with Appendix, issued by Hercules Powder Co, Research Center (Refs 17 & 18) and also on descriptions given by Pierson in Ref 11, p1350–64

The flow-sheet of analysis given here as Fig 1 is taken from Ref 11, p1354. Actually it is a modified copy of Fig 1, listed in Ref 17, Method D90

Pierson's Procedure D–1 (Ref 11, p1353 & Ref 17, Method D90–1) *Moisture by Carbon Tetrachloride Distillation.* See Section 1A in this write-up under GENERAL METHODS OF ANALYSIS APPLICABLE TO COMMERCIAL BLASTING EXPLOSIVES, INCLUDING DYNAMITES. *Procedure D–1a.* Moisture by Karl Fischer Method (Ref 11, p1354 & Ref 17, Method D90–1a). See Section 1B under GENERAL METHODS, etc

Pierson's Procedure D–2. *Ether Extraction* (Ref 11, p1354 & Ref 17, Method D90–2). See Section II of this work. Extraction of sulfur from residue left after ether extraction in the Hercules method is done with CCl_4 instead of CS_2, as in BurMines Standard Method No 30

Pierson's Procedure D–3. *Treatment of Ether Extracts* (Ref 11, pp1355–57 & Ref 17, Method D90–3. See "Flow Sheet" of BurMines Standard Method No 30 listing tests which differ from those described in Refs 11 & 17 (Compare with Section II of this work)

Nitrocompounds and Low-Freezing Compounds obtained as a residue after evaporation of the ether from its extract depend somewhat on the Dynamite compn. If the residue is essentially solid [indicating absence of explosive oil (NG+NGc)], omit treatment with methyl sulfuric acid (See below). If sulfur is absent, omit the filtration. If, however, both NG and sulfur are present, proceed as follows:

Transfer ca 1.0g accurately weighed, of the filtered dried expl oil (from the ether extraction into a tared 100-ml beaker. Add 10ml of freshly prepd methylsulfuric acid (obtd by gradually adding, under the hood, 5ml of concd sulfuric acid to 5ml of pure methanol, while stirring and keeping the temp below 40°C). Heat the beaker and contents on a steam bath for 30 mins to destroy the NG. Cool, add 40ml of distd w and transfer the soln to a 250-ml separatory funnel. Extract with 50ml of pure

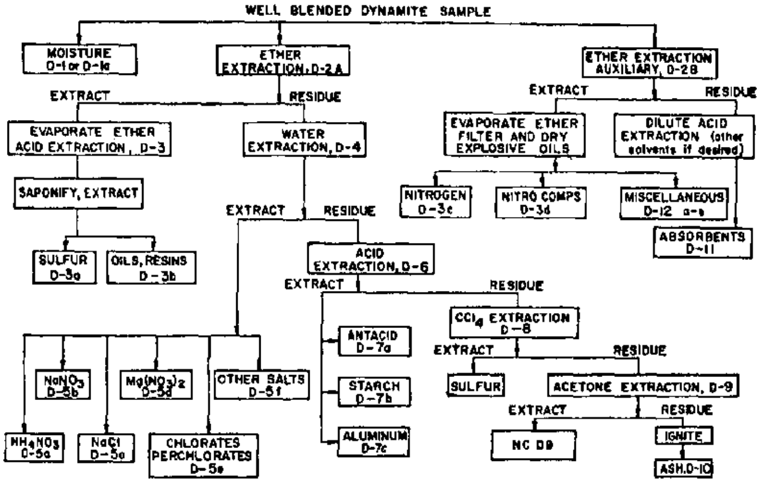


Fig 1 Dynamite Analysis Flow Sheet

ether, and draw the water layer off into a 2nd separatory funnel. Repeat the extraction with two 25-ml portions of ether, and then combine the three extracts. Wash the combined extract first with 25 ml of saturated aqueous $NaHCO_3$ soln, followed with 25 ml of distd w Discard the wash solns. Transfer the ether extract (100 ml) to the original 100-ml beaker, evaporate to dryness on a steam bath, finish drying by placing in a desiccator for 30 mins at RT and weigh

Hercules Method D90–3d gives grams of LF (low-freezing) compds and their percentage is calcd from the equations:

$$\%\text{ LF compds} = \frac{\text{grams of LF compd x 100}}{\text{grams of oil taken (1.0 g)}} \quad \text{and}$$

% LF compds on basis of original sample =

$$\frac{\%\text{Ether sol}-(\%\text{Moist}+\%\text{Oils}+\%\text{Resins}+\%\text{S})\text{x}\%\text{LF compds}}{100}$$

Pierson (Ref 11, p1356) gives the following equation:

% Nitrocompounds =

$$\frac{\text{grams Nitrocompound x \% Ether soluble}}{\text{grams of explosive oil weighed}}$$

Colorimetric Tests for Nitrocompounds (Ref 11, pp1356–57)

a) Dissolve 0.1 g of Nitrocompds in 10 ml of acetone contd in a test tube and add 5 ml of 5% aqueous NaOH soln. Mix and observe the color after 3–5 mins. Acidify the soln with 1:1 HCl, mix and again observe the color after 3–5 mins. DNT gives a blue color before acidification and greenish-yellow after acidification. TNT gives intense wine red color before and after acidification. MNT does not produce any change in its original yellow color

Note 1: When both DNT & TNT are present, the blue color of DNT will be obscured by the red color of TNT. In the method described in Ref 7, Vol 3, Chap XX, p2, it was suggested to dissolve a small sample in acidified ethanol and add a few drops of KOH soln. If the color is betw red and violet (the mixture is present), wait until the DNT coloration fades and the liquid becomes red. If the color is violet and fades completely on standing or warming, DNT is present alone. If the color is red and does not change on standing, TNT is present alone. This method can be used as a quantitative test

b) Dissolve another 0.1 g portion of the Nitrocompounds in 10 ml of acetone contd in a test tube and add 5 ml of methanol & 5 ml of filtered $(NH_4)_2S$ soln. Mix, allow to stand for 3–5 mins and observe the color. DNT does not produce any coloration, while TNT gives a red color

Note 2: It is advisable to make, simultaneously, colorimetric tests on known samples of DNT and TNT, and compare in colorimetric tubes the exact colors formed

Note 3: The explosive oils may be analyzed for nitrate nitrogen and nitro nitrogen by the ferrous-titanous procedure described in Ref 11, pp1384 & 1391

Hercules Method D90–3e (Ref 17, p11). *Nitroglycerin by Periodic Acid Oxidation.* NG and some other nitrate nitrogen esters which may be present in expl oils can be quantitatively denitrated to their respective alcohols by action of methanol and concd HCl. On oxidizing the residue of polyhydric alcohols with periodic acid, H_5IO_6, the secondary carbon and alcohol group in glycerin are oxidized to formic acid, which can be titrated with standard alkali, thereby giving a measure of the NG present. Ethyleneglycol, diethyleneglycol and diglycerin do not react with H_5IO_6 to form HCOOH. Some of the expl mixtures in which NG may be detd by the periodic acid oxidation are given in Table 2

Table 2

Combination of Explosive Oils in Which NG May Be Determined by the Periodic Acid Oxidation Method

Nitroglycerin Plus:

NGc (Nitroglycol) (EGDN)
DEGDN (Diethyleneglycol Dinitrate)
TNDG (Tetranitrodiglycerin)
SON (Sugar Octonitrate)
SON–EGDN

Note: SON (Nitrosugar) must be separated chromatographically before NG can be detd by the periodic acid oxidation procedure

A detailed description of periodic acid oxidation procedure is given in Hercules pamphlet D90 (Ref 17), Rev 5–10–61, pp12–13

Periodic Acid Oxidation Method

Dissolve an accurately weighed 2- to 4-g portion of the explosive oil in methanol and make up to 100 ml in a volumetric flask. By means of a pipet remove an aliquot of methanol soln to contain ca 0.25 g of NG and place in a tared (to 0.1 g) 250-ml Erlenmeyer flask for denitration. Add 10 ml of concd HCl, heat on a steam bath until all but ca 5 g of liquid is gone. The amt of liquid remaining in the flask can be detd by weighing the flask occasionally, beginning after 1½ hrs of heating on a platform balance. Cool the flask and wash down its sides with ca 15 ml of distd w. Add 3 drops of bromcresol green methyl red indicator (See Note 1) and make a rough neutralization of the residual HCl using 2N NaOH soln. Discard any soln at this point that requires either less than 7 ml or more than 13 ml of 2N NaOH. Make several new solns using different amts of explosive oil and treat them in the manner described above. At least three of them must meet the requirement of 7 to 13 ml of 2N NaOH for neutralization

Wash down the sides of each of the three flasks with distd w and make a final neutralization with 0.1N NaOH to green color of indicator. Dilute the contents of each flask to 50 ml, add 25 ml of 0.25M H_5IO_6 (using a pipet or buret), mix well and allow to stand for 10 mins or more in order to achieve oxidation. Add 2 drops of the above indicator (to compensate for the bleaching which takes place) and titrate with 0.1N NaOH to a green color

Calculation:

% NG in Expl Oil =

$$\frac{(A-B) \times (N\ NaOH) \times 0.2271 \times 1.087 \times 100}{\text{Wt of Expl Oil}}$$

where: A=ml NaOH required to titrate the sample
B=ml NaOH required to titrate 25ml of H_5IO_6 soln alone (See Note 2)
0.2271=ml wt NG/1000
1.087=Correction factor 100/92 based on the analysis of known samples of NG

Note 1: Bromcresol green-methyl red mixed indicator is prepd by adding 0.4g methyl red and 0.2 g bromcresol green to 100ml of 2B ethanol in a flask and warming, while shaking, to complete dissolution

Note 2: Periodic Acid 0.25M Solution is prepd by dissolving 55 g of G. Frederick Smith Chem Co H_5IO_6 in one liter of distd w, filtering thru a fritted glass funnel any insol material and storing a filtrate in a dark glass bottle provided with a glass stopper. For standardization of H_5IO_6, pipet exactly 25 ml of the above soln into a 250 ml Erlenmeyer flask, dilute with 50 ml of distd w and titrate with previously standardized 0.1N NaOH, using bromcresol green-methyl red indicator, to a green color. Standardization must be done each day that tests for NG are conducted

Note 3: Recoveries of less than 95–100% NG indicate the presence of NGc (Nitroglycol) or LF (Low-Freezing) compounds. If the latter were found to be present (by other tests), calculate:

$$\%\ NGc = A - (B + C)$$

where: A = % Total ether soluble
B = % NG by H_5IO_6 Method
C = % LF Compounds

If no LF compds are present, and the % of nitrogen is 18.20% or higher, a mixture of NGc & NG is present and

$$\%\ NGc = 100 - \%\ NG$$

Pierson's Procedure D-4. *Water Extraction* (Ref 11, p1357 & Ref 17, Method D90–4). Distilled water is used for extracting the residue from the ether soluble determination. Compare with BurMines Standard Method No 32 in which extraction is done with 1:1 ethanol-water soln. (See also Section III, steps 1 to 3, of this work)

Pierson's Procedure D-5. *Treatment of Water Extract–Determination of Salts* (Ref 11, pp 1357–59 & Ref 17, Methods D90–5a to D90–5f). Compare with Section III, steps 4 to 12 of this work

The following salts are determined: NH_4NO_3, $NaNO_3$, NaCl, $MgNO_3$, Chlorates & Perchlorates and Other Salts [such as ZnO, $Ca(NO_3)_2$, etc]. Sugar, occasionally present, may be estimated either by means of a polarimeter or by the Fehling soln method. Usually it is detd by difference

Magnesium Nitrate may be detd by a gravimetric method, weighing $Mg_2P_2O_7$ as the final product or by titrating an aliquot of the water extract with EDTA (Ethylenediamine-tetracetic Acid) at a pH of ca 10 using Eriochrome Black T indicator. This method is described in Ref 17, D90–5d, p15

Chlorates are determined in the presence of chlorides in the following manner: Acidify a 25-ml portion of the water extract with 2 ml of 1:3 nitric acid, add 5 ml of 0.1N $AgNO_3$ soln, heat to boiling and digest until AgCl coagulates. Filter off the ppt and collect the filtrate. Wash the ppt, dry for 2 hrs at 110°, cool in a desiccator and weigh. Calculate as NaCl and KCl and designate as "a" and "b" (See below) Dilute the clear filtrate to ca 150 ml, add 10 ml of 6% H_2SO_3 soln and boil the soln until the odor of SO_2 is no longer perceptible (usually 10 mins). Add 2 ml of 1:3 nitric acid, followed by 50 ml of 0.1N $AgNO_3$ soln, cover the beaker with a watch glass and heat on the steam bath for 30 mins. Then filter the ppt of AgCl, wash with distd w, dry for 2 hrs at 110°, cool in a desiccator and weigh. Save the filtrate for the perchlorate test. Calculate as $NaClO_3$ or $KClO_3$

$$\% NaClO_3 = (\% NaCl - a) \times 1.821$$
$$\% KClO_3 = (\% KCl - b) \times 1.644$$

$$\text{in which: } \% NaCl = \frac{\text{Wt AgCl} \times 0.4078 \times 100}{\text{Wt of sample}}$$

$$\% KCl = \frac{\text{Wt AgCl} \times 0.5202 \times 100}{\text{Wt of sample}}$$

a = % NaCl originally present as such in aliquot
b = % KCl originally present as such in aliquot

Perchlorates. Since perchlorates are not reduced by H_2SO_3, they will remain (if present) unchanged in the filtrate after removal of chlorides and chlorates. For a quantitative test, transfer that filtrate to a platinum crucible and evaporate to dryness on a steam bath. Add 5 g of anhydrous Na_2CO_3, mix and heat over a burner until a clear melt is obtd and then for 15–20 mins more. Cool the melt, dissolve in dilute HNO_3 and determine the chloride by pptn with $AgNO_3$ soln

$$\% NaClO_4 = \frac{\text{Wt AgCl} \times 0.8543 \times 100}{\text{Wt of sample}}$$

$$\% KClO_4 = \frac{\text{Wt AgCl} \times 0.9667 \times 100}{\text{Wt of sample}}$$

Note: If test is conducted starting with a 25-ml aliquot of original water extract, the wt of AgCl determines the sum of chloride, chlorate, and perchlorate. On subtracting chloride and chlorate from the sum, perchlorate is obtd

Pierson's Procedure D–6. *Acid Extraction* (Ref 11, pp1359–60 & Ref 17, Methods D90–6, D90–6a & D90–6b, pp19–20). Procedures are similar to those described in BurMines Standard Method No 33 and in Section IV of this work

Pierson's Procedure 7. *Treatment of Acid Extract* (Ref 11, pp1360–61 & Ref 17, Methods D90–7a to D90–7d). Procedures are essentially the same as described in Section IV of this work, Items 1 to 7

Pierson's Procedure D–8. *Carbon Tetrachloride Extraction for Sulfur* (Ref 11, p1361 & Ref 17, Method D90–8). Same as in BurMines Standard Method No 34, described in Section V, Step 1 of this work

Pierson's Procedure D–9. *Acetone Extraction for NC* (Ref 11, pp1361–62 & Ref 17, Method D90–9). Same as in BurMines Standard Method No 34, described in Section V, Step 2

Pierson's Procedure D–10. *Ash Determination* (Ref 11, p1362 & Ref 17, Method D90–11). In this procedure ash is determined by carbonizing the residue after acetone extraction, prior to visual (and microscopic) examination of absorbents, as is done in the BurMines Standard Method No 35, described in this work as Procedure VI, Step 1. There is no advantage in burning the residue before it is examined for absorbents because a new residue, contg unburnt absorbents, will be required

It seems that the procedures described in BurMines Standard Method No 35 of burning the residue to ash after examining its absorbents must be preferred (See Procedure VI, Steps 2 & 3 of this work)

Pierson's Procedure D–12. *Extended Examination of Explosive Oils* (Ref 11, pp1362–64 & Ref 18, Methods D90, p1). Section II, Step 2 of this work describes the BurMines procedure of Standard Method No 30 for separating explosive oils. Section II, Step 3 describes the separation and examination of Nitrocompounds, but not the separation and identification of some other compds, such as SON(Sucrose Octanitrate), LF (Low-Freezing) Compounds, TeNDG (Tetranitrodiglycerin), NGc (Nitroglycol), DEGDN (Diethyleneglycol Dinitrate). Their separation and identification are described, however, in Refs 11 & 18

For a complete analysis of an explosive oil, it is recommended to prepare at least 10 g of the filtered dry expl oil before starting any tests. For preparing such a quantity, place a piece of rapid filter paper in a 3-inch

Büchner funnel and fill the funnel from ¼ to ¾ full of the Dynamite sample. The amt will depend on the expl oil content, which is detd from the ether extraction conducted as follows: Pour in 50ml of ether, cover the funnel with a watch glass, let stand to impregnate the sample and then apply suction. Repeat with 3 addnl 50-ml portions of ether and transfer the extract to a low beaker. Evaporate the ether at 50–60°C while passing over it a stream of dry air, taking care not to introduce any moisture. Continue evaporation until disappearance of ether odor and then for 15 mins longer. Filter off the resins, sulfur and other insol substances from the explosive oils, using a very small, fast filter paper (such as No 41 Whatman) into a rubber-stoppered bottle contg the expl oil previously obtd from detn of ether extract. Store it in a $CaCl_2$ desiccator

Separation and Identification of Low Freezing Compounds and Nitrocompounds of Explosive Oils are briefly described here under Pierson's Procedure D–3 (See above). The tests are colorimetric. Some of these compds may be identified by IR and X-ray spectroscopy (Ref 11, p1363)

Nitrated Polymers: Tetranitrodiglycerin (TeNDG) and Diethyleneglycol Dinitrate (DEGDN) (Ref 11, p1363 & Ref 18, pp2–3). These compds are relatively insol in 60% AcOH, whereas NG & NGc are completely sol. For their identification, weigh 5.0g of the filtered expl oil into a large test tube, add 50ml of 60% AcOH, stopper and shake well. Insol oily drops collecting on the bottom of the test tube indicate the presence of nitrated polymers. In this case, decant the liquid and repeat the extraction of oil with two addnl 50-ml portions of AcOH. Wash the residue with two 50-ml portions of distd water and separate by decantation. Dissolve the residue in 30ml of ether and add an excess of anhyd Na_2SO_4 to take up the remaining w. Either decant or filter the ethereal soln, evaporate to dryness and place in a vacuum desiccator for one hr. A partial identification of components can be made on the basis of a nitrogen detn: TeNDG contains 16.19%N while DEGDN has 14.29%N. Their mol wts are 346.17 & 196.12, respectively, while mol wt of the residue can be detd by an ebullioscopic method

Nitroglycerin Determination by Periodic Acid Oxidation is described as Hercules' Method D90–3e (Ref 17, p11). This method is also described here at the end of Pierson's Procedure D–3

Sucrose Octanitrate (SON) or Nitrosugar Identification (Ref 11, p1362 & Ref 18, pp2 & 11). Dissolve a small portion of the expl oil in methanol and add a little NH_2S (or Zn dust + dil HCl). After the reaction is complete and solids have settled, decant the supernatant liquid and test for sugar with Fehling's reagent, as indicated in StdMethodsChemAnalysis, Vol **2B** (1963), pp1362–63. A bright red ppt of Cu_2O indicates reducing sugar. The optical rotation of SON by polarimeter may also be used for a quantitative detn. The optical (specific) rotation of sugar (sucrose) is +66.5° and it drops after nitration to 56.05°. No other common ingredients of Dynamites are optically active

SON can be separated from NG & the other expl oils by a chromatographic technique, and either weighed, as such, or measured by polarimetry. Take an aliquot portion of ether extract of Dynamite, evaporate to dryness and dissolve the residue in 2:1-benzine-ligroin. Treat chromatographically, using a silicic acid column. The NG, NGc and similar expl oils are washed thru the column, while SON is retained. It can be removed by elution with benzene-ligroin and acetone (Ref 11, p1363) (Chromatography is described in Vol 3 of Encycl, pp C289 to C298) (See also Ref 18, pp4–6)

Mixture of NGc and NG, separated from other interfering ingredients by suitable solvents or by a chromatographic method, may be quantitatively analyzed by IR (infrared) spectrophotometry, as briefly described by Pierson (Ref 11, pp1363–64)

Note: The outline presented by Pierson is based on the method of W. Huff, M. Halik & F. Pristera described in PATR **2472** (Dec 1957) of the Feltman Research Laboratories, Picatinny Arsenal, Dover, NJ and also on the paper of F. Pristera (of PicArsn) in Applied Spectroscopy **7**, 115 (1953) (See also Ref 18, pp7–9)

Analysis of Wrapper for paraffin, NG, paper, ash, and moisture is described in detail by Pierson (Ref 11, p1364)

EXAMINATION OF SOME COMMERCIAL AND MILITARY DYNAMITES AS DESCRIBED IN US MILITARY SPECIFICATIONS, AMENDMENTS AND ENGINEERING CHANGE ORDERS

Dynamites (Commercial) (Used for Military Purposes). Their compositions and some of the properties and tests are listed in Specification MIL-D-60365 (MU), 25 Feb 1966 and in AMENDMENT 1, 15 Nov 1968

There are five types of Dynamites listed in the above Spec and its Amendment

Table 3

Ingredient, %	Percent,Type				
	I	II	IIa	III	IV
Explosive Oil	16±4*	25±5	29±4	50±4	64±4
NH_4NO_3	62±4	13±4	28±4	–	–
$NaNO_3$	10±4	47±4	31±4	39±5	28±5
Fuel & Antacid	Carbonaceous combustible and antacid are left to the discretion of the manufacturer				
Rate of Detonation ft/sec, minimum	10000	12000	16000	16000	18000

* May be NS (Nitrostarch)

Requirements and Tests:

1) *Moisture Content* shall not exceed 2% when detd in accordance with a suitable method that appears in a standard or testbook reference and previously approved by the contracting officer

2) *Stability.* The Dynamites shall cause no discoloration of KI-starch test paper for a minimum period of 10 mins, when tested at 82.2±1.0°C as specified in 4.3.3, p4 of MIL-D-60365 (MU) (Code No 03001). A brief description of this test, known as Abel's Test, is given in Vol 1 of Encycl, p A2-L

3) *Rate of Detonation* (Code No 04001) shall be not lower than indicated in Table, when detd by the Dautriche Method (which is described in Vol 3, p C311-R) or by other methods, such as Mettegang's, described under Chronographic Methods, p C311 to C318 (See also in Vol 4, Sect 11, pp624ff)

4) *Workmanship* shall be of the highest grade throughout and in accordance with the best standard practice

5) *Uses:*

Type I – in general blasting of rocks & stumps, and ditching in dry areas by individual initiation of sticks

Type II – in blasting of very hard rocks and general blasting of rocks, stumps and earth in wet areas

Type IIa – not specified, but probably for the same purposes as Type II

Type III – in submarine blasting, cutting of trees, shearing of light steel and ditching in wet areas by self-propagation

Type IV – in heavy duty submarine blasting, cutting of large trees and shearing heavy steel

Analytical Procedures are not given for the above Dynamites in Spec MIL-D-60365 (MU) or in AMENDMENT 1

Dynamite, Military, Low Velocity (LVD), developed at Picatinny Arsenal by B. Voigt, is described here under DYNAMITES, Class X and in PATR **1740**, which was superseded by AMCP **706-177** (1967), pp122–24. It was never standardized nor manufactured. Therefore, no Specification was issued

Dynamite (Military), M1, known as **MVD** (Medium Velocity Dynamite). Its requirements, physical tests, and analytical procedures are covered by Specification MIL-D-45413A (MU) 15 Aug 1962 and Amendment 4, 15 Nov 1968. It supersedes MIL-D-45413 (ORD), 15 Jan 1960 and several Engineering Change Orders originating at Picatinny Arsenal

I. Requirements and Physical Tests

1) *Materials* used in manuf of Dynamite shall consist of Desensitized RDX (complying with Spec MIL-R-13742), grained TNT (complying with Spec JAN-T-248 for Grade I crystalline material), Polyisobutylene/Oil Gel (complying with Spec MIL-P-13822) and Guar Gum (complying with Spec MIL-G-51133). Materials are uniformly mixed in proportion to form a Dynamite contg RDX 75±1.0, TNT 15±1.0, Process Oil plus Polyisobutylene 5±0.5 & Guar gum 5±0.5%. This mixture is packed in cylindrical paper shells forming cartridges 1¼ inches in diam and 8 inches in length. The paper is manila weighing ca 70 lbs per ream (500 – 24x36 inch sheets) and shall be sprayed or dipped in molten paraffin before being filled with Dynamite

2) *Consistency* shall be such that it can be

molded by hand to a cohesive mass after being held in a covered container for 24 hours in a refrigerator maintained at –70±5°F

3) *Sensitivity to Blasting Cap.* Initiation by a blasting cap (complying with Spec MIL-C-14003) inserted at one end of a 4-ft long column, shall cause complete detonation thruout the entire length of the column. The column is formed by placing cartridges end-to-end in contact on smooth dry ground. The rates of detonation shall be determined in the first and last cartridges of the column and they shall not differ by more than 10% and not be below 17716 feet/sec (5400m/sec). The rates are detd either by the Counter Chronograph Method or by the Dautriche Method (Counter Chronograph Method is described in Vol 3 of Encycl, pp C309-R & C310-L, while Dautriche Method is described on pp C311 & C312)

4) *Sensitivity to Detonating Cord.* Initiation by detonating cord (complying with Spec MIL-C-17124A, Type I, Class C) of column of cartridges 4-ft long shall cause complete detonation thruout the entire length of the column. For this test, the cord shall be attached to the first cartridge, one inch from the end, by a girth hitch and one extra wrap thru the girth hitch. Observation shall be made for deton of the cartridges thruout the entire length. The rate of deton shall be determined for the first and last cartridges by the same methods as indicated under Sensitivity to Blasting Cap

7) *Sensitivity to Bullet Impact.* No detonation shall take place when a block of Dynamite 3x3 inches and 1 inch thick, packed at normal density, is struck by a bullet from an M2.30 caliber ball cartridge at a range of ca 100ft. The block is placed vertically with one of the 3x3 faces covered by a 3/8-inch thick mild steel plate, while the other, open end, is struck by the bullet

6) *Water Immersion Test.* The MVD Dynamite shall function high order after immersion in water. For this test, several cartridges, randomly selected from the lot, are immersed in 12 feet of water for a period of 24 hours. Then the sticks are removed, dried and subjected to one of the detonation rate determinations listed under Sensitivity to Blasting Cap. The rate shall not be lower than obtd for cartridges not submitted to immersion in water. If any stick (sample) fails to comply with requirements, the lot shall be rejected

II. Analytical Procedures

1) Extraction with Carbon Tetrachloride

There are two methods for this extraction. The earlier method, which does not use an extraction apparatus is described on p5 of Spec MIL-D-45413A issued in 1962, while the alternate method using a Bailey-Walker Extraction Apparatus is described on p4 of Amendment 4 issued in 1968. Here is the description of the alternate method:

Weigh accurately ca 2.0g of sample in a tared filtering crucible and install it in a Bailey-Walker Extraction Apparatus (such as No 9-630 of Fisher's catalog) (See Fig 1), contg carbon tetrachloride. Heat on a water-bath (bp of CCl_4 is 76.8°C) for ca 45 mins or until most of yellow TNT color is gone from the crucible. After complete extraction, cool the app to RT and transfer the extract to a 250-ml volumetric flask, followed by three 25-ml CCl_4 washings. Transfer the crucible to a Fisher Filtrator (such as shown in Fisher catalog as No 9-788) (See Fig 2), and wash with ca 100ml CCl_4 using suction. Transfer the washings to the 250-ml volumetric flask contg the filtrate with three 25-ml washings, fill with addnl CCl_4 to the mark and save for the TNT determination. Remove the crucible from the filtrator, dry for one hour in an oven maintained at 70±5°C, cool in a desiccator and weigh. Save the crucible with

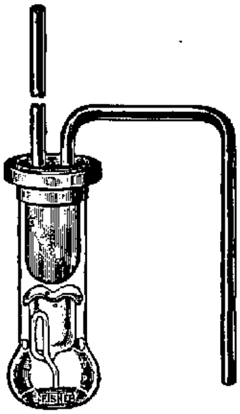


Fig 1. Bailey-Walker Extraction Apparatus

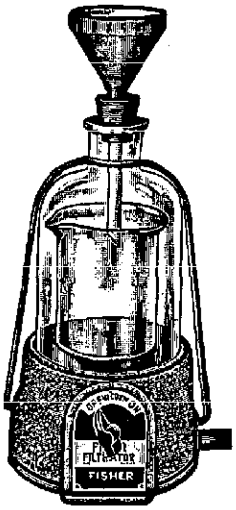


Fig 2. Fisher Filtrator

residue for RDX and Guar Gum determinations

2) **TNT Determination by the Titanous Chloride Method.** It is based on the following reaction:

$H_3C.C_6H(NO_2)_3 + 18TiCl_3 + 18HCl \rightarrow$
$H_3C.C_6H(NH_2)_4 + 18TiCl_3 + 6H_2O$ and standardization of $TiCl_3$ soln is based on the reaction:

$6TiCl_3 + K_2Cr_2O_7 + 14HCl \rightarrow$
$6TiCl_4 + 2KCl + 2CrCl_3 + 7H_2O$

a) *Preparation of 0.2N Standard Titanous Chloride Solution from 20-Percent Solution.* The method is the same as used for prepn of 0.15N soln described in Vol 1, pp A415-R & A416-L of Encycl, except that a larger proportion of commercial 20% $TiCl_2$ soln is used

Procedure. For each liter of soln mix 150 ml of 20% $TiCl_3$ soln with 100 ml 38% HCl and dilute with distd, oxygen-free, water to 1000 ml. Mix the soln thoroughly by passing a current of oxygen-free inert gas thru it. If necessary, filter the soln thru No 41 Whatman filter paper until the filtrate is completely clear. Agitate the soln thoroughly with the inert gas for a few mins, and transfer the soln into an amber storage bottle, provided with a two-hole stopper. Maintain the inert atmosphere during storage, using the apparatus shown in Fig 3 (Ref 19)

b) *Preparation of Standard 0.2N $TiCl_3$ Solution from Titanium Hydride* (Method Developed by Mr N. Liszt of Picatinny Arsenal)

Procedure. For each liter of soln warm a flask with 100 ml of 38% HCl to 70–80° on a hot plate in a well-ventilated hood, remove the flask and add in small portions (to avoid excessive frothing) 12 g of titanium hydride. Add ca 250 ml of distd, oxygen-free, w and mix thoroughly for 5–10 mins by passing a current of oxygen-free inert gas thru the soln. Filter the soln thru No 41 Whatman filter paper until the filtrate is completely clear. Agitate the soln with the inert gas, and store in an amber bottle. Maintain the inert atm during storage, using the apparatus shown in Fig 3 (Ref 19, p2)

c) *Standardization of $TiCl_3$ Solution with $K_2Cr_2O_7$ Procedure.* Weigh accurately ca 0.35 g of pure dried $K_2Cr_2O_7$ (NatlBurStandards Sample No 136 dried for 2 hrs at 100°) into a special titration flask of 500-ml capacity shown as F in Figs 3 & 3a. Add 100 ml distd water to dissolve the dichromate and sweep out the air by passing in a current of CO_2(or N_2) during 5 mins. Add 50 ml of 10% H_2SO_4 soln and titrate with 0.2N $TiCl_3$ soln (to be standardized) from buret C of Fig 3, connected by tube to Kipp Apparatus K. The current of CO_2 (or other inert gas) must pass thru flask F during titration. Near the end point, which can be judged by change in color from brownish-purple, and to a distinct green, add 5 drops of 0.32% Ba diphenylsulfonate indicator soln, as specified in Method 708.1 of MIL-STD-286B (1967). Continue the titration until the end point is reached as shown by the appearance of a light green color, which does not change to a light blue color for at least 30 seconds. Then the blue color will remain

Make a blank determination to correct for the presence of iron as follows: Add 5 ml of 20% ammonium thiocyanate indicator soln to the flask after completion of above titration. If iron is present, a ferric thiocyanate complex will form and the soln will change from light blue to deep red. Back-titrate the ferric thiocyanate complex with 0.2N $TiCl_3$ soln from the same buret until the light blue color reappears. Determine the mls of $TiCl_3$ soln required to complete both the titration and the back-titration. Add the two values and apply buret and temperature corrections as given in the Table on p A417 of Encycl, Vol 1

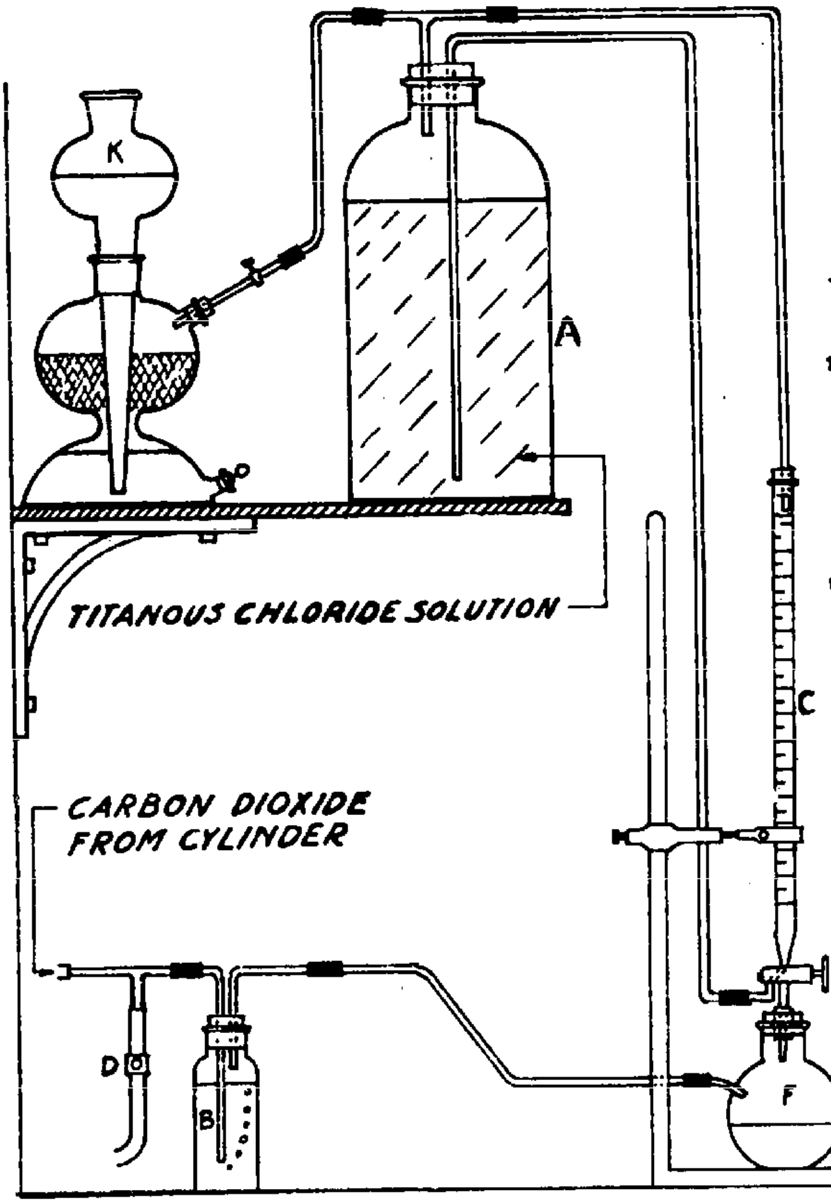
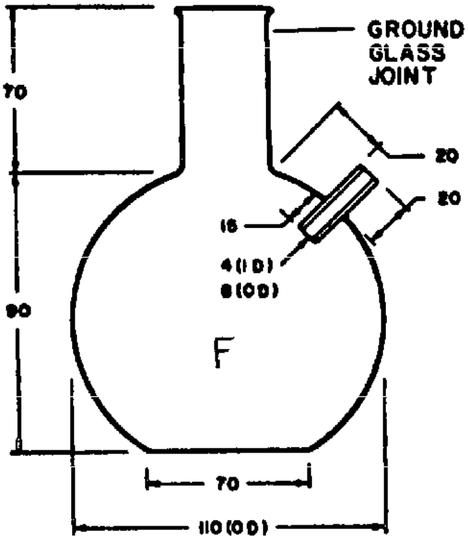


FIG 3
APPARATUS FOR STORING AND USING TITANOUS CHLORIDE SOLUTION



PYREX GLASS FLASK
BULB CAPACITY — 500 ml
ALL DIMENSIONS IN MILLIMETERS

Fig 3a. FLASK F

Note: The app shown in Fig 3 is similar but not the same as shown on the left half of apparatus in Fig on p A416-L of Vol 1

Calculate the normality (N) of $TiCl_3$ soln using the following equation:

N of $TiCl_3$ soln = A/(0.04904B),

where: A = wt of $K_2Cr_2O_7$ and

B = corrected reading of buret (Ref 19, p3)

The normality of $TiCl_3$ soln shall be checked daily by means of cross-titration with ferric ammonium sulfate soln, as indicated in item (d)

d) *Preparation and Standardization of 0.15N Ferric Ammonium Sulfate Solution.* Interaction between titanous chloride and ferric ammonium sulfate proceeds accdg to the following equation:

$$2TiCl_3 + Fe_2(SO_4)_3.(NH_4)_2SO_4.24H_2O \rightarrow TiCl_4 + Ti(SO_4)_2 + FeCl_2 + FeSO_4 + (NH_4)_2SO_4 + 24H_2O$$

Prepare 0.15N ferric ammonium sulfate soln in the same manner as described in Vol 1 of Encycl, p A415-R for prepn of 0.05N soln, but make it three times stronger by using ca 75 g of $Fe_2(SO_4)_3.(NH_4)_2SO_4.24H_2O$ for each liter of reagent

For its standardization with $TiCl_3$ soln, measure accurately 45 ml of the above prepd ferric ammonium sulfate into titration flask F of Fig 3, add 25 ml of 15% hydrochloric acid and, while passing CO_2 thru the flask, titrate from buret C with 0.2N $TiCl_3$ soln (already standardized by potassium dichromate) until near the end point. Then add 5 ml of 20% ammonium thiocyanate indicator and continue titrating until the red color just disappears. Apply the buret and temperature corrections and then calculate:

Normality of ferric ammonium sulfate = AN/B.

where A = vol of $TiCl_3$ soln used; N = its normality; B = vol of ferric ammonium sulfate soln used (45 ml)

It is assumed that the strength of the ferric ammonium sulfate reagent will remain constant for an indefinite period

e) *Procedure for Determination of TNT*

Take the 250-ml volumetric flask contg material extracted by carbon tetrachloride as shown here under II. Analytical Procedures, step 1, and transfer from it 25 ml (using a calibrated pipet, previously rinsed with CCl_4 and dried, to the titration flask F. Evaporate the CCl_4 to dryness with a slow stream of air and then sweep the air off with a stream of CO_2 from a cylinder (or from a Kipp generator). While continuing to pass CO_2 thru the flask, introduce 25 ml of glacial acetic acid to redissolve the residue. Then add to the flask (which is always connected to a CO_2 cylinder) an accurately measured excess of 0.2N $TiCl_3$ soln (25 ml) and 25 ml of 15% hydrochloric acid together with some glass beads or broken pieces of porcelain. Install a reflux condenser and boil the soln for 5 mins on a hot plate. Increase the current of CO_2 and, while the flask is still connected to the reflux condenser, cool to RT by cautious immersion in a large beaker contg cold water. While continuing to pass CO_2, disconnect the condenser and add to the flask 5 ml of ammonium thiocyanate. Titrate from buret C contg 0.15N ferric ammonium sulfate to the first permanent red color

Run a blank by sweeping air with a stream of CO_2 from an empty flask F, and, while continuing to pass CO_2, add 25 ml of glac AcOH, 25 ml of 15% HCl soln and 20 ml of 0.2N $TiCl_3$ soln. This is followed by boiling for 5 mins. While the flask is connected to a reflux condenser, cool and titrate with 0.15N ferric ammonium sulfate in the presence of 5 ml of 20% NH_4SCN soln

Calculation:

$$\%\ TNT = \frac{1.261[(AN - BF) - (CN - DF)]}{W},$$

where: A = ml of $TiCl_3$ soln added to flask; N = normality of $TiCl_3$ soln; B = ml of ferric ammonium sulfate soln added from buret C; F = its normality; C = ml $TiCl_3$ soln added to blank; D = ml of ferric ammonium sulfate soln used for blank; W = wt of sample represented by the aliquot taken (ca 2.0 g)

3) RDX Determination

Insert the filtering crucible (contg the residue after extraction with CCl_4 of 2 g sample in opn II, 1) in the adapter of a suction flask and add 20 ml of acetone before applying suction. After the acetone starts to drip thru the filter, apply suction to remove the rest of acetone. Then stop the aspiration and repeat the procedure until a total of five 20-ml extractions have been made, or until the following qualitative test for RDX is negative: transfer a small quantity of the last 20-ml portion of washing to a 25-ml beaker, evaporate to dryness on a steam bath and add several drops of chromotropic acid in 36N sulfuric acid. The formation of a reddish-brown color indicates the presence of RDX. Some addnl washings shall be made in this case until the test produces no color. Then aspirate until the odor of acetone in the crucible is no longer detectable, dry the crucible in an oven at 70±5°C for one hour, cool in a desiccator and weigh. Calculate as follows:

$$\% \text{ RDX} = \frac{(A - B) \times 100}{W}$$

where A = wt of crucible with contents before acetone extraction; B = wt of crucible with residue after acetone extraction; W = wt of sample (ca 2 g) before carbon tetrachloride extraction

4) **Guar Gum Determination**

Percentage of Guar Gum, used since 1968 to replace cornstarch, is calcd from the equation:

$$\% \text{ Guar Gum} = \frac{(B - C) \times 100}{W}$$

where B = wt of crucible with residue after acetone extraction; C = wt of empty crucible; W = wt of sample (ca 2 g) before carbon tetrachloride extraction

5) **Polyisobutylene and Process Oil Determination:** The sum of their percentages is calculated by subtracting from 100% the total of the percentages of TNT, RDX and Guar Gum

References on Examination of Dynamites and of Other Blasting Explosives (practiced in USA): 1) W.O. Snelling & C.G. Storm, "The Analysis of Black Powder and Dynamite", USBurMines Bull **51**(1916) 2) C.G. Storm, "The Analysis of Permissible Explosives", USBurMines Bull **96**(1916) 3) S.P. Howell & J.E. Tiffany, "Methods for Routine Work in the Explosives Physical Laboratory of the Bureau of Mines", Technical Paper **186**(1918) 4) C.A. Taylor & Wm.H. Rinkenbach, "EXPLOSIVES: Their Materials, Constitution and Analysis", Bull **219**(1923), pp116–118 (Physical examination of Blasting Explosives); 118–19 (Dope-size test); 119–43 (Chemical Analysis of Blasting Explosives) 5) C.E. Munroe & J.E. Tiffany, "Physical Testing of Explosives at the Bureau of Mines Experimental Station, Bruceton, Pennsylvania", USBurMines Bull **346**(1931) 6) Dr C.G. Storm, pp1662–79 in the 5th edition of Scott's "Standard Methods of Chemical Analysis", edited by Dr N.H. Furman, and published in 1939 by VanNostrand, NY 7) Clift & Fedoroff, Vol **1**(1942), Chapter XI, pp18–24 & Vol **3**(1943) 8) Anon, "Analytical Methods for Powders and Explosives", AB Bofors, Nobelkrut, Bofors, Sweden (1960), pp15–17 (Water content by Karl Fischer Method) 9) Fedoroff & Sheffield, "Encyclopedia of Explosives and Related Items", PATR **2700**, Vol **1**(1960) 9a) CondChemDict (1961), pp XIII & XV 10) PATR **2700**, Vol **2**(1962) 10a) US Military Standard MIL-STD-650 (1962), Method 101.4 11) F.J Welcher, Editor, "Standard Methods of Chemical Analysis", Vol **2**, VanNostrand, Princeton, NJ (1963), Part **A**, pp 436-38 (Microdetermination of Water by Titration With Karl Fischer Reagent); Part **B**, Chapter **32**, "Analytical Procedures for Explosives and Propellants" by R.H. Pierson 12) Dr R.W. Van Dolah, Research Director, Explosives Research Center, USBurMines, Pittsburgh, Pennsylvania; private communications July 31, 1964 and August 17, 1964 13) PATR **2700**, Vol **3**(1966) 13a) S.A.H. Amas & H.J. Yallop, Analyst **91**(1082), 366–67 (1966) & CA **65**, 3659 (1966) (For detection of dinitro- and trinitro- aromatic bodies in industrial blasting expls, a 5–10 mg sample of expl to test is placed on a spot plate and a drop of Me_2CO-EtOH and a drop of 25% aq Me_4NOH soln are added. The formation of a blue color indicates the presence of 2,4-DNT; while 2,4,6-TNT gives a dark red color. A slight yellow color is formed initially with NG. If NG & 2,4-DNT are both present, a green color is initially obtd. None of the other common components of blasting expls interfered. It is not indicated in CA which color is produced by m-MNT) 13b) H. Poesehl & K. Haisler, Rudy (Prague) **14**(4), 162–65 (1966) (Czech) & CA **65**, 6987 (1966) (Some problems in the testing of blasting means and technique) 13c) D. Harzt, FreibergerForschungsh **A371**, 115–83 (1966) (Ger) & CA **65**, 6987 (1966) (Measurement of deton vel under actual operating conditions and the utilization of technical expln parameters to characterize the explosives) 13d) M. Niimi, Kôgyô-KaykuKyôkaishi **27**(2), 71–2 (1966) (Japan) & CA **65**, 6987 (1966) (A review of detn sensitivity of expls with 6 refs) 14) Anon, "Military Explosives", **TM9-1300-214/TO 11A-1-34**

(1967), pp5-1 to 5-24 15) Dr R.W. Van Dolah; private communication, April 8, 1971 with enclosure of analytical procedures (Methods Nos 21, 28, 30, 32, 33, 34 & 35) currently used at the BurMines Laboratory, Bruceton, Pa for Blasting Explosives (including Dynamites) 16) US Military Specifications and other papers describing requirements and tests of Dynamites used for military purposes. These papers were collected by Mr Nathan Liszt of Picatinny Arsenal. They include the following: a) Purchase Description PA-PD-525, 9 June 1954, "Dynamite, Military M1, M2 and M3, superseded by Spec MIL-D-45413A(MU), 15 August 1962 with four Amendments of which No 4 is of 15 Nov, 1968 b) Purchase Description PA-PD-659, 16 Sept 1955, "Dynamite, Commercial", superseded by Spec MIL-D-60365(MU) 25 Feb 1966 and Amendment 1, 15 Nov 1968 17) Hercules Powder Co, Wilmington, Delaware, Research Center: "Dynamite – Complete Analysis", Methods D90–0 to D90–11. The latest Rev 5-10-61 18) Ibid, "Dynamite–Explosive Oil Analysis", Appendix, Methods D90(1-30-57) 19) MIL-STD-286B (1 Dec 1967), Method 601.1.1, "Titanous Chloride (0.2N Standard Solution)" 20) Frank Pristera, "Explosives" in Vol 12 of Encyclopedia of Industrial Chemical Analysis, Wiley, NY (1971), pp443–45 (Nitroglycerin and Dynamites, Analysis); 445 (PETN); 445 & 451 (Ethyleneglycol Nitrates); 451 (Other organic nitrate esters); 451 (Ammonium Nitrate); 452–60 (Identification of explosives by Infrared spectroscopy); 460–62 (Analysis of unknown HE's); 461–67 (Nitrogen content determinations); 467–70 (Other methods for quality control) ; 470–71 (67 references on analytical procedures)

Foreign Methods of Examination of Dynamites are essentially the same as the American methods. They are briefly described in Refs 1 to 7

Following are some foreign terms:

French. Épreuves de dynamites: Acidité, Humidité, Stabilité, Exsudation, Consistence (Consistency); Analyse chimique (Ref 2, pp 557–59)

German. Untersuchung: Azidität Feuchtigkeit (Moisture), Stabilität (Chemische Beständigkeit), Ausschwitzung (Exudation); Chemische Analyse [Asche, Kieselguhr, Nitroglyzerin, Vaselin, Fett (Greese), Kamfer, Anorganische Salze, Schwefel (Sulfur), Zellulose, Nitrozellulose, Kollodiumwolle (Collodion Cotton), Harz (Rosin), Nitropolyglyzerine and Dinitrochlorhydrin) (Ref 3, pp449–63)

Italian. Prove delle dinamiti: Acidità, Umidità (Moisture), Stabilità, Essudazione; Dosatura (Determinazione della nitroglicerina e della materia inerte; nitrocellulosa solubilile, resine, canfora, e nitroderivati aromatici (Ref 1, pp310–12 & Ref 7, p463)

Russian. Ispytaniye dinamitov: Kislotnost' (Acidity), Vlazhnost' (Moisture), Stoykost' (Stability), Eksudatsiya; Khimicheskii analiz (Nitroglitserin, Nitroglikol, Piroksilin, Inertnyiye veshchestva, etc) (Ref 6, pp37, 38, 94, 95)

Spanish. Pruebas de las dinamitas: Asidez, Humedad, Higroscopicidad, Estabilidad, Exudación; Analisis quimico [Nitroglicerina, Nitrocelulosa, Nitroclohidrina, kieselguhr, carbón, serrin (sawdust), caolin, etc] (Ref 4, pp758–63 & Ref 5, pp271–76)

Refs: 1) R. Molina, "Esplodenti", Hoepli, Milano (1930) 2) L. Vennin, E. Burlot & H. Lécorché, "Les Poudres et Explosifs", Ch. Béranger, Paris (1932) 3) H. Kast & L. Metz, "Chemische Untersuchung der Spreng und Zündstoffe", Vieweg, Braunschweig (1944) 4) A. Pérez Ara, "Tratado de Explosivos", Editorial, Cultural SA, La Habana (Cuba) (1945) 5) M. Vivas, R. Feigenspan & F. Ladreda, "Pólvoras y Explosivos Modernos", J. Morata, Madrid, Tomo IV (1946) 6) A.G. Gorst, "Porokha i Vzryvchatyiye Veshchestva" (Powders and Explosive Substances), GosIzdatOboronProm, Moscow (1957) 7) M. Giua, "Trattato di Chimica Industriale", UTET, Torino, VI(1)(1959)

Note: We express our appreciation to Mr N. Liszt, Analytical Chemical Branch Chief, Propellants Division, FRL, Picatinny Arsenal for reviewing the Analytical Section; and to Dr R.W. Van Dolah, Research Director, Explosives Research Center, US Bureau of Mines, Pittsburgh, Pa for supplying current BurMines analytical procedures

Additional References on DYNAMITES AND THEIR SUBSTITUTES:

Note: Many references dated prior to 1960 are already given in Vol 1 of Encycl, under "Ammonium Nitrate Blasting Explosives", pp A350 to A353; in Vol 2, under "Blasting Explosives", pp B203 to B211, and in Vol 3, under "Commercial or Industrial Explosives", pp C456 to C460. Below are listed important references from 1907 to 1960 which were not included in Vols 1, 2 & 3 and also references from 1960 to 1970, incl

1) DuPont Co Pamphlet and SS **2**, 170(1907) (For thawing small quantities of Dynamites, they may be packed in tight tin cans and placed in fresh manure. For larger quantities a double boiler is recommended. If Dynamites have to be thawed frequently, it is recommended that a small, steam-heated building be used, with source of steam located at a distance)

2) G.F. Samuel, EngrgNews **59**, 692(1908) & CA **2**, 245(1908) (Magazine and thaw house for Dynamites)
2a) Editor, Engrg & MiningJ **88**, 422(1909) & CA **3**, 2872(1909) (Description of an electrical thawing box for Dynamites)

3) Anon, SS **5**, 97(1910) & CA **4**, 1456(1910) (Description of electrical thawing apparatus used in the Republic mine in Michigan)

4) A. LaMotte, ArmyOrdn 198–200(1926) (Review of the development of NG explosives)

5) US Army, Chief of Ordnance, "Safety and Storage Manual for Explosives and Ammunition", O.O. Form No 5994, Section XXII, p2, Washington, DC(1928), Destruction of Dynamites (In bldgs where Dynamite is stored, special attention will be given to oily stains or exudations of NG. The stains of this nature appearing on the floor of the magazine must be neutralized by scrubbing the floor thoroughly with a soln of ½ gallon of water, ½ gallon of wood alcohol and 2 lbs of Na or K sulfite)

6) F.H. Bergeim, USP 1751437(1930) & CA **24**, 2605(1930) (A Gelatin Dynamite consisting of NG, NC and nitrated surbitol or other nitrate of a polyhydroxy alcohol contg 4 to 6 carbon atoms)

7) H.H. Fassnacht, USP 1966090(1934) & CA **28**, 5672(1934) (NG and Tetranitrodiglycerin are gelatinized with NC in the presence of 0.5 to 2.0% "gelatinization accelerator", such as ethyleneglycol monoethyl ether)

8) IG Farbenindustrie AG, FrP 848652 & 848740(1939); CA 6114 &5912(1941) (Low-freezing Dynamites contg glyceryl monoacyl derivatives)

9) F. Schwarzer, SwissP 228654(1943) & CA **43**, 2437(1949) (Dynamite prepd by mixing 40 parts of NG with 50 parts of pulverized Al & kieselguhr, by volume)

10) R.W. Lawrence, USP 2338120(1944) & CA **38**, 3478(1944) (Dynamite consisting of AN mixed with NG and at least 15% of a nitrated aliphatic paraffin, such as Nitromethane, Dinitroethane, etc. These Dynamites were less sensitive to shock than those consisting of only NG and AN)

11) M. Bonotto, USP 2413946(1947) & CA **41**, 2246(1947) (Non-extruding Dynamites produced by blending the usual constituents with dry vegetable protein meal, such as soybean meal)

12) S. Fordham et al, BritP 586224(1947) & CA **41**, 7120(1947) and USP 2454643(1948) & CA **43**, 3619(1949) (Plasticity of Gelatin Dynamite is improved by incorporating a compd of polethyleneglycol ether contg at least 8 carbon atoms joined together and at least 2 ethyleneoxy groups joined together. Example: polyethyleneglycol monoethyl ether. Such Dynamites were easier to extrude.

13) H.R. Wright & Wm.G. Allan, USP 2439328 (1948) & CA **42**, 4350(1948) (Plastic Dynamite contg PETN or HeNMnt and RDX distributed in a mixture of Dinitrocumene, Dinitro-tert-butylbenzene and nitrated product of sec-butylbenzene)

14) J. Whetstone, USP 2460375(1949)& CA **43**, 2776(1949) (A granular Dynamite is prepd by emulsifying a molten HE, such as TNT, by the aid of agar-agar, and mixing the emulsion with a melt comprising AN with at least one compd which is nonvolatile at the temp of the melt and which lowers the melting point of AN.

Such compds include: $Ca(NO_3)_2 \cdot 4H_2O$, $Pb(NO_3)_2$, $Mg(NO_3)_2 \cdot 6H_2O$ and anhydrous Na acetate)

15) Dr A. Stettbacher of Zurich, Switzerland; private communication (1950). Following are Swiss fabrics and Dynamites manufd by them:
a) Schweizerische Sprengstoff-Fabrik AG, Dottikon (Aargau). It manufd: *Dynamit F* (NG or NG/NGc 62.5, Collod Cott 2.5, Na nitrate 27.0 & woodmeal or ryemeal 8.0%, to which was added 0.5% of prepd chalk (Schlämmkreide). Also were manufd other Dynamites, including *Aldorfit* & *Gelatine-Aldorfit*
b) Schweizerische Sprengstoff AG Cheddit & Dynamit at Liestal and Isleton. It manufd *Sprenggelatine* (Blasting Gelatin) (with or without NGc) and safety Dynamites, such as *Telsit* and *Gelatine-Cheddit*.
c) Société Suisse des Explosifs, at Gamsen-Brigue. It manufd Blasting Gelatin (with or without NGc) and safety Dynamites, such as *Gamsit* and *Gelatine-Aldorfit*

16) W.J. Taylor USP 2541389(1951) & CA 45, 4930(1951) (Powdery Dynamites are rendered cohesive by incorporating small quantities of polybutenes. E.g. NG 6.0, AN 80.4, Na nitrate 5.0, woodpulp 2.0, fine apricot-pit pulp 4.1, yellow corn flour 1.5, chalk 0.5 & 0.5% of a soln contg 7.5% of a solid polybutene in a liquid polybutene)

17) J. Taylor & G.P.Sillito, TransInstMiningEngrs **110**, 355–60(1951) & CA **45**, 8771(1951) [EqS expl "Unigex", equal in safety to sheathed permitted expls, such as NG (with NGc) 15.7–17.7, NC 0.1–1.1, Na nitrate 43.5–46.5, Amm chloride 28.0–30.0, woodmeal 1.0–3.0, wheat flour 0.5–1.5, Na carboxymethylcellulose 0.5–1.5, Ca stearate 0.1–1.1 & China clay 3.0–5.0%] [See also Giua **VI**(1)(1959), 388]

18) Ibid, **110**, 616–21(1951) & CA **45**, 8771 (1951) [EqS expl "Unikol", especially suitable for producing lump coal: NG (with NGc) 9.0–11.0, AN 34.1–37.1, Na nitrate 12.5–14.5, NaCl 28.9–31.9, plant fiber 9.0–11.0, resin 0.0–0.5, diammonium phosphate 0.0–0.5 & acid magenta 0.01–0.05%]

19) K. Hatsumi & Y. Tsurusaki, JapanP 146'51) & CA **46**, 11689(1952) (Waterproofing of a Dynamite may be achieved by incorporating a small amt of oxidized paraffin or higher fatty acid. E.G. NG 26.0, NGc 3.0, NC 1.1, AN 62.2, starch 4.5, TNT 1.5 & oxidized paraffin or fatty acid 1.7%)

20) K. Hino, JapanP 2247('53) & CA **48**, 4840 (1954) (A gelatinous Dynamite contg: NG 28.0, NC 1.2, AN 43.3, starch 1.0, Na chloride 20.0, soft wax 0.5 & urea 6.0% is pressed into sticks and wrapped in paper)

21) K. Hino & J. Sato, JapanP 6196('53) & CA **48**, 11790(1954) (Colloidal Dynamite consisting of: TNT 9.0, NC 1.0, AN 52.5, urea 8.0, urea nitrate 24.2 & GuN (Guanidine Nitrate) 5.3%)
Note. Mr H.A. Aaronson questioned the advisability of using GuN because the compd is unstable and gives off free nitric acid spontaneously. This would attack the NC rather quickly and make it useless as a binder [See also Giua, Trattato **VI**(1)(1959) 373]
21a) W.L. Reinhart, USP 2674526(1954) & CA **48**, 7904(1954) (Gelatin Dynamite compn contg pelletized sulfur)

22) R. Clogau, USP 2676877(1954) & CA **48**, 1322–23(1954) (Gelatinous Dynamite compn contg a surface-active agent, such as triethanolamine oleate added in quantity of 1%)

23) J.B. Bronstein, Jr, USP 2683164(1954) & CA **48**, 11062(1954) (A stable expl 1,2,6-Hexanetriol Trinitrate for use as substitute for NG in Dynamites is recommended. Its N content is 15.36%)

24) S. Mitsuma & Y. Nemoto, JapanP **3500** ('54) & CA **49**, 6610(1955) [Colloidal Dynamite contg NG gel 23.0, AN 64.85, wood pdr 1.0, K nitrate 5.0, starch 1.5, naphthalene 2.4, polyvinyl acetate: ethanol–60:40 paste 1.0 & $o\text{-}C_6H_4(CO_2Bu)_2$ 0.25%]

25) Atlas Powder Co & J. Smith Jr, BritP 725438(1955) & CA **49**, 14325(1955); USP 2727814(1955) & CA **50**, 4510(1956) (Incorporation in Gelatin Dynamites, in order to improve their plasticity and extrudability, of 0.01–0.1% of substituted oxazolines, such as 2-heptadecenyl-4-methyl-4-hydroxymethyl-2-oxazoline or 2-heptadecenyl-4-methyl-4(hydroxydiethoxymethyl)-2-oxazoline)

26) Max Bender, MP **38**, 109–28(1956) (Examination of Dynamite cartridges by acoustic method in order to determine the state of deterioration)

27) D.L. Kouba, USP 2759807(1956) & CA **51**, 719(1957) [Semigelatinous Dynamite, such as contg a liquid expl nitric ester 10–40, aromatic nitrocompds 5–30, hydrogenated terphenyl 1–5, NC 50–80, inorg oxidizing salt 50–80, carbonaceous material 1–12 and antacid 0.3–1.0 part]

28) C. Fréjacques, FrP1114381(1956)& CA **53**, 11840(1959) (Explosive oils by nitration of ethylene)

29) E.J. Russell, USP 2821466(1958) & CA **52**, 6795(1958) [Water-resistant Dynamites prepd by coating mixts, such as contg AN 72, Na nitrate 3.5, 1,1,1-Tris(hydroxymethyl)-ethane Trinitrate 1.0, NS (contg up to 13% N) 20, carbonaceous matter 3.0 & chalk 0.5%, with an adhesive material prepd by mixing at RT NS 5 parts with above Trinitrate 1.5 parts]

30) S.H. Davidson & Th.J Reid, USP 2845833 (1958) & CA **52**, 19144(1958) [Seismographic exploration expl units resistant to hydrostatic pressure prepd by heating thin metal cans contg AN 75% (at least) with usual fuels, sensitizers and up to 1% water. Temp of heating (usually in water-bath) must be such that only upper layer of expl in the can will melt and this layer will harden forming a crust serving to provide support against the collapse under pressure of the thin metal cans]

31) S.A. d'Explosifs et de Produits Chimiques, FrP 1156393(1958) & CA **54**, 18961(1960) (An expl mixt is obtd by adding to granular AN 4–8% of a combustible liquid, esp a petroleum hydrocarbon fraction, such as gasoline, refined oil or gas oil)

32) Ibid, French Addn 73386(1960) to the above patent; CA **56**, 14524(1962) (Sensitivity of above expls is increased by use of porous, low density, AN and by incorporating some pulped cellulose or other carbonaceous material; some methanol or ethanol may be added)

33) P.O. Marti, USP 2879133(1959) & CA **53**, 11840(1959) (Anticaking agent for dynamite-grade AN consists of finely divided particles of H_2S-treated ferric or ferrous ferrocyanide and their complexes)

34) F. Brown, USP 2879149(1959) & CA **53**, 11840(1959) (A low-cost expl prepd by mixing ca 20 parts of C black with 80 parts AN and then saturating with liquid O)

35) L. Avogadro, AnnChim (Rome) **49**, 352–57 (1959) & CA **53**, 15567(1959) [Sensitization of dynamite gelatin by inert additives, such as pumice, quartz, alum, hematite, pyrites, etc]

36) D.L. Coursen & F.A. Loving of DuPont Co, BritP 814590(1959) & CA **53**, 22956(1959) (Dynamites which do not lose their propagating power when immersed in water at high pressure)

37) W.H. Eckels, USP 2902355(1959) & CA **54**, 900(1960) (A plastic expl comprising a dispersion of HE material in a polysilicone)

38) A. Rost & G. Knöffler, GerP 1057514(1959) & CA **55**, 6868(1961) (Expls castable at <110° are prepd by combining the eutectic mixt consisting of 49% $NaNO_3$ and 51% $Mg(NO_3)_2$ with other ingredients, e.g. AN 50.65, eutectic 12.66, PETN 11.69 & RDX 25.00%)

39) G. Knöffler & A. Rost, GerP 1059333(1959) & CA **55**, 9880(1961) (Castable expls contg hexamethylenediaminedinitrate 10, RDX 20, AN 55, Ca nitrate 10 & Na nitrate 5%)

40) I. Huthanen. Explosivstoffe **9**, 189–94 (1960) & CA **55**, 2105(1961) (Potassium chlorate–oil mixtures as explosives)

41) M.A. Cook, Science **132**, 1105–15(1960) & CA **55**, 2108(1961) (Modern blasting agents. A review of developments in the past half decade with 36 refs)

42) N.A. Hamilton, USP 2932251(1960) & CA **55**, 3983(1960) [A self-contained, waterproof, expl cartridge is made from 90–96% granular AN, contg 3–7% TNT and 1–3% binder (such as starch, dextrin, or a synthetic hydrophilic gum) adhering to the surface of the granules. The granules are encased in an adherent protective coating 0.010-inch thick, consisting of 40–50% poly(vinyl ace-

tate)-poly(vinyl chloride) polymer, 40–50% gelatinized NC & 3–6% plasticizer (such as DEtPh)]

43) A.M. Ball, USP 2965466(1960) & CA **55, 7843**(1961) [An expl compn produced by mixing a small proportion of a polymerizable vinyl material with nonaromatic expls (such as azides), org nitrates (or org nitramines), and polymerizing in situ]

44) K. Hino & M. Yokogawa, KogyoKayaku-Kyokaishi **21**, 218–26(1960) & CA **55**, 11848 (1961) (Expls contg AN coated with liquid fuels, such as coal tar or petroleum)

45) K. Malkovsky et al, CzechP 96044(1960) & CA **57**, 6202(1962) (Gelatin Dynamites, such as NG 17.1, NGc 24.7, NC 1.9, DNT 3.8, AN 44.2, wood flour 3.3 & $BaSO_4$ 5%)

46) P.E. Wilt & R.D. Nelson, USP 2968541 (1961) & CA **55**, 7843(1961) [Nondusty Nitrostarch expls are obtd by using NS rendered nondusty by adding 0.75–1.25% of a tacky polybutene of viscosity 135–620 secs (210°F) or by adding a tacky soln of rubbery polybutene, natural rubber in a liquid polybutene. The tacky material is added as an emulsion, and the mixt is then dried. Emulsifiers, such as certain polyoxyethylene tallow amines or polyoxyethylene ethers of higher fatty alcohols; stabilizers, such as 0.25–1.00 mineral oil are included]

47) F.W. Cox, Jr & J.J. Roemer, USP 2997378 (1961) & CA **56**, 1664(1962) (Blasting expls consisting of porous AN prepd by special process, fuels & sensitizers)

48) Waternaaux & LeGall and État francais, FrP 1242324(1961) & CA **56**, 2628(1962) · [Permissible expls prepd by substituting reed meal, peat & NaCl by Al silicates (perlite type), kieselguhr, or by substances. of volcanic origin and by expanded plastic materials]

49) H.H. Fassnacht & D.L. Sagers, USP 2999014(1961) & CA **56**, 1664(1962) (A sensitive Dynamite free of NG and noxious fumes causing headaches is provided by a compn contg PETN 7–20, AN 75–90 & combustible material 3–10%. AN may contain up to 25% $NaNO_3$)

50) W.H. Eckels, USP 2999744(1961) & CA **56**, 1664(1962) (A plastic expl prepd by the polymerization of a mixt of catalyzed silicone gel and a cryst HE, such as RDX, TNT, PETN, etc)

51) L.E. Sentz, Jr & M.A. Curtis, USP 3003862 (1961) & CA **56**, 623(1962) (Dynamites waterproofed by amylopectin. E.g: NG 11.8, DNT oil 2.2, AN 37.0, $NaNO_3$ 36.4, woodpulp 2.0, amylopectin from potato starch 8.0, coal 1.6 & chalk 1.0%)

52) J.P. Blackwell, USP 3009801(1961) & CA **56**, 5012(1962) [Mixts of fertilizer-grade AN and a liquid hydrocarbon (such as Diesel oil), are sensitized to initiation by an ordinary blasting cap by adding a saturated soln of AN at >150°F, followed by drying]

53) D.L. Sagers, USP 3011883(1961) & CA **56**, 8988(1962) (Nontoxic Dynamite contg no volatile aliphatic nitrate esters, such as AN 62–89, PETN 2–7, $NaNO_3$ 0–25, Al 0.3–2.0 & combustible carbonaceous fuel 4–11%)

54) B.C. Winchell, USP 3013872(1961) & CA **56**, 8988(1962) [A waterproofed AN for use in expls, prepd by coating the prills or granules with 6% of a mixt of 3% polyethylene (mol wt 2000 to 25000) and 97% microcryst wax (of mp 145–190°F and ASTM color 1.5–2)]

55) G.B. Young, USP 3014789(1961) & CA **56**, 8988(1962) (Waterproof Blasting Explosive, consisting of a fertilizer-grade AN prepd by coating its prills by a resinate and lubricating oil, using a special process described in the patent)

56) P.R. deWilde, BritP 882555(1961) & CA **56**, 11876(1962) [A plastic explosive prepd by intimately mixing AN >60, a nitrate of an alkylamine (such as that of mono-, di-, or triethanolamine $\geqq 8$, H_2O up to 15% and dispersing thruout the dough-like mixt, so obtd, 20% of finely divided HE, such as PETN or RDX. A gelling agent (0.5–1%) is also present]

57) Consolidated Coal Co, BritP 883276(1961) & CA **56**, 15720(1962) (A Blasting Explosive consisting of AN and up to 8% by wt of normally liq hydrocarbon, such as No 2 fuel oil)

58) H. Zenftman & Th.J. Reid, BritP 883918 (1961) & CA **56**, 11875(1962) [An explosive prepd by stirring 24.5 parts granular poly (Me methacrylate) of mol wt 1000000 with 75.5 parts 98% HNO_3 to give a stiff, sticky gel of density 1.35 and of O balance near zero. After standing overnight the gel is extrusion-loaded into cartridges]

59) H. Schlueter, GerP 1095722(1961) & CA **57**, 10097(1962) [Gelatinized expls compounded of NGc, NC, MNT, AN (or $NaNO_3$), sawdust & Fe_2O_3 are suitable for mud-capping and for use under water]

60) A. Berthmann & E. Ludolphy GerP 1096813 (1961) & CA **56**, 3716(1962) (Waterproofing of Dynamites by octadecyl derivs, which are also suitable as sensitizing and flame-proofing agents)

61) Th. Klueber, GerP 1102623(1961) & CA **57**, 10097(1962) (Water-resistant AN expls prepd from AN shielded by coating with a mineral fat melting above 90° and solidifying at –20°. One of such fats was prepd by soaking Ba soap in a diester)

62) L.J. Novak, GerP 1106659(1961) & CA **56**, 622(1962) (Explosives from homogeneous semisolid or solid nitroparaffins are made by adding to liq nitroparaffins dextran nitrates or dextran sulfates at temps and pressures at which the nitroparaffins cannot detonate. Org amines may be added as sensitizers)

63) Ph.G. Butts & C.R. Fingerhood, GerP 1109577(1961) & CA **56**, 6232(1962) [Expl gas-forming masses consist of AN 70–80 intimately mixed with a binder 12–18 (based on rubber), Na barbiturate $\sim$1.3 & a ballistic modification agent 2–10% by wt. The agent may be GuN, NGu, RDX, or 2,4-Dinitrophenoxyethanol, single or together. The binder may consist of a mixed polymer of butadiene with acrylic acid, acrylonitrile or vinylpyridine]

64) E. vonHolt, GerP 1110562(1961) & CA **57**, 10098(1962) [An expl prepd by mixing 100 parts NC(of N=12.2%) (previously moistened with H_2O) with 40ps TeNMe, 20p Diglycol Dinitrate & 0.5 part stabilizer]

65) B.B. Enoksson, GerP 1111079(1961) & CA **57**, 10098(1962) (Storage-stable Dynamites contg AN treated with dodecylamine or octadecylamine)

66) E. Ludolphy & A. Berthmann, GerP 1112935 (1961) & CA **56**, 3716(1962) (Waterproofing of Dynamites without desensitizing NG using an aqueous emulsion of N,N^1-dimethylhexadecylamine, which is superior to the amine without emulsion)

67) F. Lebrun & L. Waterlot, GerP 1117014 (1961) & CA **57**, 1142(1962) [A mixt of NG 10, $NaNO_3$ 56, NH_4Cl 32.5, carboxymethyl cellulose 1, and a synthetic silicic acid (Hi-Sil 233) 0.5% gave a semigelatinous, stable expl safe against coal dust and mine gas]

68) A. Berthmann & E. Grosch, GerP 1119741 (1961) & CA **57**, 1143(1962) (Plastic expls consisting of PETN or RDX plasticized by mineral oils with Ba salts of long-chain acids, such as Ba laurate)

69) K. Shiino & S. Oinuma, JapanP 6797('61) & CA **57**, 6201(1962) (Gelatinous expls prepd by blending NC with bis(methylnitro) urea, AN & wood powder. A small amt of NG may also be added during blending)

70) J. Kota, HungP 146961(1961) & CA **58**, 1927(1963) (From Ref ZhKhim **1962**, Abstr No 2L448) (Expls suitable for blasting, such as NG 60 & carbon black 40% and NG 5.0, carbon black 6.8 & AN 88.2%)

71) N. Nakajo et al, KôgyôKayakuKyôkaishi **22**, 254–58(1961) & CA **58**, 2317–18(1963) (Addn of starch or polyvinyl alc during gelatinization of NG or NGc with NC gave uniform gel)

72) R. Heggstad & A.M. Heltzen, TekUkeblad **108**, 197–205(1961)(Norweg) & CA **62**, 394 (1965) (Expl compns from AN grit and 5–6% fuel oil)

73) Hercules Powder Co, BritP 893590(1962) & CA **57**, 1142(1962) [A Blasting Explosive suitable for underwater use is prepd by mixing 25 parts $NaNO_3$ with 16.8ps H_2O & 2.1ps of

Na carboxymethyl cellulose, followed by heating to 50–60° (to remove air bubbles), cooling and mixing with 41.9ps AN & 14.7 of a fine grade propellant]

74) Th. Lan, GerP 1125333(1962) & CA **57**, 1144(1962) (Plastic safety expls contg Trinitroaniline and NGc gelatinized with NC)

75) A. Berthmann et al, GerP 1123963(1962) & CA **57**, 2489(1962) (Water-contg expls prepd as H_2O-in-oil emulsions of NG or TNT, and with 1% emulsifier and stabilizer added. E.g. 50 ml NG, 50ml H_2O, 0.5 g Zn hydroxystearate & 0.5 gPyroxylin)

76) H. Aarflot, NorwP 99360(1962) & CA **57**, 8792(1962) [Expls consisting of AN & fuel oil, part of which may be replaced by sawdust, soot or nitrated hydrocarbons, Al stearate may be used as thickener of fuel oil. These expls my be incorporated into NG based expls]

77) A.G. Iroda, BelgP 613336(1962) & CA **57**, 14045(1962) [Expls prepd by mixing powdered AN, Al, K (or Na) nitrate with a gelatinous mixt of NG, NC and solvent, such as MeOH. After pressing to desired density the solvent is evaporated]

78) H. Mager, FrP 1292468(1962) & CA **57**, 15404(1962) (Expls contg metals or metal salts and coated with nitro compds, such as DNBz)

79) Dow Chemical Co, BritP 893260(1962) & CA **57**, 16954(1962) (An expl mixt prepd by combining granules of light metals, esp Mg & Mg-Al of 20–80 mesh, with aqueous solns of AN)

80) E.M. Scott Jr, USP 3016488(1962) & CA **58**, 407(1963) (Expls provided by mixing 70 parts uncoated AN prills, 30ps of $NaNO_3$ and 6ps Diesel oil)

81) J.M. Downward, USP 3018201(1962) & CA **56**, 13153(1962) (Gelatinous expl compns are provided by mixts contg expls such as NC, NS or RDX and a nonexplosive gel consisting of water-insol petroleum hydrocarbons such as a vulcanized copolymer of isobutylene and isoprene)

82) W.E. Kirst, USP 3026221(1962) & CA **56**, 14522(1962) [A Blasting Explosive, such as consisting of $NaNO_3$ prills 54, high density (essentially spherical) TNT 36 & FeSi 10%]

83) J.P. Blackwell, USP 3032450(1962) & CA **57**, 2490(1962) [An expl prepd from clay-coated fertilizer-grade AN by heating and agitating it with a combustible material which is liquid at the temp of operation (such as Diesel oil, kerosene, etc. Heating is continued until clay-coating is replaced by the combustible material. Then the clay-bearing excess liquid is removed and the compn is dried by sweeping with a gas, such as natural gas]

84) B.P. Enokson, USP 3039903(1962) & CA **58**, 1298(1963) [An expl or proplnt compn is prepd by coating AN with a C_{12-20} alkylamine, esp dodecyl-, hexadecyl- & octadecyl. Combustible materials (such as carbonaceous fuels or finely divided metals) and expl sensitizers (such as NG or TNT) are included]

85) W.A. Proell & W.G. Stanley, USP 3044912 (1962) & CA **57**, 12780(1962) (AN-cyanide expls, such as contg AN 81, $K_3Fe(CN)_6$ 4 & asphalt 15%)

86) S.R. Brinkley Jr & G.V. vonElbe, USP 3046887(1962) & CA **57**, 14046(1962) (Expl consisting of prilled AN and 2 to 6% fuel oil No 2 produced deton velocities ca 11100 ft/sec)

87) W.E. Gordon, USP 3046888(1962) & CA **57**, 14046(1962) (Expls of preceding patent were shown sensitive to deton by detonating fuse, such as filled with PETN)

88) A.W. Baker, USP 3047441(1962) & CA **57**, 12779(1962) (Expls prepd by mixing concd hydrogen peroxide with finely divided solid combustible materials, such as wood pulp, fine sawdust, bagasse, ground corn cobs, etc. Hydrogen peroxide can be stabilized with acetanilide, phosphoric acid or phosphate and may be replaced in part by AN)

89) Ch.W. Plummer, USP 3049570(1962) & CA **57**, 15404(1962) (Expls aliphatic and aliphatic-aromatic compns contg trinitromethyl groups)

90) C.O.Davis et al, USP 3052578(1962) & CA **57**, 15404(1962) (AN-based nitrocarbonitrate-type Blasting Explosive, which provide max efficiency because of their ability to eliminate all voids in a bore hole, are prepd by blending AN 80, satd aqueous AN 15 & petroleum oil 5%. A small amt of $N(C_2H_4OH)_3$ is incorporated in petroleum as emulsifier and some TNT may be added as a sensitizer)

91) C.O. Davis & J.E. Hughes, USP 3053707 (1962) & CA **58**, 1297(1963) [Blasting agent is obtd by allowing droplets of a molten mixt of AN & $NaNO_3$ (or other inorg nitrate) to fall into and thru a hydrocarbon fuel which is liquid below the mp of the nitrates. As result of this the droplets crystallize inwardly from the surface to form geods, the outer surfaces and inner cavities of which are covered and filled, resp, with fuel. This fuel is pulled thru the not-entirely-impervious walls as cooling and shrinking take place. Examples of fuels: kerosene and DNT]

92) O. Osborn et al, USP 3053710(1962) & CA **58**, 6642(1963) [Mixts of Mg hydride (up to 50%) with inorg oxidizers (such as perchlorates, chlorates, nitrates, permanganates or persulfates) constitute expls which can be stored under moist conditions, do not change in sensitivity with time and are easy to handle]

93) H.H. Fassnacht & D.L. Sagers, GerP 1130342(1962) & CA **58**, 4370(1963) (Dynamites causing no headache, such as contg PETN 7–20, AN 75–90 & combustible matter 3–10%. One quarter of AN may be replaced by $NaNO_3$)

94) E.M. Scott, Jr, USP 3061488(1962) & CA **58**, 4370(1963) [Correction of USP 3016488 (1962); CA **58**, 407(1963)]

95) M.R. Zhivadinovich & R. Zhivadinovich, USP 3066139(1962) & CA **58**, 6640(1963) [High-energy fuel & explosive, such as prepd by mixing 2 moles hexamethylenetetramine, 1 mole $Al(NO_3)_3.9H_2O$ and 40% $KClO_3$ based on the combined wt of the above two]

96) Ch.E. Silk & G.R. Stambaugh, BelgP 614453(1962) & CA **58**, 8846(1963) (Water-resistant slurry expls contg particulate NC & AN with sufficient water to give such viscosity that the particulate material remains in suspension and hence in uniform distribution thruout the slurry. The viscosity may be improved by adding thickeners such as guar gum, polyacrylamides, etc, with or without cross-linking agents. Up to 50% NC may be replaced with TNT, Tetryl, PETN, RDX or HMX up to 25% AN may be replaced with alkali metal nitrates, such as $NaNO_3$)

97) F. Frutiger, BelgP 614511(1962) & CA **58**, 1298(1963 [Plastic AN expls contg no NG but a plasticizer (such as MNBz, DNBz or TNT), swelling agent (such as agar, dextrin, starch or polyvinyl alc), oxidizing agents (such as perchlorates of Ca, Mg & Mn) and fuel-oil compns)

98) Dynamit-Nobel AG, BelgP 616720(1962) & CA **58**, 408(1963) (Desensitation of NG or other nitric esters by polyglycols)

99) S.R. Brinkley & R.W. Van Dolah, "Research and Technologic Work on Explosives, Explosions and Flames", Fiscal Year 1960, USBurMinesInformCirc No **8106**, 39pp(1962) & CA **58**, 6637(1963)

100) R.J. Grubb, InternSympMiningRes, Proc-Symp, Columbia, Mo **1961**, Vol **1** (Pub 1962), pp15–27 & CA **58**, 5443(1963) (Some factors influencing the explosive properties of AN-fuel mixtures)

101) G.B. Clark et al, Ibid, pp29–45 & CA **58**, 5443–44(1963) (Performance parameters of densified AN-fuel blasting agents)

102) D.T. Bailey et al, Ibid, pp47–57 & CA **58**, 5444(1963) (Slurry explosives for use underground)

103) Ulf Henning, Ibid, pp59–66 & CA **58**, 5444(1963) (Blasting with AN-fuel oil explosives underground at Boliden)

104) R.W. Van Dolah et al, Ibid, pp77–89 & CA **58**, 5444(1963) (Fumes from AN-hydrocarbon mixtures)

105) K. Hino & M. Yokogawa, Ibid, pp1–13 & CA **59**, 13760(1963) (AN-fuel-surfactant explosives. Their fundamentals and performance)

106) M.H. Hoepli & Theo Lang, FrP 1301358 (1962) & CA **58**, 5448(1963) (Plastic expls with aromatic nitramine base, such as contg TNA 75–84, NGc 15–24 & NC 1%. Up to 30% NGc may be replaced by NG, and up to 10% powd Al can be incorporated to increase the deton rate)

107) A. Berthmann, GerP 1141572(1962) & CA **58**, 6640(1963) [Readily detonated water-containing expls. Addn to GerP 1123963 (1963); CA **57**, 2489(1962)]

108) Albright & Wilson Ltd, BritP 910374 (1962) & CA **58**, 6640(1963) (Slurry Blasting Expls, such as comprising $NaClO_3$ 62 & TNT 38% with added 12 parts water, contg 1.6% guar gum)

109) Dynamit-Nobel AG, BelgP 619617(1962) & CA **58**, 7782(1962) [Detonable emulsions, such as contg NGc 20, DNT 6, Pyroxylin (stabilizer) 0.8, Diesel oil 0.9, lanolin 0.1, AN 71, wood flour 1 & Fe_2O_3 0.2%]

110) ZakladyChemiczne "Krywald", PolP **45**, 579(1962) & CA **58**, 8846(1963) (Rock Blasting expl, such as contg AN 76, ferrosilicon 15, TNT 3, NG 4 & wood flour 2%)

111) E.B. Hall & Bird & Co, IndianP 73391 (1962) & CA **58**, 13704(1963) [Substitution of part of AN with K or Na nitrate in mixtures with Diesel oil resulted in less deliquescent FO (fuel-oil) explosives]

112) Dynamit-Nobel AG, BelgP 622458(1962) & CA **58**, 13706(1963) (Improved sensitivity, power & brisance of AN expls are achieved by heating AN crysts with liquid fuel to above the crystal transition point. Upon cooling, fissures form in the AN particles and the liq covers these new surfaces, as well as the old ones and this causes the improvement. E.g. AN 54.9, NGc 22, Collod Cotton 1, $NaNO_3$ 10, DNT 6, TNT 5, wood flour 1 & Fe_2O_3 0.1%)

113) Z.G. Pozdnykov, Vzryvnoye Delo, Nauchn-TekhnGornObshchestvo, Sbornik No **49/6**(1962); Ref ZhKhim **1963**, Abstr No **2N321** & CA **59**, 2585(1963) (Rus commercial explosives based on granulated AN, impregnated with fuel oil and contg other ingredients, such as TNT, powd Al, etc)

114) B Ya Svetlov, Ibid, **49/6**(1962); Ref ZhKhim **1963**, Abstr No 20N/390 & CA **61**, 6849(1964) (Modern Russian nonpermissible explosives and methods for their improvement) [Included are **Ammonits** No 6, 7, 9 & 10; **Igdanits, Granulits,** and **Granulated Granulits.** They are described later in this Volume]

115) M. Aoki & Sh. Motoya, JapanP 4444('62) & CA **60**, 6695(1964) (A slurry compn is obtd by pouring molten TNT at temp 90° into an agitated vessel thru which hot water was passed. After further agitation the material was dehydrated to 10% water content and packed into polyethylene containers. This method produced TNT of varied particle size)

116) K. Tsujibashi, JapanP 15798('62) & CA **60**, 5270(1964) (Permissible expls such as prepd by mixing NG 6, NC 0.1, low-d AN 61.2, TNT 2, woodmeal 2, starch 2 & "reducing agent" 26.7%. The "reducing agent" is prepd by mixing H_2O 8, gum arabic 10 & 150 mesh NaCl to form a paste. The paste is then dried at 50° & 30mm for 1 hr and then passed thru a 20-mm sieve)

117) R.E. Barnhart & F.C. Sawyer, USP 3072509(1963) & CA **58**, 6641(1963) [Gelled AN Blasting Explosive consisting of an HE (such as TNT or RDX), satd aq AN & guar gum with its cross-linking agent borax. Part of AN can be replaced by $NaNO_3$]

118) G.B. Barany & J.D. McIrvine, CanP 658221(1963) & CA **58**, 12304(1963) [Improved aqueous slurries contg, by wt 8–26% water, 15–40% granular HE's (such as TNT, Pentolite, Comp B or smokeless proplnt) 23–75% oxidizing salt (such as AN with/or without $NaNO_3$), and small amts of carbon black]

119) G.L. Griffith et al, USP 3082689(1963) & CA **58**, 13704(1963) [Cartridges of insensitive Dynamites (such as contg AN 85 & ground anthracite coal 15%) can be easily detonated if they are sheathed in a more sensitive expl (such as Dynamite contg AN 40%)]

120) G.L. Griffith & F.B. Wells, USP 3083127 (1963) & CA **58**, 13704(1963) [Aqueous Nitrostarch expl slurries, having high power, brisance & sensitivity to initiation and low sen-

sitivity to impact are provided by mixts contg 15–25% NS, 50–75% inorganic nitrate (such as AN alone or mixed with Na nitrate), 10–40% water, conventional fuels (and/or pulverized metals), thickeners and neutralizers]

121) Dynamit-Nobel AG, BritP 922931(1963) & CA **58**, 13705(1963) [The cryst water-sol ingredients of expls are waterproofed by spraying their surfaces with a soln of hydrophilic materials (such as $CaCl_2$ or $ZnCl_2$), followed by spraying with a soln of ionic waterproofing agents (such as stearic acid, NH_4-stearate, or Na higher-alkyl iminodiacetate, whereby a waterproofing substance is formed *in situ* on the surface of the particles. Small amts of HE's, such as NG (gelatinized with NC) or Tetryl are also incorporated]

122) F. Mueller, GerP 1143425(1963) & CA **58**, 13705(1963) (AN expls with reduced impact sensitivity are prepd using AN previously treated with 0.05–0.1% of 4-aminodiphenylamine-2-sulfonic acid or 4,4'-diaminodiphenylamine-2-sulfonic acid. Other ingredients of expls are TNT, DNT & woodmeal)

123) Max Wolf, GerP 1144637(1963) & CA **59**, 1434(1963) (Increased deton propagation and velocity of AN expls are obtd by heating AN crysts to ca 85° and then cooling them to ca 25° with agitation, to achieve cryst transition. Treatment can be carried out twice. Other ingredients of expls with treated AN are TNT and woodmeal)

124) F.H. Fenix, USP 3090713(1963) & CA **59**, 1434(1963) [Permissible expl prepd from fertilizer-grade AN prills (with/or without $NaNO_3$) and small amts of fine Al, Fe_2O_3, fuel (such as activated charcoal), sulfur, ground glass & lime dust)

125) F.H. Fenix, USP 3090714(1963) & CA **59**, 1434(1963) [A dehydrating mixt of $NaClO_3$ (or $KClO_4$) & $PhNO_2$ with atomized Al & PbO_2 are incorporated in mixt of above patent]

126) E. vonHolt, GerP 1147159(1963) & CA **59**, 4970(1963) [Homogeneous solid expls prepd by gelatinizing either highly nitrated hydrocarbons with a small amt of polyalcohol nitrate or moderately nitrated hydrocarbons (such as NC) with larger amts of energy-rich polyalc nitrates, such as NG or NGc. Al grindings may be incorporated]

127) H. Riedl & H. Schlueter, GerP 1148924 (1963) & CA **59**, 3712(1963) (Non-extrudable Gelatin-Dynamite is prepd by gelatinizing NG with 12–12.15% N–NC, part of which was degraded)

128) H.E. Staadt, USP 3091559(1963) & CA **59**, 4970(1963) [A low-cost cap-insensitive expl is provided by a mixture of 8–10 mesh fertilizer-grade AN 58–83, *Driver's Liquid* (satd soln of AN in anhyd NH_3) 14–42 & 0.5–5%. *Spensol D*(a soln of AN 60, NH 34 & water 6% may be substituted for the Driver's Liquid, and water-sol high-mol-wt natural or synthetic gums may be added as thickeners]

129) J.R. Hradel & C.K. Bjork, USP 3094069 (1963) & CA **59**, 8540(1963) [Blasting Explosive prepd by mixing FGAN with Al (or other metals such as Mg, Mn, Zn, etc) and kerosene]

130) J.H. Hoffman & H.L. Bowkley, USP 3097120(1963) & CA **59**, 8540(1963) [Gelled expls in non-settling slurry are provided by a compn contg FGAN 30–40, $NaNO_3$ 22–24, TNT (RDX or proplnt) 28–32, water 8–15, a gelling agent (such as polyacrylamide) 1–2, and an acidic cross-linking agent (such as Al sulfate or borax) 0.1–0.2 part]

131) A. Kunz, HungP 150115(1963) & CA **59**, 8540(1963) (An improved expl prepd by mixing highly dispersed AN with pectized sodiocellulose or NC in volatile solvent, and evapg solvent with simultaneous granulation of the mass)

132) D.L. Coursen & F.A. Loving, USP 3101288(1963) & CA **59**, 9731(1963) (An improved Gelatin Dynamite with good plasticity and loading characteristics, which retains its propagating power when exposed to moisture or high water pressure, is provided by incorporating in a compn such as: NG 50, NC 2.2, $NaNO_3$ 32.8, chalk 0.5, starch 4.5 & unaccounted 1%, up to 4 parts of water-insol thermosetting synthetic resin balloons of 2–360 μ-diameter)

133) E. Yoshikawa, et al, JapanP 397('63) & CA **59**, 13763(1963) (Powdered AN expls prepd by mixing ingredients, such as AN 60, $NaNO_3$ 12, KCl 4.5, NaCl 3.5, DNN 7, flour 1 & urea 3%, followed by heating to 120° with agitation. The resulting melt is then sprayed thru a nozzle into a dry, cool flue to crystallize as a powder of desirable particle size)

134) S. Grossmann, USP 3103457(1963) & CA **59**, 13764(1963) (A porous AN is prepd by heating nonporous pelleted AN existing in cryst form IV at 32–84° until it is converted to form III. After cooling and screening the material it is impregnated with 3–5% hydrocarbon oil, such as naphtha, kerosene, gas oil or fuel oil. This gives an expl of density 0.93–1.0 suitable for blasting purposes)

135) J.R. Hradel & M.F. Hradel, BritP 936042(1963) & CA **59**, 13765(1963) (Expl compn contg a light metal sensitizer. E.g. AN prills 50.7, Mg billet scalpings 23.1, coarse Al chips 17.2 & urea 9.0%)

136) J.D.McIrvine, USP 3108917(1963) & CA **60**, 1534(1964) [TNT-tetraborate gelled, aqueous explosive slurry which does not gel up to 24 hrs after prepn is provided by a mixt of 15–40% particulate TNT, 60–40 Comp B or smokeless proplnt; 31.5–77% granular AN (alone or in admixt with NH_4 chlorate or perchlorate); 8–26% water; 0.1–2% monogalactan and 0.002–0.2% (calcd as B_2O_3) of fused, glassy tetraborate]

137) C.J. Breza, GerP 1155044(1963) & CA **60**,5270(1964) [Granulated expls contg 30–60% AN particles (of smaller than 0.147 mm), 60–90% (of smaller than 0.208 mm) and 3–25% by wt ferrophosphorus]

138) N. Tomoishi & K. Oishe, JapanP 16148 ('63) & CA **60**, 7866(1964) (Conventional Gelatin-Dynamites show a decrease in rate of deton and propagation of deton when the pressure of the surrounding medium becomes higher than 5 kg/cm² Incorporation of 1.5 to 2.5% of Fe_2O_3 pdr renders Dynamites detonating perfectly at pressures far higher than 5 kg/cm²)

139) W.A. Gey & R.W. Van Dolah, USP 3110640 (1963) & CA **62**, 1508(1965) [Expl mixts contg an HE, such as RDX, exhibit improved insensitivity to mech shock when they are mixed with 5–20 parts of org fluorine compd such as NH_4 perfluorocaprylate, an ether of dihydroperfluorobutanol, or poly(chlorotrifluoroethylene)]

140) W.O. Ursenbach & L.L. Udy, USP 3113059 (1963) & CA **60**, 7866(1964) (Slurries contg Al, water, AN & other ingredients, such as $NaNO_3$, TNT, ferrophosphorus, smokeless proplnt, NGc, guar gum & sulfur, may be prevented from gassing by addn of 0.1–2.0% of an inhibitor such as tri-, di-, or mono-basic phosphate of NH_4 or an alkali metal)

141) R.A. Cooley, USP 3113060(1963) & CA **60**, 6696(1964) (A blasting agent contg equal parts of whole-prill and whole-prill with crushed prill AN blended with No 2 fuel oil 5% and Ethyleneglycol Dinitrate 2%)

142) H.J. Fisher, USP 3116189(1963) & CA **60**, 7865(1964) [A plastic expl prepd from 76 parts RDX, 8 ps of 1:2.5:0.75 mixt of polyisobutylene binder, bis(2-ethylhexyl) sebacate and its plasticizer; also 8 ps of a C_{8-18} nonaromatic hydrocarbon oil, 5 ps DNT oil and 3 ps MNT oil]

143) I. Hayashi & T. Sato, JapanP 16147 ('63) & CA **60**, 9094(1964) [A gelatinous Dynamite can be prepd by dipping NC (1.2 parts) in a paraffin emulsion contg water, paraffin & surfactant previously mixed with 30 parts NG-NGc soln and the resulting mixture kneaded with AN 63.8 starch or wood flour 4.5 & oily material 0.5 parts. It is claimed that this method of prepn requires less time than the conventional method)

144) T. Sato, JapanP 3445('63) & CA **60**, 10472(1964) [A gelatinous Dynamite is obtd by incorporating liquid Dinitroethylbenzene (or DNBz) and naphthalene into mixt contg NG, NC & AN]

145) N. Tomoshi & M. Masu, JapanP 9590 ('63) & CA **60**, 9094(1964) [Commercial non-NG expls are prepd by mixing 0.5% polyethyleneglycolmonostearate with the material consisting chiefly of AN, sensitizers (such

as TNT or Nitronaphthalenes), fuels (such as woodmeal, starch & heavy oil) and NaCl]

146) K Hino et al, JapanP 9591('63) & CA **60**, 7866(1964) (AN-fuel expls obtd by replacing part of the oil by phenol, cresol, xylenol, aniline, etc)

147) I. Hayashi & T. Sato, JapanP 16147('63) & CA **60**, 9094(1964) [A gelatinous expl prepd by dipping 1.2 parts of NC in ca 1 part emulsion consisting of water, nonionic paraffin & surfactant (such as Tween). After mixing the product was kneaded with AN 63.8, starch or wood flour 4.5 and an oil material 0.5 parts]

148) K. Ito & Y. Inumaru, JapanP 20394('63) & CA **60**, 10472(1964) (A waterproof, semi-gelatinous, permissible Dynamite is obtd by incorporating in formulations contg AN, Amm perchlorate, Na nitrate, nitrocompound, etc up to 5% of N,N'-methylene-bis-amide or a methylolamide of a C_{12-18} fatty acid, singly or together with other waterproofing compns)

149) Th.W. Royer & J.S. Brower, BelgP 624796 (1963) & CA **60**, 13090(1964) [Blasting expls contg AN prills and a lower nitroalkane, such as $MeNO_2$ (preferred), $EtNO_2$, $PrNO_2$, $BuNO_2$, or their mixts and a low fatty alc, such as MeOH (preferred), EtOH, PrOH, BuOH or their mixts]

150) Ibid, BelgP 624797(1963) & CA **60**, 13090(1964) (Blasting expls consisting of porous AN prills and nitroparaffin mixts contg a coupling agent such as a ketone)

151) Aerojet-General Corp, BelgP 627768 (1963) & CA **60**, 13091(1964) [Expls which can be prepd at the site and poured into a borehole are provided by mixts contg lower nitroalkanes (such as $MeNO_2$, etc) and a sensitizer (such as aralkyl amine, etc). Absorbents (such as sawdust, powd coal, charcoal or starch) may be used to produce solid expl compns]

152) Wasag-Chemie AG, BelgP 630156(1963) & CA **60**, 11840(1964) (Cap-sensitive expls such as: AN 90, NH_4SCN 8, & Diesel oil 2% or AN 86 & NH_4SCN 14%. Omission of NH_4SCN resulted in compns not cap-sensitive)

153) Nitroglycerin Aktiebolaget, BelgP 628844 (1963) & CA **61**, 1701(1964) [Expls having little physiological effect and low impact sensitivity contain large amts of 1,2- or 1,3-propyleneglycol Dinitrate (in lieu of NG & NGc) and of Amm & Na nitrates, together with small amts of NC, DNBz(or DNT), wood flour & chalk]

154) N.S. Bakarevich, VzryvnoyeDelo, Sbornik No **52/9**,189-9(1963);ReferZhKhim **1964**, Abstr No **7N433** & CA **61**, 13116(1964) [Sensitization of Russian permissible AN expls with liquid nitro esters such as 60:40 mixt of NG & DEGDN)]

155) Norsk Spraengstofindustri A/S, NorwP 102157(1963) & CA **62**, 8926(1965) [Powdered expls prepd by coating AN grains with fusible org compds (such as a paraffin, wax, resin, aromatic or aliphatic nitrocompd, nitramine, etc), previously heated to above their mp's. Cooling was achieved with air at 20°]

155a) I. Fukuyama & T. Kai, KôgyôKayaku-Kyôkaishi **24**(3), 140–47(1963) & CA **63**, 6677(1965) (Two types of NG contg expls with the salt pairs NH_4Cl/KNO_3 and $NH_4Cl/NaNO_3$ were investigated. Both types offered greater safety than the usual mining expls. However, they detonated incompletely and left a solid residue with an ammoniacal smell. Expl contg the salt-pair $NH_4Cl/NaNO_3$ was hygroscopic and gave a poorer transmission value, but it was less expensive than expl contg K salt)

155b) W. Hofmeister, Explosivstoffe **11**(2), 27–34 & **11**(3), 56–63(1963) & CA **66**, 1242 (1967) (Effect of packing on blasting as a function of priming-cartridge position)

156) K.W. Nielson, USP 3118796(1964) & CA **60**, 10472(1964) [Trilaminar expl was prepd by coating granular AN at 115° with 2–5% Nitronaphthalene (mp 90°). Then the temp was reduced to 80°, and the grains coated with 2–20% TNT (mp 74°). Finally the mass was placed in a rotary drum with wood balls (maintained at 50°) and, while agitating, the grains were coated with 2–10% mixture of Al, activated charcoal (or powd coal)]

157) Dow Chemical Co, BritP 947046(1964) & CA **60**, 11840(1964) (An expl prepd from a mixt of coarse Mg & Al chips and an aqueous

ammoniacal soln of AN was placed in a plastic container and inserted in a borehole)

158) J.R. Hradel, USP 3124495(1964) & CA **60**, 14328(1964) [Expl compns are provided by a mixt contg AN 10–83, particulate light metal (such as Mg, Al or their alloys) 15–60 and liquid carrier (such as 85% aqueous ammoniacal AN soln) 2–35%]

159) Dow Chemical Co, BritP 952837(1964) & CA **61**, 528(1964) [Expl compns contg ammonia, such as 85 parts of a soln (consisting of AN 69.8, liquid NH_3 23.8 & water 6.4%), 7.5ps Mg shavings and 7.5ps Al shavings]

160) E. Saelzle, GerP 1167615(1964) & CA **61**, 4144(1964) (Expls prepd by treating porous AN grains with vapors of volatile organic substances. Immediately after the adsorption of vapors, the vapors are sealed by a coating with small amts of waxes)

161) H.G. Carlevato, USP 3129126(1964) & CA **61**, 4143(1964) (A high-bulk strength slurry-type blasting compn, such as contg AN 26.5, water 11.0, TNT 27.0, $NaNO_3$ 15.0, ferrophosphorus 20.0 & guar gum 0.5%)

162) J.S. Logan & J.A. Zaslowsky, USP 3135635(1964) & CA **61**, 5445(1964) [Expl compn prepd by tumble mixing 186.2 g AN (100% <40-mesh) with 13.8 g yellow solid (100% <100-mesh), obtd in pyrolysis of boron hydride B_2H_6]

163) Ch.M Saffer Jr, USP 3135637(1964) & CA **61**, 5446(1964) (AN-aliphatic hydrocarbon-urea clathrate expls)

164) W.E. Proell & W.G. Stanley, USP 3137598 (1964) & CA **61**, 5445(1964) (An AN expl contg hydrogen-sulfide treated Prussian blue)

165) Dow Chemical Co, BritP 952838(1964) & CA **61**, 6852 (1964) (A nearly saturated soln of 3.5 lb AN in water was admixed with 1.5 lbs Mg band-saw chips and the resulting mass placed in a borehole. After undergoing autoreaction the load was fired 48 hrs later electrically with a shaped chge and a crater 6 ft in diam was produced. No crater was produced on firing 15 lb load contg FGAN 94 & fuel oil 6%)

166) Nitroglycerin Aktiebolaget (Sweden), BritP 954114(1964) & CA **61**, 2898(1964) [Expls contg AN powd (coated with ca 0.1% of mono- or di-octadecylhydrazine, stearoylhydrazine, etc) 100 parts, NGc 6, wood flour 4 & Al 4 parts]

167) E.E. DeMaris, USP 3140212(1964) & CA **61**, 6852(1964) (Blasting expls useful in oil-well perforating operations are provided by a mixt of 95–98% Tetranitro-2,3:5,6-dibenzo-1,3a,4,6a-tetrazapentalene or Tetranitro-2,3:4,5-dibenzo-1,3a,6,6a-tetrazapentalene and 2–5% TNB or TNT. Other ingredients, such as $KClO_3$ or PbO_2 may be present)

168) P.H. Cardwell, GerP 1171786(1964) & CA **61**, 8124(1964) (Satd soln of AN in liquid NH_3 is pumped into drill holes and penetrates permeable earth layers. Shaped charge fuse is used for initiation. This method permits one to explode the total chge, because of the continuous distribution of expl in the earth formation)

169) A.H.B. Weeks, CanadP 688413(1964) & CA **61**, 9350(1964) [Slurried Blasting Explosives consisting of 5–15% (by wt) of a granular org HE (such as PETN, Tetryl, RDX, etc), 5–25% of metal (such as Al, Mg, B, etc), 17–20% water, 0.5–1.5% gelling agent, and 0.5–1.5% oxidizing salt (such as AN or its mixt with $NaNO_3$)]

170) A.H.B. Weeks, CanadP 688577(1964) & CA **61**, 9350(1964) [A Blating Explosive consisting of AN (or its mixt with up to 45% $NaNO_3$) up to 75, Pentolite 4–12, water 12–20, fuel (unspecified) 2–15 & gelling agent 0.5–2.0%]

171) A.H.B. Weeks, CanadP 688919(1964) & CA **61**, 8123(1964) (Explosive slurries of of improved sensitivity to initiation, such as comprising: AN 45.3, $NaNO_3$ 22, TNT 15, water 12.7, guar gum 1 & sugar 4%)

172) T. Yoshida et al, KôgyôKayakuKyôkaishi **25**(2), 76–84 (1964) & CA **61**, 11838(1964) (AN-FO Blasting Explosives)

173) G.L. Griffith & F.B. Wells, USP 3147163 (1964) & CA **64**, 11841(1964) [Expl slurries of

excellent power, brisance, and sensitivity despite low O balance are provided by mixts of 50–75% inorg oxidizer (such as AN), 15–25% expl sensitizer (such as DNT, TNT, RDX or PETN), 1–25% metal fuel (such as Al or ferrosilicon), 7–50% oil (such as kerosene, fuel oil, etc) and 0–10% water]

174) African Expls and Chem Inds Ltd, BritP 970975(1964) & CA **61**, 15924(1964) (Expl compns prepd by first mixing prilled AN with fuel oil and then adding a solid fuel, such as milled anthracite and light metal or alloy)

175) W.C.G. Baldwin, BritP 973770(1964) & CA **62**, 1508(1965) (A gelatinous expl consisting of 60:40–NG:NGc mixt 16, MNT 1.3 & NC 1.3 parts gelled together and mixed with Na carboxymethyl cellulose 0.5, AN 56.2, Na nitrate 8, wood meal 4.7, wheat flour 5.0 & water 7.0 parts)

176) D.S. Partridge & W.S. Partridge, USP 3148095(1964) & CA **62**, 2662(1965) (A blasting mixt which detonates in the presence of a certain amt of water is prepd from FGAN prill coated successively with paraffin, a mixt of gilsonite with A145 Petroleum Wax, mica and Al)

177) J.S. Logan & J.A. Zaslowsky, USP 3150019(1964) & CA **61**, 14460(1964) (Blasting Explosives, such as AN 94.5, kerosene 5 & decaborane 0.5%)

178) C.J. Breza & C.H. Noren, USP 3153606 (1964) & CA **61**, 15924(1964) (Slurried Blasting Explosives contg Al & AN, such as: AN 51.7, Al pdr 4.0, $NaNO_3$ 15.2, water 22.1, guar gum 1.9 & powd coal 5.1%)

179) W.A. Proell & D.J. O'Connor, USP 3154446 (1964) & CA **62**, 1508(1965) (AN compns contg pyridazinedione combustion catalyst)

180) C.W. Vriesen, USP 3155552(1964) & CA **62**, 395(1965) [A castable expl compn provided by curing a mixture contg 50–80% powdered hydrazine nitrate, 13–40% binder (such as a polyester of mol wt 500–5000 derived from polyhydric alcs and polybasic acids), and 3–10% curing agent (such as a diglycidyl ether of bisphenol A or F), powd metals (such as Mg, Al, Ti, etc) 5–15%, and minor propns of burning rate modifiers (such as Amm dichromate), modifiers (such as polyethylene- and polypropylene-glycols), curing rate modifiers (such as Mg stearate) and curing catalysts, such as 4,4'-methylenebis(2-chloroaniline), n-phenylenediamine & tris(dimethylaminoethyl) phenol may be present also]

181) Imperial Chemical Industries Ltd, NethAppl 6401151(1964) & CA **62**, 3877(1965) [Expl consisting of AN sensitized with >3.5% of a solid thermosetting formaldehyde (-melamine or -phenol) resin (as an alk soln <10% of the wt of AN) and dried in air at >70°]

182) M.A. Cook, USP 3155554(1964) & CA **62**, 3877(1965) [Liquid-blanketed chlorate or perchlorate blasting agents are prepd by mixing Na chlorate or perchlorate particles (or their mixts with other oxidizers such as nitrates) with liq fuels (such as fuel oil, benzene, toluene, nitromethane, 2-nitropropane, o-MNT, DNT, etc). The solids are introduced into a borehole and then the liquid fuel (in amt required to achieve "zero O balance") is poured over solids to cover them]

183) M.A. Picciano & B.V. Carlson, USP 3156185(1964) & CA **62**, 1508(1965) (An expl mixt, such as contg AN 80–85 parts and 15–25ps Al powd, can be initiated by a bridge wire)

184) A. Koenig, FrP 1368222(1964) & CA **62**, 6334(1965) (Expls in which urea nitrate is substituted for AN are claimed to be less sensitive to atm moisture. A mixture contg 20% TNT is satisfactory)

185) R. Kaltenbach, FrP 1368827(1964) & CA **61**, 15702(1964) (Expls consisting of porous AN granules with fuel oil absorbed in pores)

186) H. Mager, FrP 1370801(1964) & CA **62**, 3877(1965) [The disadvantages of on-site mixing of AN & fuel oil can be avoided if the AN (93–96 parts) is kneaded in a mixer with 1–4ps colloidal grease of up to 130° Engler viscosity (such as naphtha or petroleum thickened with stearic acid etc) and glycerol which contains 1–4ps powd Al, B, Bi, Mg etc]

187) K.W. Nielsen, DanP 99972(1964) & CA **62**, 7580(1965) (An expl with a deton vel ca 6000 m/sec prepd by heating to 115° 77 parts of granulated AN with 2–5 ps Nitronaphthalene, 2–10 ps gelatinized liquid TNT & 2–10 ps Al bronze flakes or 2–10 ps finely divided activated charcoal)

188) F.B. Wells, USP 3160535(1964) & CA **62**, 5135(1965) (Free-flowing granular blasting expl of controlled particle size, esp suited for use in wet holes, is provided by a mixt consisting of particulate water-insol HE's, such as TNT, Pentolite, Comp B or Tetryl and a finely divided water-sol oxidant, such as nitrates or perchlorates which fill the interstices betw insol expls particles. AN or NH_4NO_3 waterproofed with a wax or rosin may be used instead of insol HE's. In this case water-sol, OH contg fuels, such as alcohols, glycols, glycerol, etc can be added)

189) R Aitchison, USP 3160536 (1964) & CA **62**, 5134(1965) [Blasting expl contg granular AN, a liquid hydrocarbon (ca 10% of expl wt) and a metallic soap. Thus 7.5% by wt Al soap of 2-ethylhexoic acid was added to kerosene and dissolved by stirring and heating to 120–150°F. To the jelly-like soap obtd on cooling, AN prills were added to form a free-flowing slurry)

Note: USP 3064572(1962) included the same info as in the above patent and also gave description of method and app for charging the borehole

190) J.A. Zaslowsky & J.S. Logan, USP 3160538 (1964) & CA **62**, 5134(1965) (Water-resistant gelatin blasting expls contg AN, water, HCl and a gel-forming proportion of a mixt of guar gum and glutaraldehyde)

191) R.S. Egly & A.E. Neckar, USP 3161551 (1964) & CA **62**, 12969(1965) [A water-in-oil emulsion, such as contg AN 60.9, water 26.1, fuel oil 11.0 & 1-heptadecyl-4,4-bis(hydroxymethyl)-2-oxazoline 2.0% is poured into a borehole filled with 4 parts of AN grains]

192) N.E. Gehring, USP 3164503 (1965) & CA **62**, 6334(1965) [Blasting compns contg an aqueous soln of HNO_3, AN and an insensitive carbonaceous fuel which is immiscible with HNO_3 soln. Such fuels can be petroleum products (such as Diesel oil), paraffinic waxes, vegetable oils and finely divided C]

193) J.F.M. Craig et al, CanP 697802(1964) & CA **62**, 7580(1965) [Slurried Blasting Explosives which remain pourable up to 24 hrs after mixing are prepd from 30–75% inorg oxidizer (such as AN and/or an alkali or alkali-earth metal nitrate); 15–45% of particulate org expl (such as TNT, PETN, RDX, Pentolite or Comp B); 0.2–2.0% of gel-forming polysaccharide (such as mannogalactan); 0.005–0.1% of cross-linking agent (such as $Na_2Cr_2O_7$, KCr_2O_7, $ZnCrO_4$ or $BaCrO_4$) 0.005–0.5% of cross-link delaying agent (such as tartaric, gluconic or citric acids or their salts); and 8–25% water]

194) Imperial Chemical Industries Ltd, BelgP 636956(1964) & CA **62**, 7580–81(1965) [A Blasting Explosive was prepd by mixing for 15 mins 88 parts granular AN & 12 ps of a 50% aq AN soln in a rotary blender equipped with paddles. The resulting mass was spread on sheets and dried at 45° for 14 hrs. The crystal cake was crushed, forced to pass a sieve (BS No 6) and then mixed with 9 parts of fuel oil for each 100 ps of sieved material]

195) H.M. Hurtado, SpanP 299960(1964) & CA **62**, 7581(1965) (Expls contg 20–80% urea & 80–20% AN are safe and easily handled)

196) K. Malkovsky & R. Mecir, Rudy **12**(9), 345–49(1964) & CA **62**, 8924(1965) (Description of Czech expl **Permon Extra**, which is suitable for large-diameter boreholes in surface blasting)

197) D. Sonntag, Ger(East)P 34615(1964) & CA **63**, 5440(1965) (Water-contg expls, such as consisting of TNT 15, NC 0.3, Ca or Mg stearate 0.5 and 85 parts AN soln emulsified at 85° with an inorganic nitrocompd)

197a) Institut National des Mines, BelgP 645374(1964) & CA **64**, 4856(1965) (Ion exchange safety expl: alkali metal nitrate & NH_4Cl in mole ratio of 1:1, 1:1.55 & 1:2 had a combustion pressure of 43, 52.5, 47.5 kg/sq cm and %N oxide production of 0.31, 0.06 & 0.02, respectively)

198) Ch.W. Kaufman, USP 3166450(1965) & CA **62**, 10286(1965) (Blasting Explosive prepd by stirring granular Na or K dichromate into a melt of AN maintained at 180° and then the mixt is cooled, ground and sensitized by tumble-mixing with No 2 Diesel fuel oil)

199) W.E. Gordon, USP 3166452(1965) & CA **62**, 8926(1965) [AN-hexamethylenetetramine complex, $(CH_2)_6N_4.2NH_4NO_3$, mp 122° and sol in org solvents can be prepd by dissolving 700 g hexamethylenetetramine and 800 g AN in 700 g water at 20° and then evaporating water at RT with a stream of air for 25 hrs to give a heavy slush contg crysts of above complex. The crysts were filtered and washed with anhydrous acetone. This complex is identical with an adduct described in USP 3166555 (1965). Its mixt with 4 parts AN prills is explosive]

200) W.E. Gordon, USP 3166555(1965) & CA **62**, 7580(1965) [AN-hexamethylenetetramine adduct of empirical formula $(CH_2)_6N_4.2NH_4NO_3$, prepd by crystn from either aq soln or melts, is a weak and insensitive explosive. When hammer-milled and boostered with a Tetryl pellet in a steel tube a deton velocity of 10400 ft/sec at density of 0.84 results. To obtain the necessary O balance for an expl formulation, 1 part adduct and 4.02 ps AN prills are blended, giving a cap sensitive stick expl with a deton vel of 13000 ft/sec in a 1.25 x 8-inch cartridge of density 0.95 and primed by a No 6 blasting cap] [See also USP 3166452(1965)]

201) J.R. Hradel, USP 3177102(1965) & CA **63**, 426(1965) (Solid expl compns produced by mixing 70–73% by wt AN in soln with NH_3 and water with 27–30% of a light metal, such as Al or Mg)

202) E.M Scott, Jr, USP 3178325(1965) & CA **62**, 15986(1965) (Metal nitrate expls contg mononitrated aromatic sensitizing agents. E.g. a) Na or Ca nitrate 84.4–84.7, Diesel oil 9.1–9.2 & granulated cork or sawdust 6.2–6.4%; b) Na nitrate 78.3, o-, m-, or p-MNT 15.5 & wood flour 6.2%; c) K nitrate 79.9, $PhNO_2$ 14.7 & sawdust 5.4%)

203) E.M. Scott Jr, USP 3180768(1965) & CA **63**, 426(1965) (Expl compns contg prilled uncoated AN & Ca nitrate with 4.5 to 8.5% of Diesel oil)

204) W.H. Rinkenbach & F.B. Wells, USP 3180773(1965) & CA **63**, 2844(1965) (Water-resistant blasting expls contg commercial fertilizer grade AN, ground meal as a waterproofing agent 2% in mixts such as consisting of AN 51.6, grained $NaNO_3$ 20.8, ZnO 0.3 & mineral oil 0.3%)

205) S. Hodgson, CanP 707268(1965) & CA **63**, 1650(1965) (Blasting expl prepd by mixing prilled AN with 5–6% by wt No 2 fuel oil and comminuting)

206) Monsanto Co, BritP 988095(1965) & CA **63**, 1650(1965) (Blasting expl prepd by mixing 6 parts of a light fuel oil with 94 ps high-density microprills and adding 5 ps of hydratable salt, such as nitrates of Mg, Al or Ca or Mg sulfate)

207) R.T. Dickerson, USP 3180844(1965) & CA **63**, 14103(1965) [Expls contg salts of monocarboxylic acid copolymers as thickeners for nonpolar solvents. For this 0.5% $CH_3:CH.CO_2H$ (I) and 99.5% styrene (II) were polymerized in a recirculating-coil reactor at 100 psi and 130° to give 40–50% copolymer (III) contg 0.9% I. To solns of 2–8% III in toluene, 0.43–3.3 equiv of the Li, Na, or K salts of dodecylphenol was added to give solns of viscosities as high as >100000 cp. The above thickened solvents may also be used in rocket fuels]

208) Th. Royer et al, USP 3184351(1965) & CA **63**, 5442(1965) [Safe, low-cost expls result from mixing a solid inorg oxidizer (such as AN, K or Na nitrate, K or Na chlorate or perchlorate) with a liquid chlorinated org sensitizer mixt (such as RCl_n with n = 1 to 10) mixed with a C_{1-6} hydrocarbon). For greater expl power, a nitroalkane (such as $MeNO_2$) may be introduced into the sensitizing mixt. For example 83.6 parts prilled AN, mixed with 16.4 pts liquid sensitizer (consisting of 63% $ClCH:Cl_2$, 20% $MeNO_2$ & 17% fuel oil) formed a satisfactory expl]

209) F.D. Patrick, USP 3188253(1965) & CA **63**, 5441(1965) [Expls contg metal particles

distributed uniformly thru them. These particles are prepd by spraying, as with a wire-fed metallizing gun or by flame-spraying of a light metal (such as Al, Mg or their alloys), onto coarse particles of either light metal (such as turnings, face scalpings, Grignard chips, etc) to obtain a product consisting of 10–90% coarse metal and 90–10% fine metal particles adhering to it. By loading these particles into a container, and by filling the interstices with an oxidizer, such as aqueous-ammoniacal AN soln, powerful, sensitive expls are obtd. Part of the AN may be replaced by another expl]

210) Jack Linsk, USP 3189495(1965) & CA **63**, 5441(1965) [AN compns contg N-aminoalkyl-morpholine combustion-catalyst system. E.g. AN (finely divided) 65–85, Prussian blue (sol or insol) 0.5–8.0, N-aminoalkylmorpholine (combustion catalyst having a C_{1-4} alkyl group) 0.5–8.0%, and binder 15–45%. The binder contains 15–45% polymeric material, such as poly (vinyl acetate) or poly(vinyl chloride) with the lower alkyl esters of NC and 65–85% oxygenated hydrocarbon plasticizers (such as lower alkylene glycols & their oxalates, maleates, diglycolates or nitrodiphenyl ethers, etc). E.g.: AN 78, Prussian Blue 3, N-aminoalkylmorpholine 3, binder 12, carbon black 3 & MgO 1%]

211) J.F. Wilson, USP 3190774(1965) & CA **63**, 5441(1965) [Free-flowing storage stable AN expl compns are provided by coating the AN with 1–3% of a two-component mixt consisting of 1–10% of the Na salts of methyl-(I) and dimethylnaphthalenesulfonic acids and 90–99% of silica-alumina (II) contg SiO_2 60–80, Al_2O_3 7–14 & Na_2O 3–10%. Thus 5 parts I and 95 parts II were dry-mixed with rapid agitation and the mixt sealed and stored for $\geqq$24hrs. When 3 pts of this mixt were tumbled 10mins with 97ps AN, the resulting mixt remained free-flowing during a prescribed caking test]

212) CnadInds, Ltd, BritP 1001216(1965) & CA **63**, 11242(1965) [Antistatic sensitized AN/Fuel Oil or AN/FO/Metal compns are obtd by incorporating a quarternary ammonium salt contg $\geq$ long-chain alkyl radical, a long-chain alkyl pyridinium salt of a polyoxyethylene nonyl (or dinonyl) phenol. Thus addn of 0.1% tallow trimethylammonium chloride (I) to a mixt of AN prills 94 & Diesel oil 6pts reduced static electricity in charges pneumatically loaded into simulated dry boreholes to 3% of the value obtd with the AN/FO mixt alone. With a metallized mixt (AN 87.1, Diesel oil 2.4 & Al 10%), addn of 0.5% I reduced the static chge to 8.4%, while addn of 0.5% polyoxyethylene nonylphenol reduced it to 4.2% of that of the AN/FO/Metal mixt alone]

213) J.D. Crisp, USP 3202556(1965) & CA **63**, 12964(1965) [Blasting expls contg gelled galactomannan gum are described. The gum is prepd by stirring 2 parts guar gum into 198pts water until the gum is uniformly dispersed and is free of lumps. Antimony oxide (0.2pts) is then stirred in, followed by 2pts MgO. Gels having a viscosity of over 2 million cp may thus be produced. A blasting agent may be obtd by mixing a water-sol inorg salt (such as AN, Na, K or Ca nitrate) $\geq$ 20%, a fuel, water and the components of the above described gels. HE's (such as TNT, PETN), oils, waxes, metals (such as Al, Mg or Al-Mg alloys) may also be incorporated]

214) J.W. Lawrence, USP 3203844(1965) & CA **63**, 14630(1965) (Expl compns contg sorbitan tetranitrate, an inorg oxidizing salt, and a fuel are described. One such compn consists of sorbitan tetranitrate 15, fine AN (100% thru a 50-mesh USStd screen) 35.7, $NaNO_3$(14-mesh) 29.4, $NaNO_3$(90% thru a 20-mesh and and 50–75% thru a 100-mesh screen) 11.0, corn flour 2.9, S 5.0 & guar flour 1.0%]

215) CanadInds, Ltd, BritP 1002671(1965)&CA **63**, 17791(1965) [An improved slurried, nonsegrating expl compn consists of water 5–25, fine Al or alloys 5–35, an inorg O-supplying salt (such as AN; Na, Ba, K or Ca nitrates) 20–80 a gel-forming polysaccharide (mannogalactans, e.g. guar gum or carob seed gum) 0.2–2.0, a metal chromate or dichromate 0.01–2.0, a particulate expl (TNT, PETN, etc) 3–50 and a cross-link delaying agent (metal oxalate or citrate; oxalic, tartaric, glyconic acids) 0.002–0.1%. The gelling agent may be mixed with a small amt of NG or NGc as a dispersant]

216) J.S. Logan & J.A. Zaslawsky, USP 3214307(1965) & CA **63**, 17791(1965) [Blasting gels which have low sensitivity and are substantially devoid of noxious fumes can be prepd by successively mixing into 40–70 parts AN(as a 50% aq soln), $K_2Cr_2O_7$ 0.5–1.5, guar gum 1.0–3.0, powd Al, Mg or their mixts 10–25 and water 20–45 pts. Alternatively, the guar gum thickener may be added stepwise to permit its hydration. The mixt forms a homogeneous gel on standing for 15 mins at RT]

217) F.B. Wells, USP 3216872(1965) & CA **64**, 1896(1966) [Blasting agent: fine TNT 20, mill AN 39.5, mill $NaNO_3$ 24.2, bagasse 1, "Hydroseal 3B" 3, ZnO 0.3 & water 16ps. Density 1.47, ballistic pendulum 9.5, sensitivity in 1.25-inch pipe 3gm PETN, rate of deton at d 1.47 in 2.0-in pipe 4380m/sec, and crater value (cubic ft earth moved/lb slurry) 10.2]

218) W.L. Schwoyer, USP 3222232(1965) & CA **64**, 4857(1966) [Expl slurry: Nitrostarch 28.2, AN 45.1, $NaNO_3$ 15.5, flake Al 1.9, No 5 fuel oil 0.3, Na carboxymethyl cellulose 0.7, guar gum 0.7, ZnO 0.8, anhyd $Na_2S_2O_3$ 1.2 & water 7.7% is prepd at 66°F and loaded into 1.25-in diam string-wound polyethylene (2–3 mil thick). This slurry has a deton vel in a 2.25-in diam bore hole of >5000m/sec]

219) J.F. Wilson, USP 3223185(1965) & CA **64**, 6153(1966) [The instability of AN (used in expls and fertilizers) in storage results from change in water content or changes in vol at the phase transition points. The improvement in stability is achieved by coating the AN particles with a compn consisting of a major amt of a clay (such as attapulgite, kaolin or diatomaceous earth), and a minor amt of an oil-sol alk-earth metal salt of petroleum sulfonic acids (such as Ca petroleum sulfonate)]

220) T. Seguiti, IndMinerari (Rome) **16**, 289–98(1965) & CA **64**, 7959(1966) [Uses & props of AN-oil expls: Max expl effect is obtd from mixts contg 5–8% combustible oil & prilled, spherical AN with 85% being less than 50 mesh & 70% less than 80 mesh. In prepg the mixt, 6% oil is added to AN and the mixt is digested for a min of 24 hrs. A mixt of AN 94.5 & No 2 fuel oil 5.5% requires at least 20% of AN particles to be less than 200 mesh. The min diam of the blast hole for these mixts is approx 20mm. A mixt of AN 60 & $NaNO_3$ 40% formulated with 6% oil has a 60% increase in expl power. Surface-active agents (0.5%) have possible benefits in increasing expl power. Increased power can be obtd by slurrying with water but stronger fuses are reqd for deton]

221) T. Seguiti, IndMineraria (Rome) **16**, 413–22(1965) & CA **64**, 7960(1966) [Underground uses of AN-oil expls: Pneumatic loading machines have made use of these expls possible in small diam holes. The mixt is transported with 2–4 kg/sq cm of air via a tube to the bottom of the hole, which is slowly filled as the tube is withdrawn. Static elec buildup among the AN granules is a problem (10000 volts is common). A min of toxic gases is produced. For 1kg AN-oil (5.7%) mixt, 20 liters of CO and 4 liters of NO_2 are liberated; whereas 1kg of Dynamite liberates 7 liters of CO & 4 liters of NO_2]

222) W.E. Gordon, BritP 1014071(1965) & CA **64**, 7962(1966) (Expl compn: AN 90, hexamethylene tetramine 9 & abietic acid 1% when melted at 145°, chilled and passed thru a 16-mesh sieve. The product when packed in a 1.5-in diam steel pipe, 12 in long, gave deton vels from 18700ft/sec at d 1.24gm/cc to 13400ft/sec at d 1.44gm/cc)

223) W. Dick & O.A. Gurton, BritP 1016462 (1966) & CA **64**, 9502(1966) (Safety expl: NG 8.5, NC 0.1, AN 10, $NaNO_3$ 36.5, NH_4Cl 33.4, Ca formate 10, guar gum 1 & Ca stearate 0.5%)

224) P. Lingens et al, GerP 1209034(1966) & CA **64**, 11022(1966) (Gelatinized expl: 1, 2 & 3% finely powdered polymethacrylate added to a 60/40 NG/Nitroglycol mixt having a viscosity of 11cp at 20° gave after 90 mins a completely transparent product having viscosities of 450, 2100 & 4250cp, resp)

225) T.D.S. Schott, JournInformCorpsGrasInd-Chim, HuilesInd, Acides Gras, Lipochim, Paris **1965**, 75–81; discussions 81–3 (in Fr);

& CA **64**, 12227(1966) (A review of application of fatty amines as lumping inhibitors for expls, fertilizers, etc and as flotation agents)

226) H. Dabrowski, Cement-Wapno-Gips **20**, 267–74(1965) (Polish) & CA **64**, 12453(1966) (New Polish mining expl **Iponit**)(See at the end of this Volume)

227) C.H. Grant, BelgP 651264(1965) & CA **64**, 14019(1966) (Water resistant expl: prepd by adding AN to 13 parts $HCONH_2$ to form a supersatd soln at 25°, guar gum 1, water satd with AN 12, $NaNO_3$ 10, finely divided Al 20, AN 20–25 parts, followed by 400cc of a mixt contg 200cc of AN in liq NH_3 and water to yield a very viscous liq of d 1.2gm/cc)

228) K. Ito, KôgyôKayakuKyôkaishi **26**(3) 140–41(1965) & CA **64**, 17344(1966) [Sensitivity of **Kuro**(Black) **Carlit**: In Japan coml expls contg NH_4ClO_4, ferrosilicon & wood meal are called Kuro (black) Carlit. Surface activated NH_4ClO_4 with surfactants or a mixt of NH_4ClO_4 & $Na_2Cr_2O_7.2H_2O$ has a higher rate of decompn and reactivity. Consequently, the deton rate & sensitivity to initiation of Carlit contg such NH_4ClO_4 are larger than those values of Carlit without such NH_4ClO_4. The effects of such NH_4ClO_4 on falling-hammer sensitivity are not significant as compared with those of ferrosilicon & glass powd] (See also Addnl Ref 319)

229) Dynamit-Nobel AG, FrP 1409082(1965) & CA **65**, 568(1966) [Fluid nitric esters, nitro aromatic compds or their mixts can be thickened and made plastic by addn of soluble polymers that contain neither nitro nor perchloro groups. The gelled compds are useful in the manuf of expls and proplnts. Polymers used include: poly(Me-methacrylate), poly(vinyl acetate), EtOAc-vinyl chloride, phenol-HCHO, poly-(vinylpyrrolidinone) and poly(vinylpyrrolidinone)-poly(vinylacetate)]

230) L. Deffet & J. Boucart, BelgP 655464 (1965) & CA **65**, 2059(1966) [Safety expls of the ion-exchange type (such as NH_4Cl/KNO_3 compns) are obtd by the addn of a halogenated org inhibitor (such as hexachloroethane, hexachlorocyclohexane, DDT, chlordan & iodoform). The reaction betw the inhibitor and the oxidizing component produces an inorg halide which inhibits the methane/air reaction. Addnl energy produced by this reaction augments the energy of the expln. The proportion of oxidizing agent is increased slightly over the conventional figure to allow for the inhibitor, which is usually added in the proportion 1.5%. For example, a typical compn is: NG 6.34, NGc 3.16, NH_4Cl 27.0, KNO_3 59.75, $C_6H_6Cl_6$ 1.50, stearate 1.50 & kieselguhr 0.75%]

231) Nobel-Bozel, BelgP 655471(1965) & CA **64**, 19311(1966) (A firedamp-proof Dynamite contg NG 33.5, NC 1.5, AN 20, NaCl 40 & guhr 5% has a deton rate of 2350m/sec in free air w/o confinement. When guhr is replaced by an equal amt of finely ground Zr silicate, the firedamp-proof quality is retained, except that the rate of deton, under the same conditions, is 5590m/sec)

232) PoudreriesRéunies de Belgique SA, BelgP 655796(1965) & CA **65**, 3662(1966) [Expl compns having high safety characteristics in the presence of firedamp and coal-dust, were provided by mixing 6–12% sensitizer (such as NG with NGc), 87–89% ion-exchange mixt of an oxidizer (such as Na or K nitrate) with Amm chloride, up to 0,5% waterproofing agent (such as Ca stearate) and ca 2.5% other additives. E.g: 70/30-NG/NGc mixt 9.5, $NaNO_3$ 54, NH_4Cl 33.5, Ca stearate 0.5 & other additives 2.5%; its unconfined deton vels in 30 and 90mm chges were 1665 and 1544m/sec, resp. A similar KNO_3-based compn had 1500 and 1340m/sec]

233) SERADEX, BelgP 658124(1965) & CA **64**, 4857(1966) (Safety expl: NG 11, carbohydrate 5.7 & AN/NaCl intercryst mix 83 parts. This Dynamite did not cause expln of atm when fired in CH_4 & coal powd, at 3% in a small mortar or at 9% for a suspended chge of 1.5kg)

234) R.M. Kaltenbach, BelgP 659035(1965) & CA **64**, 522(1966) (Low-density expl prepd by mixing spherical shape prills of AN (apparent d 0.75) with 5% gas oil could be detonated in 35- or 40-mm cartridges with a No 8 cap)

235) A. Berthmann, GerP 1203653(1965) & CA **64**, 1897(1966) [Permissible explosive: NG/Nitroglycol (40/60) 9, inert material (silicic acid/Zn stearate (0.55p/0.15p) 0.7, KNO_3 58.9 & NH_4Cl 31.4 parts. The deton propagation was 50 cm free lying on sand, and 0.15 cm in a paper tube]

236) A.B. Andrews et al, BelgP 667018(1965) & CA **65**, 3663(1966) [Improved seismic exploration charges which are not sensitive to No 8 cap and which allow for a reduction in chge wt for a given impulse are provided by mixt of 40–83(preferably at least 60) % particulate AN (consisting of large and small particle of sizes given in patent), 10–50(preferably 5–25) % 1600γ particulate Al, 0 to 5 (preferably 2–3) % hydrocarbons-liquid (preferred) or solid of mp <150°, and 1–10 (preferably 7–8) % mono- and dinitro- aromatic hydrocarbons. When a chge prepd from the above materials and contg 20% Al was fired under water, the wave received at the sensor had an initial pressure of ca 540kg/sq cm; this fell to 70kg/sq cm in ca 0.9 millisec and to 0 in slightly less than 2 millisec. A similar compn contg no Al produced a pressure of ca 420kg/sq cm which fell to ca 70 in 0,4 millisec and to 0 in 2.1 millisec]

237) F. Mezner & S. Uran, Rudarsko-Met-Zbornik **1965**(3–4), 327–37 (in Slovenian) & CA **65**, 6987(1966) (Two new expls, Nitrol & Kamex, introduced in Mezica Mine, Yugoslavia are discussed. **Nitrol** consists of approx 94.5% AN and ~5.5% fuel oil, sp gr 1 g/cc, while **Kamex** is a mixt of AN 14.4–67, $NaNO_3$ 0–38, NG proplnt 0–55, NC proplnt 0–38, TNT 0–16.6, water 8.8–14, and surfactants (not named) 0.8–1.2%; sp gr 1.50–1.75 g/cc. Nitrol is a powdery expl and may be used with/or wo cartridges. Kamex can be used in polyethylene bags or in the liquid state. Liquefied, it usually contains ca 14% water and can be pneumatically packed in boreholes)

238) Asahi Chem Industry Co, Ltd, BelgP 669299(1965) & CA **65**, 10419(1966) (An automatic and continuous process for mixing, gelatinizing, and kneading Dynamite components, developed in Japan, is described)

239) V.G. Gomes de Oliveira, MineriaMet (Mexico) No **35**, 93–132(1965) (Span) & CA **66**, 2947(1967) (Some general notions on explosives and the role they fulfil in the seismic process)

240) Milton Roth, "Evaluation of Inorganic Nitrates as Heat Test Standards", ICRPG Working Group on Analytical Chemistry, Round Robin No 24, PicArsn, Dover, NJ **AD626171**, Avail CFSTI $3.00 cy, 46pp(1965) & CA **66**, 106398p(1967)

241) J.D. Ferguson, USP 3235423(1966) & CA **64**, 12455(1966) [Stabilized aq slurry blasting compn: Ethylene glycol, $(CH_2OH)_2$, 18 was dissolved in water 15, pine oil 0.1 stirred in & guar gum 1 part and a trace of CH_3COOH added. This was followed by successive addn of grained AN 8.9 fine $NaNO_3$ 8, granular Al 14 & 20 mm single-base proplnt 35 parts to give an expl slurry of d 1.4 g/cc, pH 6.6, confined deton rate in 3-in column with a 20 gm PETN booster 5500 m/sec. The slurry was soft & fluid at –60°F after 36 hrs]

242) C.T. Butler, USP 3235424(1966) & CA **64**, 12455(1966) [High density blasting expl: AN 25.96, $NaNO_3$ 14.6, single-base proplnt (0.04-in diam x 0.1-in grains) 36.6, water 4.1, guar gum 0.34 & 1:1 ferrosilicon 18.4% provides a blasting compn of d 1.6 g/cc and deton rate 5240 m/sec in 2.5-in diam pipe (after 24 hrs under w at 300 psigm). Another mixt AN 11, $NaNO_3$ 23, proplnt 30, water 10, guar gum 1 & ferrosilicon 25% had a calcd d of 2.01 gm/cc but reqd a 500 gm Pentolite booster for deton in 3-in diam pipes]

243) F.B. Clemens & R.W. Lawrence, USP 3235425(1966) & CA **64**, 12456(1966) [Slurry-type blasting compns: AN 49.4, $NaNO_3$ 8.4, water 16, guar gum 0.7, propellant (NC contg 12–13.5% N) 25.0, $NaHCO_3$ 0.5% and 1% borax added at the end to cause cross-linking. The mixing sequence is important. The product of d 1.14 gm/cc is insensitive to a No 8 blasting cap, but is detonated by conventional booster chges such as PETN & RDX. This compn is used advantageously in water-containing boreholes]

244) L.W. Towle, USP 3236180(1966) & CA **64**, 14020(1966) (Blasting chge: grained AN 85, water 10, guar gum 0.4–0.8 & paraffin oil 4%. Such a slurry is useful in a wet borehole)

245) G.L. Griffith et al, USP 3238074(1966) & CA **64**, 15671(1966) (Extrudable expl: dry milled Nitrostarch 27.00, fine-grained AN 47.25, fine-grained $NaNO_3$ 10.75, flake Al 2.50, guar gum 2.50, No 5 oil 1.00, water 8.00 & ZnO 1.00%. This compn is stiff at RT, but is extrudable at 20psi thru 1.25"diam nozzles into cartridges 2 ft long and 2-in diam, made of heavy cardboard 0.082-in thick. The detonation rate of such a cartridge with a booster initiated with a detonator is 5.5km/sec)

246) E.A. Lawrence, USP 3239395(1966) & CA **64**, 17348(1966) [Nitromethane-amine expl: Preferably not more than 4–6% of an amine (such as diethylamine, aniline, tetraethylenepentamine or morpholine) is used, but up to 40% may be used with Nitromethane. A modified Trauzl Pb block expansion test and a modified Brinnell-type app were used to demonstrate the superiority of this expl over TNT]

247) J.F. Wilson, USP 3240641(1966) & CA **64**, 14019(1966) [AN-oil expl: AN prills 94 & various oils (meeting special specifications, including flash p 270–390°F) 6% showed oil losses of only 0.1–4.2% after blowing air at 76°F over a 0.75-in thick bed with a 20 sq in surface for 100 hrs]

248) N.E. Gehrig, USP 3242019(1966) & CA **64**, 19310(1966) (Solid emulsion blasting agent comprising nitric acid, nitrates & fuels. Division of USP 3164503. Disclosure is similar but claims are diff)

249) B.L. Atkins & R.N. Bashaw, USP 3242020(1966) & CA **64**, 17349(1966) (Gelled expl: a 50% aq soln of N-vinyl-2-pyrrolidinone was subjected to γ-ray irradiation at 0.16–0.32 megarad/hr. The resulting polymer was mixed with iso-propyl alcohol in an amt of 2% by wt of polymer to give a firm gel of low freezing point)

250) B.L. Atkins & R.N. Bashaw, USP 3242022(1966) & CA **64**, 17349(1966) [Gelled Nitroalkane expl: N-vinylpyrrolidinone 100, divinylbenzene 1.0 & 0.3 parts azodiisobutyronitrile are mixed and heated between RT & reflux temp for 0.5–1.0 hr. Cross-linked water-swellable poly (N-vinylpyrrolidinone) is separated, dried, and added to nitropropane until the resulting gel consists of 3% polymer & 97% nitropropane. This compn is useful in the gelation of fuels and the prepn of blasting compns]

251) African Explosives & Chemical Industries Ltd, BritP 1024558(1966) & CA **64**, 17350 (1966) (Prilled AN of high bulk d & high porosity, for blasting compns, is described)

252) Canadian Industries Ltd, BritP 1025637 (1966) & CA **64**, 19311(1966) [Blasting compn: 0.1p poly(oxyethylene)-nonylphenol dispersed in 4.9p diesel oil and mixed with 94.0ps AN prills, and 1.0p 7/93 Na carboxymethyl cellulose/water gel added does not develop high charges of static electricity during pneumatic loading into boreholes. Similar compns also contg 1–25% finely divided Al or Al alloy give similar results]

253) H. Eckhardt, ZErzbergbauMetallhüttenw **19**(4), 163–66(1966) & CA **65**, 563(1966) (Details of application in German mining of NG-free expls consisting of AN and C compds are described. The expls are not water-resistant and therefore cannot be used in water-filled drill holes)

254) R. Amberg, Ibid **19**(4), 169–72(1966) & CA **65**, 563(1966) (Introduction of AN-FO expls in cartridge form at the Gonzen iron ore mine, Sargans, to replace conventional blasting expls, resulted in improved output and reduced costs)

255) R.W. Van Dolah et al, "Explosion Hazards of Ammonium Nitrate under Fire Exposure", USBurMinesReptInvest No **6773**, 79pp(1966) & CA **65**, 564(1966) [The conditions under which AN and its systems may explode when subjected to intense fire exposure (never previously defined) were investigated at the USBurMines. The intrinsic sensitivity was studied by card-gap techniques. Fertilizer-grade AN was detonated, but at RT very large chges and strong expl donors were required. Prills coated

with 3% clay were less sensitive than those with 0.5%. Water in small amts increased the shock sensitivity of AN-fuel oil mixts. Transition to deton was obtd with AN intimately mixed with fuel oil, polyethylene, or paper. Hot AN was detonated by high-vel bullet impact, and AN-FO was sensitive to initiation by fragments from another AN-FO chge several diams away]

256) L.A. Fomenko, RussP 184675(1966) & CA **66**, 1998(1967) (Apparatus for continuous prepn of AN granular industrial explosives)

257) J.S. Wilson et al, USP 3237189(1966) & CA **66**, 2948-R(1967) (Water-resistant grease-like coating for blasting expls was prepd by mixing 562g of AN prills and 5.9g of powd Al oleate, followed by addn 24.1g No 2 Diesel oil and thorough mixing. Separately 234g AN was dissolved in 120g of water and 4g of guar gum was added slowly with continuous stirring. Then the coating and AN soln were mixed and simultaneously, 2ml of NH_4OH (28% NH_3) was added. The resulting product was a pourable slurry of d ca 1.2g/cc. A sample of slurry placed in water for prolonged period remained unchanged)

258) H.R. Nicholls & W.I. Duvall, USBurMines-ReptInvest No **6806**, 22pp(1966) & CA **66**, 3785(1967) (Effect of charge diameter on explosive performance) (A study was made of the effect of varying the diam of expl chges on the generation of propagation of strain waves, comparing strain amplitude, impulse, energy, and pulse shape. Charges of prilled AN-fuel oil and 45% Gelatin Dynamite showed a strong detonation rate–diameter dependency. Deton of these 2 expls was considered nonideal)

259) W.E. Gordon, USP 3247033(1966) & CA **65**, 566(1966) [Hexamethylenetetramine (HMeTeA)–AN expls consist of AN crysts (79–91.2 parts) encased by a fuel (20–8.3pts) consisting of HMeTeA, urea, dicyandiamide or their mixts. Up to 1% secondary fuels, such as powd C or dextrose may be added together with 0.3% abietic acid serving to modify the crystal structure]

260) R.B. Clay et al, USP 3249474(1966) & CA **65**, 566(1966) [AN expls contg coated Al, water and some other ingredients are described. Suitable coatings for Al powder are gilsonite, paraffin, Ca stearate, stearic acid, double-base proplnt, Armeen T & polyisobutylene. The compns contg coated Al possessed improved sensitivity to deton with densities up to 1.43. Generally the coatings were applied in amts of 2–45g to 4500g powd Al. Thus, a compn of AN 37.8, $NaNO_3$ 10.0, powd S 7.0, water 10.0, and an inhibitor (?) 0.2 part (to prevent excessive action of the powd Al & water) was mixed to form a sol. This was heated to 45° and the following added: powd Al 12, S 6, gilsonite 1.5 & guar gum 0.5 part to form a slurry explosive. After cooling the slurry to 25°, it was poured into 2–6-in diam cartridges, each being 6 diams long. Such mixts sustained deton in chges as small as 2.5 in diam, while mixts contg uncoated Al failed at 4 in]

261) R.B. Clay & L.L. Udy, USP 3249476(1966) & CA **65**, 2059(1966) and its USReissue 26804(1970) & CA **72**, 123587r(1970) [The crystn point of the AN soln used to prep AN-soln-based slurry type expl compns is lowered without substantial loss of energy in the final slurry and a more detonable blasting agent is provided by substituting a portion of 39–85% AN in the primary liquid with 2–40% of material which lowers the crystn point while increasing its solids content. Such materials are $NaNO_3$, $NaClO_4$, KNO_3, $Ca(NO_3)_2$ and urea. The lowered crystn point thus obtd allows for safer and less corrosive working conditions and provision of a safer and less sensitive blasting slurry. Conventional insol and low sol sensitizers, such as TNT, NC, S & powd Al may be included. Thus an aq compn which possesses satisfactory fluidity and provides a powerful slurry base contains AN 39, $NaNO_3$ 11, $Ca(NO_3)_2$ 37 & H_2O 13% has a crystn point of 23°, while 85/15, 80/20 & 75/25–AN/H_2O bases have crystn points of 77°, 59° & 43°, resp]

262) J.F.M. Craig et al, CanadP 729555(1966) & CA **65**, 3662(1966) [A thickened water-slurried expl compn which is resistant to separation during storage and to penetration of water is described. Thus AN 48.8, $NaNO_3$ 8.0, H_2O 11.5 & $ZnCrO_4$ 0.2g were mixed in a ribbon-type mixer and 10g Al powd was added. After a few mins TNT 20, tartaric

acid 0.01 & NGc (contg 1% guar gum) 0.5 g were added. When a homogenous mixt at 50–140°F was poured into a 20-inch high glass cylinder and stored 30 days at 90°F, no sepn took place. Similar mixts were obtd on substituting PETN, Comp B or Smokeless Proplnt for TNT; Ba nitrate for Na nitrate; 0.01–0.1% Na (or K) dichromate or 0.1–2.0% Ba chromate for 0.1–2.0% Zn chromate; and citric or gluconic acid for tartaric acid. The chromates reduced the sepn of the slurry and cross-linked with the gel-forming monogalactan (of guar gum) to give a more cohesive slurry resistant to water attack. Small amts of organic acids delayed this cross-linking long enough to easily package the slurry. A supplementary expt showed that only 0.01–0.1% Zn chromate was needed to prevent sepn of the compn when ordinary guar gum was replaced by self-cross-linking guar gum. No cross-linkage-delaying agent was then needed]

263) CanadIndsLtd, BritP 1031556(1966) & CA **65**, 3662(1966) [Static-resistant expl compns suitable for loading by an automatic eductor consist of 60–98% AN (or its mixt with Na nitrate), 2–20% oleaginous fuel (such as petroleum oils, greases or waxes and partially nitrated aromatics), 0.05–1.0% static-proofing agent (consisting of the reaction product of C_{1-5} secondary and tertiary aliphatic amines and C_{13-21} aliphatic acids); up to 0.5% acid acceptor (such as Ca, Na or Amm carbonates and Et_3N), and 1–25% (if desired) light metal or metalloid. Thus a compn contg AN 94, Diesel oil 5.65, static-proofing agent 0.25 and Ca carbonate 0.10%, when tumbled in a polyethylene-lined container for 10 mins developed a static chge with potential of only 6.2V. A similar compn contg no static-proofer developed when similarly tumbled, a 20V potential in 2.1 sec]

264) J. Dusek & R. Mecir, Rudy (Prague) **14**(3), 72–4(1966)(Czech) & CA **65**, 5292(1966)[Brief info is given on tests in Czechoslovakia of the mixt called **DAP-1** in overcast large diam (>75 mm) borehole blasts. DAP-1 is prepd by simultaneously adding 95% AN and 5% fuel oil to the borehole directly before blasting, without any special mixing. By tests with 4 120 kg chges it was found that the working ability of DAP-1 in rocks of medium and lower acoustical impedance is about the same as that of other Czechoslovakian industrial expls. The test concluded that DAP-1 is suitable for large-scale overcast blasting operations in rocks of the above mentioned props for dry, damp, and very wet boreholes and chambers. Its great advantage is a low sensitivity to mech incidence and its simple handling during charging]

265) K. Malkovsky & V. Pesata, Rudy (Prague), 141–44(1966) & CA **65**, 5293(1966) (Properties of two Czechoslovakian slurry expls: **Permon Extra 18** and **Permon Extra 9** are described but their compns are not given in CA. From the results obtd with the latter it was concluded that the application of slurry expl is advantageous esp in rocks of acoustic impedance >15. It can be also recommended for rocks of impedance 6–15, mainly for use in the bottom of drill holes, but application of these expls is not recommended for rocks of impedance <6)

266) R.B. Clay & L.L. Udy, USP 3249477 (1966) & CA **65**, 6992(1966) [Low-cost slurried blasting compns are described which can be prepd by dissolving AN (such as 54 parts) in 13.5 ps water (at 60°) and adding to it Na nitrate all that can dissolve (ca 9 ps). Granular Na nitrate (10 ps) which did not dissolve was then added, followed by 4 ps S, 4.5 ps fuel (such as powd gilsonite, asphalt or coal) & 3 ps starch. Finally 0.5 ps thickener (guar gum), was added to give a total of 98.5 parts. Addn of 2 ps paint-grade Al increased the sensitivity so that expl was self-propagating at 5°. Part of the water might be replaced by glycol, glycerol or low-aliphatic alcohol]

267) J.E. Clairborne, USP 3250652(1966) & CA **65**, 2057(1966) [Exothermic compns useful as proplnts, expls, and incendiaries can be prepd by mixing powd Zn 30, powd NH_4Cl 20, powd AN 47 & poly(vinylchloride) molding powd 3%, followed by heating to softening point and pressing into blocks. Addn of small amts of water to these mixts produces flame and hot gases]

268) H. Wanzke, Zement-Kalk-Gips **19**, 121–23 (1966) & CA **65**, 3659(1966) [The advantages of the use of ANC expls (where C stands for carbon) in large-diameter borehole blasting are discussed. The expts were carried out in a limestone quarry with a daily output of 18000 tons by blasting with boreholes 95mm in diam charged with loosely filled ANC expls and with 12–13% **Ammongelit 3** (no compn given in CA) as initial chge. The results showed a saving of 40% working time and 37.4% expls and igniters as well as 19% more loosened rock]

269) D. Sonntag, Ger(East)P 42947(1966) & CA **65**, 3662–63(1966) [Improvements in the production of expl mixts contg water-sol salts (such as AN or Na nitrate) with a tendency to cake and harden, can be achieved by incorporation of 1–5% of a mixt of high-mol-wt fatty alcs and fatty acid-glycerol esters. E.g: AN 60, $NaNO_3$ 10, DNT 15, NGc 10, sol NC 0.3, fatty alc/ester mixt 1.7, woodmeal 2 & red iron oxide 1%]

270) H. Mager, FrP 1430256(1966) & CA **65**, 6992(1966) [An improved AN expl of increased sensitivity that is non hygroscopic and pourable, can be prepd on the site by mixing 92–94% granular AN with 8–6% of a pasty suspension of fine Al, Si, Zn, Sb, Zr or Sb_2S_3 in hydrocarbon which may or may not contain O, S, or N and which is preferable to a light petroleum fraction (such as kerosene of fl p ca 72°). The paste contg 10–30% metal and 90–70% hydrocarbon forms a protective coating for AN granules. Pastes of Al suspended in ThioKol or org isocyanates makes the granules hydrophobic]

271) Dynamit-Nobel AG, NethP, Appl 6411854 (1966) & CA **65**, 6992(1966) (Liquid esters of nitric acid and aromatic nitro compds can be gelatinized with polymers of unsatd acids or unsatd alcohols and their derivs. The advantages of these polymers include increased safety of manipulation and increased rate of gelatinization. Thus a 60/40–NG/NGc soln was mixed with 3 wt % of finely powd polymethylacrylates and, after 1.5 hrs, had a viscosity of 4250 cp at 20° under shearing gradient of 15 sec^{-1})

272) G.L. Griffith et al, USP 3252843(1966) & CA **65**, 6993(1966) [Low deton-rate AN expl compns useful in seismic explorations contain AN (crushed to the size of 20-mesh) 62.5, DNT oil 5.5, NaCl 10 and perlite, sawdust, bagasse & pecan meal (in different proportions) 22%. For the four variations deton velocities were 5.3, 7.5, 6.7 & 6.8×10^3 ft/sec, resp. These low rates were retained during extended storage]

273) J.S. Brower & Th.W. Royer, USP 3255057 (1966) & CA **65**, 8559(1966) [AN expl compns sensitized with $MeNO_2$ or its mixt with a lower alc. A hydrogen-ion indicator was incorporated in the sensitized material to give it some color. Thus a sensitized expl mixt was colored to distinguish it from an unsensitized material. Suitable expl mixts contained 84 pts AN, 16 pts $MeNO_2$ (or its mixt with MeOH or iso-PrOH), which contained 0.0065% by wt of diethylaminoazobenzene. The red-colored mixt had a bulk d of 0.927 g/cc and was sensitive to No 8 cap]

274) J.E. Wyman & S.F. Bedell, USP 3255058 (1966) & CA **65**, 8660(1966) [Blasting compns consisting of 86–90% by wt AN, 3–11% o-dichlorobenzene(I) and 2–5% granular Al(II), are described. A sinergistic sensitizing effect of I & II is obtd by II may be replaced with ferric acetylacetonate. Up to 6% of fuel oil may be incorporated. The material charged into cardboard tubes of 2-in diam & 18-in long was placed to form a column 4.5-in long. Such column could not be detonated by one No 8 cap, but readily detonated with 5 No 8 caps in contact with one end. When thus initiated a deton rate ~8300 ft/sec was obtd]

275) H.F. Bluhm, USP 3256214(1966) & CA **65**, 9132(1966) [Prepn of explosive polyurethane resins, which are suitable for use as sensitive coatings for AN blasting compns and for prepn of foamed resin expls is described. The resins, which can be cured at RT, are prepd by reaction of an org polyisocyanate with a nitric acid partial ester of a polyhydroxy compd including a component contg $\geq$ OH groups reactive with the isocyanate. For example, equal parts by wt of anhydroenneaheptitol nitrate (13.77 wt %N as

nitric acid ester) and 2,4-toluene diisocyanate were blended and the blend cured at RT for 36 hrs to a glasslike mass. Such resin was impact sensitive]

276) W.G. Reynolds, USP 3259532(1966) & CA **65**, 8559(1966) (A novel expl compn suitable for blasting consists of a rod of Al sponge 1 inch diam x 1 ft long which is wrapped in Al foil so as to enclose one end and the circumference. A suspension of 20 wt % channel black in liquid O is poured into the open end of the foil container to achieve absorption of O by the sponged Al. After inserting an electric detonator, the open end is sealed with Al foil and the ensemble kept in liq O prior to insertion into a borehole in rock. Detonation shatters the rock evenly, producing considerable heat, but little smoke)

277) F Olstowski et al, USP 3260632(1966) & CA **65**, 10419(1966([AN expls contg expanded vermicular graphite are described and compared with those contg carbon black and flaked graphite. The methods for prepg expanded graphite are given. For making such expls, an aq slurry was prepd which contd (on a wet basis) AN 48, water 11, formamide 12, Al scrap 12, Na nitrate 14, expanded graphite 2 & guar gum 1%. The AN in the compn was comprised of ca 24 parts solid crushed priils and the remainder in soln]

278) C.W. Eilo, USP 3261732(1966) & CA **65**, 10419(1966) (The prepn is given of a novel aq slurry blasting agent which is stable when particulate Al and an AcOH–ZnO stabilizer are present. For its prepn are mixed: propellant 30.0 parts, water 18.0, pine oil 0.1 & ZnO 0.033. Then one-third of 21.9ps AN prills & 1.0 guar gum are added followed by the remaining AN and 13.5ps Na nitrate. Finally 14.0ps Al are added followed by 0.033ps AcOH)

279) S.F. Foster & J.F. Hamilton, FrP 1426427 (1966) & CA **65**, 10420(1966) [Description is given of a long, thin-walled, molded plastic cylinder contg a seismic expl (most of its length) and having a flexible internal watertight joint touching the expl) , and externally threaded male joint and stop-end in the wall at one extremity, and an internally threaded female joint at the other end. The external diam of the male joint being less than the internal diam of the female joint, may be attached to similar containers to form a column of expl in which the stop-ends of the male joints exert a compression force on the flexible watertight joints and the expl. Each container was loaded with 2.268kg of expl compn contg NG 49.0, NC 1.5, Na nitrate 36.0, pulp 7.0, maize flour 3.0, starch 2.0, indigo-stalk 0.5 & lime 1.0%. It is claimed that above described containers are superior to those made of paper, because they are waterproof and give a 15% increase in expl energy]

280) K. Stumpf, Glückauf(spelled Gluechauf in CA) **102**(15), 765–72(1966)(Ger) & CA **65**, 11319(1966) [An historical review of the development of expls, based on mixts of AN with org materials (preferably Diesel oil) is given. Their application, experiences and economical studies in comparison with traditional expls in different countries (with special consideration of Germany, are discussed]

281) Ch.A. Lebailiff, FrP 1429285(1966) & CA **65**, 12058(1966) [A compn, whose combustion velocity can be controlled is obtd by mixing a combustible (gelled hydrocarbons), an oxidizer (such as Amm nitrates, chlorates or perchlorates) and of an aq soln of a gum. Amines as stabilizers are added]

282) E. Schiele, Explosivstoffe **14**(3), 55–66 (1966) & CA **65**, 15138(1966) [The manuf of ANC (Ammonium Nitrate-Carbon), its expl properties, anduses are described]

283) Ch.V. Mullen, Jr, USP 3261105(1966) & CA **65**, 15143(1966) [A process for drying AN prills (or of other granular materials) which permits reduced moisture content to as low as 0.1%, is described. Moisture content of not lower than 0.2 to 0.5% has been achieved by using conventional two-stage process. Details of method are given in patent]

284) O. Osborn et al, USP 3264151(1966) & CA **65**, 12057(1966) [A high-d, water-resistant, readily deformable expl is provided by a mixt of AN, alkali metal nitrate (such as

$NaNO_3$), and halogenated hydrocarbon (such as CCl_4 thickened with metal soap), liquid carbonaceous fuel (such as fuel oil) and particulate metal (such as Al, Mg or their alloys). A compn consisting of nitrate soln 24.5, $NaNO_3$ prill 25.1, MeOH 4.9, gelled CCl_4 12.3, flake Al 16.6 & coarse Al 16.6% provided a blasting compn of d 1.40 g/cc and total energy 1.199 kcal/g. In comparison a 94:6-AN:fuel oil mixt showed d $\sim$0.86 & total energy 0.437]

285) J.F. Craig et al, BritP 1035290(1966) & CA **65**, 15144(1966) (Addn to BritP 1002671 & CA **63**, 17791c) [Slurried blasting compn stable in storage, consists of an aq slurry of inorg oxygen-donor salt(s), particulate org expl(s), gel-forming polysaccharides, finely divided Al, and small amts Na or K dichromates or Zn or Ba chromates. E.g: a slurry consisting of AN 37.5, TNT 20, guar gum 6, Al powder 17, water 16, ethyleneglycol 0.9 and 0.01% Zn chromate was suitable for blasting operation[(*Note:* The compn given in CA adds to 97.41%, instead of 100%)

286) Vincent Davies, BritP 1037567(1966) & CA **65**, 13450(1966) (Addn to BritP 990706) (See NethAppl 6601151, CA **62**, 3877b [Expl compns prepd by mixing particulate AN with a phenol and a HCHO source, carrying out the condensation and evapg the resulting water. The condensation could be accelerated by raising the temp and by addg $p\text{-}MeC_6H_4SO_3H$. Inhibitors (such as urea, MgO or $(NH_4)_2PO_4$), which also increase the stability, can be incorporated. For example, paraformaldehyde 2.1, phenol 6.7 & AN 91.2 parts were milled together so as to pass a BS 120 mesh sieve, allowed to stand at RT for 20 hrs, and then gently dried by agitation for 2 hrs in a rotary drier thru which air was passing at 70°. The product was broken by forcing thru a No 16 mesh sieve and finally had a particle size distribution such that the % passing thru BS 16, 30 & 60 screens were 100, 55 & 5, resp. Its pouring d was 0.7, and power by ballistic mortar was 78% that of Blasting Gelatin. Its deton vel 2000 m/sec was obtd when tested in steel pipe 1.5 inches diam]

287) D.P. Moore, USP 3265544(1966) & CA **65**, 16784(1966) [Dynamite-substitute expls contg 65–90% Li perchlorate & 5–25% rubber binder. Substitute for solid proplnt or HE: 75–90% Li perchlorate, 10–25% rubber and 3% sulfur (based on rubber). In making these compns, a dough-mixer was used and the resulting dough-like mass pressed in a mold and placed in a super-pressure steam-heated oven with IR lamps, where it was kept until all water was evaporated. Finally the product was vulcanized under pressure with heat]

288) G.L. Griffin, USP 3265778(1966) & CA **65**, 16784(1966) [A process is described whereby expl compns were extruded by forcing into and thru the nozzle by a screw. An example of an extrudable expl was prepd using dry milled NS (Nitrostarch) 27.00, fine grained AN 47.25, grained Na nitrate 10.75, flake Al 2.50, guar gum 2.50, No 5 oil 1.00, ZnO 1.00 & water 8.00%. In prepn of compn, the NS and mixed nitrates were thoroughly blended and the other ingredients added in order named above. The compn was stiff, and was easily extruded thru long, 1.25-inch diam nozzle, under pressure of 25 psi (using water as the lubricant at a pressure 35 psi) into tubular polyethylene casing 10-ft long and 2-inches diam]

289) AtlasChemInds,Inc NethPAppl 6514858 (1966) & CA **65**, 16784(1966) [Expls with good storage stability, insensitive to usual mech shocks as caused in transport, but sensitive to deton by percussion caps, were prepd from 30–80% by wt HNO_3 soln of K, Na or Amm nitrate, a C-based fuel not miscible with the HNO_3 soln (such as alkanes, waxes, mineral oils), an acid resistant stabilizer (such as colloidal Si dioxide, or polymers of acrylamide acrylic acid), and a gelatin catalyst (such as diethylenetriamine). For example, the expl consisted of 60% by wt of HNO_3 soln 30.0 parts, AN 47.0, $NaNO_3$ 10.0, diethylenetriamine 1.0, "Cyanogum 41" (a mixt of acrylamide polymers & copolymers) 2.0, sorbitan monopalmitate 2.0, mineral oil 2.0 and paraffin 6.0 ps by wt]

290) Herbert Bohm et al, Ger(East)P 47317 (1966) & CA **65**, 16785(1966) [A portable, spark-proof device is described which handles the charging of short boreholes with free-running explosive, such as consisting of 30 parts AN, mechanically mixed with 6 ps Diesel oil contg 1% "Marvelan" (which served as an

antistatic) (Compn of Marvelan is not given in above CA). This mixt was transferred to a reservoir to be blown from it by air pressure thru a plastic hose into boreholes]

291) M. Stammler & W.G. Schmidt, USP 3269879 (1966) & CA **65**, 18418(1966) (An impact-sensitive expl compn having a high-rate decompn at a low temp was provided by cocrystallizing AN or Amm perchlorate with certain salts such as those of the alkali and alk-earth metals, Cu, Ag, etc with acids nitric, permanganic, bromic, or iodic, in such propns that resultant cryst lattice contained 0.5–2.0% of such salts isomorphously substituted therein. Thus a cocrysd mixt contg 2% K periodate isomorphously substituted in Amm perchlorate had a 50% impact sensitivity at 27 cm with 2 kg wt vs 33 cm for RDX. The value for pure Amm perchlorate was 100 cm and for cocryst 98:2–Amm perchlorate:K permanganate was 75 cm)

292) DynamitNobelAG, NethPAppl 6516252 (1966) & CA **65**, 18418(1966) [A ternary mixt of NH_4Cl, alkali nitrates and alk-earth carbonates in an approximate molar propn of 3:5:1 gave a higher safety against firedamp explns and also a higher sp energy than the known binary inorg parts of expl mixts. A typical compn contained (by wt) NG 5.22, NGc 3.48, KNO_3 (78% <0.1 mm) 41.28, NH_4Cl (65% <0.1 mm) 36.40 & $CaCO_3$ (85% <0.1 mm) 13.62%. A cartridge of this compn placed in silica sheath, showed, on heating in an oven at 200°, a rise in temp of 20°. A cartridge with similar compn, but contg a binary inorg part (KNO_3 59.71 & NH_4Cl 31.59) showed a rise in temp of 600°]

293) L. L'Heureux & G. Towell, CanadP 738331(1966) & CA **65**, 18418(1966) [Expl compns (resistant to charges of static electricity) comprising (by wt) 60–98% AN, 2–20% oleaginous fuel (liq petroleum hydrocarbon, low-melting petr grease or wax or partially nitrated derivs of benz, toluene, xylene or naphthalene), 0.3–1.0% water, 0.02–0.5% wetting agent (ethylene oxide-nonylphenol adduct having 3–9 ethylene oxide units, quarternary cationic, amine cationic, anionic or nonionic), and 0.01–2.0% of at least one thickening additive (gel-forming mannongalactan, water-swelling cellulose ether, Na silicate, colloidal SiO_2 or Al_2O_3). Powd light metal (Al, Mg or alloys) 1–25% may be optionally added as sensitizers. Above expl compns may safely be pneumatically loaded into boreholes]

294) R.S. Egly, USP 3275485(1966) & CA **65**, 19924(1966) [AN slurries of high d and expl power and low water content are prepd by mixing, in any desired manner, a water-sol nitro-substituted alkanol sensitizer of the general configuration $R(R')C(NO_2)CH_2OH$ where R & R' are lower-alkyls or hydroxyalkyls (R also may be H) 10–50 (preferably 15–25) parts, water 2–10 (preferably 3–6) and AN 40–88 (preferably 69–82) parts. Optional conventional additives (such as C-black, ground coal, morpholine, lower alkanols, Al S, ferrosilicon, waxes and thickener-guar gum) may also be incorporated. Such slurries can safely be prepd in the field by adding the nitroalkanol-water soln to the AN perviously placed in a borehole]

295) R.B. Clay & W.N. Bryan, USP 3282752 (1966) & CA **66**, 4575d(1967) [Substantially O-balanced AN-based expl slurries were provided by substituting up to 30% of AN with another inorg nitrate such as $NaNO_3$ and addn of S in such amt that the $NaNO_3$/S ratio was well below the stoichiometric ratio and was within (preferably) limits 1–1.6. Thus a mixt contg ca 56.6% AN, 15.4 water, 12.9 $NaNO_3$, 0.1 stabilizer, 1.5 guar gum, 2.1 particulate Al, 9.0 sulfur ($NaNO_3$/S ratio = 1.4), and 2.4% powd Gilsonite (as fuel), provided a slurry which detonated at 60° (d 1.34) in a 3-in diam chge (but not in 2.5-in diam) and at 40° (d 1.37) in a 5-in chge (and not in a 4-in chge). It meant that the mixt was safe to handle at mixing temps and sufficiently sensitive for use in normal blasting operations]

296) M.A. Cook et al, USP 3282753(1966) & CA **66**, 4576m(1967) [Slurry blasting agents contg nonexplosive liquid fuel are provided by suspending a solid oxidant (such as $NaClO_3$ or $NaClO_4$ which contains some AN) 70–90 parts, in a medium such as an aq soln of water-sol nonexplosive liq fuel [such as lower aliphatic alcs, polyhydric alcs & ketones or an aq dispersion of nonexpls, nonwater-sol

liquid fuel (such as kerosene or fuel oil)] 5–20 ps and water 1–10 ps. A fine nonwater-sol fuel (such as C or Al) may also be present. Thus, addn of a premix of $NaClO_3$ 41.5, AN prills 31.125 & fine AN 10.375 to a dispersion of No 2 fuel oil 4.5 & tall oil 4.5 in a soln of Na dodecylbenzenesulfonate 0.05 in water 9.0 and subsequent addn of stabilizer (Na_2CO_3) 1.0 part provided a stable blasting slurry, d 1.55, which detonated in 5-in diam chge satisfactorily with 160 g Pentolite booster}

297) N.E. Gehrig, USP 3282754(1966) & CA **66**, 67511y(1967) [The disclosure is the same as in USP 3164503 (CA **62**, 6334e), but claims are different]

298) J.D. Ferguson & R.B. Hopler Jr, USP 3288658(1966) & CA **66**, 47941k(1967) [Aerated slurry-type expls of which a typical compn consists of water 22.5, ground single-base smokeless proplnt 25.0, AN prills 31.0, $NaNO_3$ (granulated) 19.4, ethyleneglycol 1.5 & guar gum 0.6%. Guar gum serves as a hydratable cross-linking thickener. The compn which is insensitive to No 8 cap (but can be detonated by a Tetryl or Comp B booster), may be sensitized by the inclusion of TNT, Tetryl or finely divided Mg or Al. A suspension of guar gum 1.5 in ethyleneglycol 3.0 & AcOH 0.3 part was formed by mixing at RT for 3 min and added to the above basic ingredient mixture. As the slurry is pumped into a container or a borehole, variable amts of $NaHCO_3$ (up to 0.14%) suspension in glycol are added into the expl stream and air is pumped under pressure into this stream at the same time. This produces an aerated expl slurry]

299) P.L. Swisstack, USP 3288661(1966) & CA **66**, 39497e(1967) {An aerated aqueous expl compn of improved sensitivity and regulated d and expl strength was provided by passing compressed air into a slurry contg 20–75% oxidizer (such as nitrates and perchlorates of NH_3, K, Na, Ca, Ba, etc), 4–35% water, 4–60% sensitizer (such as TNT, Pentolite, PETN, smokeless proplnt, RDX or Tetryl),Al and Mg, 0.5–2.0% thickener (such as guar gum, CM-cellulose & starch), 0.6–5.0% surfactant [such as diethanolamides of coconut & lauric acids, triethanolamine salts, alkylpoly(oxyethylene)s, arylalkylsulfonates & mixed dimethylamine oxides]. Presence of the surfactant allows for retention of up to 40% of the air used in aeration in the form of tiny bubbles. Example: smokeless proplnt 25, water 22.5, AN 7.5 & bis(2-hydroxyethyl) cocoamine oxide 1.0 were mixed together, hydratable guar gum 1.0 in glycol 2.0 parts and the material stirred 3–4 mins (to allow cross-linking), while air was bubbled thru. This produced a stable slurry, d 0.93. A slurry prepd similarly but without surfactant had a d of 1.31}

300) J.J. Yancik, USP 3291659(1966) & CA **66**, 57492d(1967) [Flat disks or flakes are prepd by passing betw rollers with a gap of 3–30 mils AN prills (8–20 mesh). These materials exhibit improved fuel oil absorption and deton properties when incorporated in blasting expls. Thus 94% (by wt) flakes were mixed with 6% No 2 fuel oil for 2 mins to give a free-flowing material with deton vel 15000 ft/sec when initiated with No 6 blasting cap]

301) G Hoberstorfer & R.O.R. Oscarsson, SwedP 202595(1966) & CA **66**, 57493e(1967) (Blasting expl: AN 100 & mineral oil 5–6 parts are conveyed by compressed air into a borehole or a plastic-foil hose)

302) Erich Lechner, BergJuettenmaennMonatsh (Austria) **111**(10), 479–84(1966) & CA **66**, 67474p(1967) [A review of the practical experiences gained in the USA during the last 10 years with AN-Diesel oil mixts in open-pit working (strip-mining). The preferred way is an installation employing mech or pneumatic methods]

303) W.E. Gordon, USP 3294601(1966) & CA **66**, 67519g(1967) [Continuation-in-part of USP 3247033; CA **65**, 566e] [A wet blasting agent prepd by mixing 61–8% (by wt) AN, 5.4–6.5% hexamethylenetetramine (I), 4.4–18.7% water; lesser amts of addl fuel (such as wood flour), oxidizer (such as $NaNO_3$ or $K_2Cr_2O_7$) and thickeners (such as okra gum and cellulose ether gum)]

304) I.N. Chernev, Fiz-TekhnProblRazrab (erroneously spelled Razarb in CA) Polez-Iskop, AkadNaukSibOtdel(Russ) **1966**(5), 119–21 & CA **66**, 77900p(1967) (**Ammonit** expls can be hazardous in underground mining because of scattering of undetonated AN par-

ticles. The completeness of deton and means for preventing scattering of unreacted particles, were tested in a bomb with chge of expl, surrounded by water, aq solns of salts, solid NaCl & KCl, ice, sand, powd gypsum, etc. After deton the bomb was cooled, contents extracted with water and the resulting soln titrated for AN. The most complete deton, with the least scattering of unreacted particles, occurred when the expl was surrounded by a 20-mm layer of water. Powd gypsum and solid NaCl or KCl were less effective, while sand was ineffective)

305) A.P. Glazkova & V.K. Bobolev, VzryvnoyeDelo **1966**(60/17), 5–20(Russ) & CA **67**, 13426b (1967) (Investigation of influence of NaCl on the burning characteristics of AN expls intended for use in gaseous coal mines, showed that the most stable burning and the highest burning velocity of mixts were observed when they contd 10% NaCl)

306) B.N. Kukib, Ibid, **1966**(60/17), 63–6 (Russ) & CA **67**, 13427c(1967) [The influence of mixing inert salts (such as Amm sulfate, Amm chloride or NaCl) with NG on its expl characteristics was studied. For detn of critical diam of deton, mixts of NG with salts, made into conical shape cartridges of various diams and with 2–4° angle of taper, were detonated with No 8 blasting cap. A lowering of NG content from 15 to 7% did not show any noticeable effect on the critical diameter of cartridge, but further lowering to 5% did sharply increase the crit diam of deton. The phys props of the inert fillers (density, mp, bp & heat capacity) did not show any noticeable effect on the crit diam of deton which depended only upon the nominal density of NG. For testing the mixts for sympathetic deton, the cartridges were hung up in the air one above the other, with various distances (gaps) between the upper end of "donor" cartridge (called in CA "active" cartridge, which consisted of 100 g 85/15-AN/NG mixt) and lower end of "acceptor" (called in CA "passive" cartridge which consisted of 100 g 85/15 inert salt/NG mixt). The No 8 blasting cap was placed at the bottom of donor. The gaps were found to be 25, 30 & 37.5 cm for mixts contg NH_4Cl, $(NH_4)_2SO_4$, and NaCl, resp]

307) B.N. Kukib, VzryvnoyeDelo **1966**(60/17), 83–96(Russ) & CA **67**, 13422z(1967) [Studies of sensitization of inert salts (such as NaCl, Amm sulfate and Amm chloride) used in safety expls by HE's (such as NG, DEGcDN, RDX & PETN) were described. The crit diam of deton and the max gap distance for sympathetic deton in the test expl were used as criteria of sensitization. Reliable sensitization by NG on NaCl occurred at a level of 8% NG, when the particle size of NaCl was 0.2–0.3 mm. At sizes <50 micron ca 20% was required. Sensitization by 1:1 mixts of NG & DEGcDN was similar to pure NG. Pure DEGcDN at a level 10% NaCl did not detonate even at a chge diam of 32 mm. The use of gelatinizing agents, such as NC or poly (Me methacrylate) decreased the sensitizing capacity of NG. RDX & PETN were effective sensitizers when measured by crit diam, but are less effective than NG with respect to gap test. Combinations of solid and liquid expls were recommended]

308) K.K. Andreev & P.D. Dien, Ibid **1966** (60/17), 107–18(Russ) & CA **67**, 33709k (1967) [The combustibility of expl mixts contg AN or Amm perchlorate was studied in a manometric bomb using both high (1.5–1.8) and low (1.0–1.1) density chges. The combustibility was judged by the wt of igniter (composed of 1:1–NC:AN, fine-grained) required for stable burning of 4-g chges of the expl. High-d chges of AN contg 4% fuel oil did not burn with 2-g igniters, while AN contg 21% TNT burned with 1-g igniter. The combustibility and the rate of burning of AN/FO mixts increased greatly with the addn of NaCl and only 0.8-g igniter was required. With AN/TNT mixts addn of NaCl reduced the igniter from 1.0 to 0.8 g. For low-d AN/FO chges the wt of igniter was higher than for corresponding high-d chges. No difference was found betw high and low-d AN/FO chges contg TNT or NaCl. Substitution of TNEtBz (Trinitroethylbenzene) for TNT resulted in significant decrease in combustibility. Mixts of Amm perchlorate contg 4% fuel oil required 0.4-g igniters for high-d chges, but only 0.2-g for low-d. Unlike NaCl, addn of SiO_2 had no effect upon the combustibility of compns]

309) N.S. Bakharevich et al, Ibid, **1966**(60/17), 126–36 & CA **67**, 13423y (1967) [Gallery tests of Russian permissible expls **Ammonits** and **Pobedits** (described later in this Volume) showed that CH_4-air mixts were more susceptible to ignition when the expl charges were distributed in a row and not in cluster. The incendivity was decreased by incorporating 8–10% NaF or KF. They were superior to conventional cooling agents, such as NaCl, bicarbonate or oxalate. Expls contg NaF exhibited complete detonation and showed superior water-resistance]

310) N.S. Bakharevich et al', Ibid **1966**(60/17), 143–56 & CA **67**, 13424z (1967) [Studies of Russian industrial expls **Metanits** and **Miporits** (described later in this Volume) showed that both expls are safe for use in gaseous and dusty coal mines]

311) B.Ya. Svetlov, Ibid, **1966**(60/17), 160–73 (Russ) & CA **67**, 5579c (1967) [Blasting efficiency (as measured by heat of expln) in dry AN/TNT mixts was max for the stoichiometric ratio 79/21 as TNT content increased, the evolved heat amt decreased reaching min for pure TNT. In the water-filled mixts, the heat of expln increased with TNT content attaining a max for 70–100% TNT. Similar relations were found in efficiency measured by brisance effects]

312) A.V. Fadeev, Ibid, **1966**(60/17), 173–88 (Russ) & CA **67**, 55798e (1967) [Critical evaluation of blasting efficiency of AN-TNT-water expls marketed in Russia since 1953 brought the following conclusions: 1) With water-filled expls a higher concn of energy in a volume-unit is obtd 2) The strongest expls are mixts with a near zero oxygen balance 3) Addn of powdered metals strengthen the expls but not as much as was reported in the literature 4) The behawior of water-filled expls at temps near fr p of AN solns is unclear and should be investigated 5) water-filled expls are economical and efficient for very hard rocks and 6) The most efficient economically is a combined construction of the blasting chge in which the powerful and expensive expl is charged only to the lower part of the borehole]

313) V.A. Usachev, Ibid, **1966**(60/17), 197–201 & CA **67**, 23700a (1967) [Studies of effect of specific surface of AN on expl props of **Igdanits** (described later in this Volume) were conducted with mixts prepd from various fractions of granulated AN. Their specific surfaces were calcd by the formula developed by Usachev]

314) J. Kaemmler & J. Ruhmannseder, Rudy (Prague), **14** 139–41 (1966) (Czech) & CA **67**, 45698h (1967) (Development of AN-fuel expls **Dekammon 1** and **Dekammon 2** at the potash mines of East Germany. Their compns are not given in CA's)

315) S. Tat & E. Hindoreanu, RevMinelor (Bucharest, Romania), **17**(11), 490–92 (1966) (Roman) & CA **67**, 55801a (1967) [Two permissible expls were developed: **A-2** and **A-3**, which contd TNT (7.00 & 7.00), MNT (0.75 & 0), glycerol (0 & 0.75), NaCl (29.25 & 31.00), AN (62.00 & 60.00) and wood flour (1.00 & 1.25). The A-2 sample of 100 g ignited an atm contg 8–10% CH_4 compared with 200 g of the NG contg Romanian safety expls **AGC, AGS** & **AG** (compns are not given); however A-3, in which the MNT was eliminated, was equiv to the latter three. The work capacity of both A-2 & A-3 was 180 cc which was higher than that of AGC and almost equal to that of AGS. The sensitivity of sympathetic deton differed from chge to chge and this was attributed to differing agglomeration of the AN particles. It was found that optimal results can be achieved by reducing the humidity and by adding sensitizers such as $BaSO_4$ or MnO_2. To reduce the humidity, 0.5% CM-cellulose was added to A-3. The exptl detn of salt granulation showed that fine AN provided better expl props for A-2 & A-3, giving higher values for work capacity and of sensitivity to sympathetic deton. Fine granulation of NaCl had an opposite effect]

316) G.P. Demiduk, SciTechAerospaceRept 4(20), 4095 (1966); NASA Accession **N66-34302**, Rept No **AD633490**. Avail CFSTI $3.00, 28pp (1966); CA **66**, 97072y (1967) (Mechanism of explosion and other properties of Russian explosives **Ammonits** and **Igdanits**)

317) S.A. d'Explosifs et de Produits Chimiques (SAEPC) NethPAppl 6608580 (1966) & CA **66**,

97094g(1967) [AN in the presence of a surfactant could be crystallized into needles, plates, fibers or dendrites with a sp surface of >1000 sq.cm/g and mixts of such AN with up to 20% combustible materials (such as fuel oil) gave expls with increased sensitivity, power, and deton velocity. Provided the sp surface was high enough, various inerts like NaCl or $BaSO_4$ could be added to make the mixt suitable for use in mines. The names of surfacts used are not given in CA]

317a) ICI of A & NZ, Ltd, BritP 1033480(1966) & CA **66**, 77934c(1967) [Fluid or plastic compns prepd from a nitric ester adsorbed onto a finely divided carbonaceous fuel, an inorg oxidizer, water, and (optionally) a fluidity-controlling agent, such that the nitric ester remains adsorbed when water is added, and sufficiently viscous so that there is no segregation of the components. For example, 1000g NG was mixed in a Schraeder mixer with 420g coarse woodmeal and after NG had been homogeneously adsorbed, 2280g coarse AN and 2200g fine AN were added, followed by 2200g $NaNO_3$ and 500g woodmeal homogenized with an equal amt of paraffin wax. The 1200g of water was added to the mixer and stirring continued for 5 mins. Finally 5g guar gum predispersed in 100g glycol was added and stirred for 3 mins. The product was a pourable slurry]

318) Y. Wakazono, KôgyôKayakuKyôkaishi **27**(5), 288–94(1966) & CA **66**, 117534f (1967) (See also CA **61**, p9347) (Effects of primer, booster, and loading methods on the deton velocity of AN-FO blasting agents were studied and described)

318a) L. Vajda, BanyaszLapok **99**, 594–98 (1966) & CA **68**, 14614u(1968) (in Hung) [The customary compn of AN-gas oil mixts, the optimal particle-size distribution of the AN, protection against atm moisture, and the prepn of expl mixts on location are discussed. In Hungary, good results were obtd with **Akremit** in boreholes and chambers. The problem of 3-month storage of Akremit and its application at −10° were solved] (Compare it with Amer Akremite described in Vol 1 of Encycl, p A119-L)

319) K. Sakamoto & H. Yoshitomi, Ibid **27**(6), 377–81(1966) & CA **66**, 117543h(1967) [Large-scale underwater gap tests were carried out using as a donor 1 kg Ammonia Dynamite and as acceptors 150-g charges of Ammonia Dynamite, Gelignite, Blasting Gelatin, PETN, Guncotton, **Kuro (Black) Carlit** (NH_4ClO_4 66–70, FeSi 8–12, woodmeal 15–19, and heavy oil 3–7%), Permissible Powdery Dynamite, Black Powder, TNT and AN-FO. Both donor and acceptor were contained in a waterproof wrapper] (See also AddnlRef 228)

319a) S.B. Wright, USP 3296041(1967) & CA **66**, 87227p(1967) (Continuation-in-part of USP 3173817; CA **62**, 12968g) [Granules of org nitrate and nitramine HE, of high bulk d, were prepd by mixing an aq suspension of binder (polyamide, polyisobutylene or chloro- or chloro-fluoro polyethylene) with water slurry of the expl. Upon addn of coagulant (alum, AcOH, acetone, or gelatin), the binder and expl were granulated. Solvent (ester, ketone, alc, or hydrocarbon) in which the binder is sol was added. Or, in the presence of protective colloid, granules were formed by mixing dissolved binder with a water slurry of expl. In either case the vol solvent was evapd, excess water removed by filtration and granules dried. Granule size was regulated by varying the duration of agitation of multi-phase system and by selecting different protective colloids. Thus, 20–50g poly(hexamethylenediamide) (I), dissolved at 65–75° in 500g BuOH was added to 470g diaminotrinitrobenzene (II) in 2000g water (at 70–5°), while stirring at 500–600rpm. The resulting emulsion was agitated to give the desired granule size, BuOH evapd, the slurry cooled to 50°, and filtered to give a product contg 2–10% I and 98–2% II; bulk d 0.6–0.7]

320) T. Quadflieg & G.A. Wetterholm, USP 3296042(1967) & CA **66**, 57494f(1967) (Plastic expl: PETN 21, AN 63.5, starch 8.2 & water 7.3% give an expl compn which can be readily detonated, but has a low sensitivity to mech impact)

321) J.D. Chrisp, USP 3297502(1967) & CA **66**, 67513a(1967) [Aqueous expl compn contg metallic fuel (Al particles)

coated with 1.67 lb oleic acid and 6.67lb "Corvus" oil per 100 lb Al. K dichromate was used as cross-linking agent. One of the final compns consisted of AN 32.7, water 17.3, $NaNO_3$ (premixed with guar gum) 15.0, TNT 25.0 & precoated Al 10.0%. Another formulation contained 75% of premixed soln (contg AN 78.9, water 4.2, $NaNO_3$ 16.0 & "Jaguar 100" guar gum 0.9 part), 20% pelletol & 5.0% precoated Al]

322) G.I. Griffith, USP 3300348(1967) & CA **66**, 67518f (1967) [Expl conitrates, such as prepd by conitration with HNO_3-H_2SO_4 of corn starch mixed with pentaerythritol, to form NS/PETN, had relatively low impact sensitivities, higher initiation sensitivities and deton rates than mechanical mixts of individual nitrates. Other substances (such as dipentaerythritol, sorbitol, erythritol, trimethylolpropane, and neopentyl alc) can be conitrated with corn starch, but PE was found most suitable, especially to form the conitrate contg 20% PETN & 80% NS (of 13.1%N)]

323) F.C. McKoy & E.C. Knowles, USP 3301721(1967) & CA **66**, 77931z(1967) [Hydrazine gel compns contg fuel metals (such as Al, Mg, Be, etc), microdimensional fibers (such as glass, asbestos, cellulose or synthetic) and thickeners, such as polyacrylamide, Na CM-cellulose, hydroxyethylcellulose, poly(vinyl)alc, & poly(Me methacrylate). Thus a soft gel was prepd. It contained hydrazine 65.20, polyacrylamide 0.67, microglass fibers 0.80 & Al powder 33.33% A similarly made gel comprised of hydrazine 58.3, polyacrylamide 0.7, cellulose fibers 1.0 & Al 40.0%]

324) H.H. Mohaupt, USP 3301722(1967) & CA **66**, 87228q(1967) (High-d, pelleted expls suitable for wet boreholes were prepd from 38–73% AN, 25–60% insol HE sensitizer, and 1–3.5% hydrocarbon binder and waterproofing agent. For example AN 68, TNT 30 & paraffin wax 2% were mixed and extruded under high pressure to give 0.25-inch diam pellets, d 1.4, which detonated at 3500m/sec when packed into 4-inch diam pipe)

325) J.D. Chrisp, USP 3301723(1967) & CA **67**, 92485w(1967) [Galaktomannan gum gels can be used in blasting expls, such as prepd by mixing 49 wt% of a 60 wt% AN soln at 120–80° F for 2–3 mins with a premix contg 25% granular $NaNO_3$ and 0.75% finely ground guar gum flour; 25% pelleted or flaked TNT was added and mixed for 1–2 mins, followed by Zr acetate (in quantity sufficient to provide 0.7 millimole Zr per g guar gum) and 0.25% MgO. The resulting thickened mass was extruded at 90–100°F into polyethylene tube and sealed. The compn was not cap sensitive but could be initiated by 100g RDX to detonate at rate of 4500m/sec]

326) G.L. Griffith & F.B. Wells, USP 3303072 (1967) & CA **66**, 67514b(1967) [Low-d, O-deficient nitrate-based blasting expl compns having pouring d $\sim$0.5 and ca twice expl power of O-balanced expls (as shown by cratering values and operational results but not by conventional test methods) are described. One formulation was prepd by mixing 81.5% AN (alone or contg a small amt of dendritic inorg nitrates), 1.5% mineral oil, 1.0% diatomaceous earth & 16.0% bagasse (or other low-d fuel such as shredded corn stalks or shredded synthetic plastics). This mixt had a deton rate of 1923m/sec/3-in diam, unconfined at d 0.42 and 2457m/sec (1.5-in confined at d 0.505). Ballistic pendulum value 10.7]

327) W.L. Schwoyer, USP 3303074(1967) & CA **66**, 77935d(1967) (Continuation-in-part of USP 3222232;CA **64**, 4857a) [Aqueous expl slurries contg sulfur and esp those contg 15% Al or other light metal were stabilized against decompn and resultant loss of effectiveness at elevated temps by incorporating into such slurries of (preferably $\gtrsim$ 0.5%) an oxide, hydroxide or salt (such as halide, nitrate or sulfate of an ionizable metal of Groups I–VIII. One of the formulations contd AN prills 47.5, $NaNO_3$ (grained) 20.0, Al 20.0, water 12.0, guar gum 0.5, $Na_2S_2O_3$ 3.0, and either Pb nitrate or acetate 1.5 parts. When placed in direct sunlight (av daily temp 85°F), it showed no expansion after 5 days]

328) R.B. Clay et al, USP 3303738(1967) & CA **66**, 106753a(1967) [Description of equipment for mixing and pumping of slurry expls, such as listed in USP 2930695 (CA **55**, 846h) and CanadP605314 (CA **55**, 9879f)]

329) R.J. Anderson & J.F. Short, USP 3304211 (1967) & CA **66**, 106754b(1967) [Free-running granular or pelletized, water-resistant expl prepd by making AN and $NaNO_3$ particles adhere to and around TNT prills, which are held together by a binder consisting of $CaSO_4$ (ligna) and poly(ethylene oxide). The resulting expl was suitable for use in boreholes contg water]

330) G. Nanquin & E.A. Demelenne, RevIndMiner **49**(1), 57–64(1967) (Fr) & CA **66**, 97066z (1967) [Improved Belg permissible expls with ion exchange were prepd using an equiv mixt of KNO_3 & NH_4Cl as the basis. A study was made in order to improve some phys props and the toxic behavior of blasting fumes. An ion exchange expl, corresponding to KNO_3+NH_4Cl when compared with Belg expl **Charbrite 418** (which contained the equiv mech mixt of the two salts) proved to be more brisant and powerful and of higher deton velocity (1550m/sec vs 1380, when detonated in free air at density not indicated in CA). Analysis of the air after shot-firing in a steel gallery or in rock showed that the gas produced by ion-exchange expl was less toxic than that produced by Charbrite and the amt of rock broken was 20% higher]

331) H. Logan & R.F. Knott, USP 3306789 (1967) & CA **66**, 117566t(1967) [Mixts of nitric acid with inorg nitrates and fuels in O-balanced proportions gave solid, water-resistant expls of high strength, and sensitive to No 6 blasting cap.. For example, a soln 30.2 parts by wt of AN dissolved in 41.8ps 98% HNO_3 was cooled to 23° and seeded with $NH_4NO_3/2HNO_3$ crysts, then mixed with 28ps DNT at 60°. The slurry was fluid momentarily to allow mixing and then set at 18° in 5–20 sec. When cooled to 5° it became a hard homogeneous solid. Its deton velocity was 2715m/sec when a 2000g 2.5-inch diam sample was initiated by 160g Pentolite at density which is not given]

332) Ch.H. Grant, USP 3307986(1967) & CA **66**, 117567u(1967) [High-energy water-resistant AN-alkali metal nitrate expls contg Al of particular size distribution and processes for their production were described. A 1000-lb batch of expl was prepd as follows: Sufficient AN was added to ~13 parts (by wt) formamide to form a satd soln at 25°C. About 1 part guar gum was added with agitation to disperse the gum, followed by ~12ps water (satd at 60°F with 17.5–20ps AN, based on total batch). After agitating the mixt to provide homogeneous mass, it was allowed to stand for $\geq$15 mins to allow the gum to swell and $NaNO_3$ 10, fine grain Al 20 and fine grain AN 20–5ps were added successively with agitation. Finally a mixt of 200ml satd soln of AN in liq ammonia dilutd with an equal vol water, was blended to cross-link the gum. The expl props were evaluated by underground firing and the results are listed in the patent and in CA]

333) L.F. Audrieth, L.H. Eriksen & W.R. Tomlinson, Jr, USP 3309251(1967) & CA **66**, 117568v(1967) (A liq expl consisting of Nitromethane and 1–20 wt% of an amine selected from the group ethylenediamine, butylamine, and morpholine. Up to 10% of a diluent such as glycerol, EtOH, and Et Cellosolve could be added. For example, the explosive, prepd by simply mixing 95 parts $MeNO_2$ with 5ps ethylenediamine had a deton rate of 6000m/sec, low sensitivity to bullet impact and to friction, a moderate tendency to separate on standing and brisance comparable to TNT)

334) S.D. Lewis, USP 3316132(1967) & CA **67**, 45711g(1967) (Stable expl compn: An intimate mixt of sorbitol hexanitrate 90.73 & Na salicylate 9.27%, both materials having a max particle size not more than 595μ, after 52 days storage at RT lost 0.19% by wt. Another sample of sorbitol hexanitrate without stabilizer, similarly tested, lost 18% of its wt)

335) J.D. Hopper & F.B. Wells, USP 3317361 (1967) & CA **67**, 23727q(1967) [Flexible plasticized expl: A water-resistant, cap-sensitive, flexible expl having improved thermal stability, high impact & friction resistance, high power, safe to handle and useful for metal forming and demolition is provided by a mixt of RDX or HMX 63, NC (12.1–12.5% N) 6–8, tributylacetylcitrate 25–30, pigment (1p lampblack & 8p chrome yellow) 0.3–0.8, and DPhA about 0.4% added. The mixt is prepd by kneading or pptn and then formed into sheets, strips, blocks, filaments or other shapes by rolling, extruding or compression molding.

Strips of d 1.4730 gm/cc had a rate of deton of 7044m/sec, ballistic mortar value = TNT, cap sensitivity No 8 cap and vac stab value (5 gm/40 hrs) at 110°, 2.85 ml gas evolved]

336) H.R.Fee & R.W. Lawrence, USP 3318740 (1967) & CA **67**, 55844s (1967) [An aqueous slurry-type blasting agent is provided by a mixt of 12–20% water, 1–18 (preferably 5–14%) sensitizer such as hexamethylenetetramine mononitrate (I), dinitrate or a mixt of the two, 45–65% AN, 5–20% $NaNO_3$ and 0.2–5.0 (preferably 0.5–2.0%) guar gum thickener in cross-linked form. A supplemental sensitizer such as fine Al, Mg-Al or C (or hydrocarbon oil) may be used, especially if less than 5% of I is present. The particle size of the inorg oxidizer is not critical. Thus a slurry contg water 17.8, I 13.6, AN prills 52.0, $NaNO_3$ (granular) 15.2 & cross-linked guar gum 1.4% had sp gr 1.23g/cc, pH 5.0 and deton vel 3850m/sec. A similar mixt contg only 5% I had sp gr 1.26, pH 4.9 & deton vel 2400m/sec; when the $NaNO_3$ in this compn was replaced with AN, the props became sp gr 1.20, pH 4.9 & deton vel 3650m/sec]

337) J.A. Arbie, USP 3321344(1967) & CA **67**, 66206m (1967) [Aq slurry-type expl compns contg AN and an expl sensitizer (such as TNT, DNT or smokeless proplnt) have attained commercial acceptance. These compns have been improved by inclusion of gelling agents (such as the cross-linked, partly hydrolyzed polyacrylamide described in USP 3097120 & CA **59**, 8540c). This invention provides expl slurries which remain pourable even when subjected to temps below 0°F for prolonged periods. Fluidity of the gels can be maintained by inclusion of urea and NaCl. Five formulations are listed in the patent and in CA]

338) J.L. Griffith et al, USP 3322066(1967) & CA **67**, 55839u (1967) (Self-destructive expl cartridge suitable for use in seismic, underwater exploration, after immersed in water, is described. The principle involved is the use of an inert, water-activated dissipator which undergoes a volume change by dissolving on coming in contact with water. A typical cast charge had the following compn: Pentolite 89.5, Na carbonate 5.0, Na tartrate 5.0 & Na CM-cellulose 0.5%. After the protective water-sol coating is dissolved, the water reacts with the gas-forming chemicals in the expl and the evolved carbon dioxide causes the insol particles to form a slurry which is forced out of the cartridge)

339) G.T. Colegrove, USP 3326733(1967) & CA **67**, 75047n (1967) [Gelled expl compns contg a Xanthomonas hydrophilic colloid mixed with aq soln of inorganic nitrate (such as AN) and borax are described. Xanthomonas is a hydrophilic, exocellular, high-mol-wt colloid prepd by culture fermentation by *X campestris* (or by other bacteria) of a medium contg 2.5% glucose, an org N source (such as corn steep liquor), K_2HPO_4, appropriate trace elements, and borax. Thus, 2g Xanthomonas was dissolved with agitation in 198g of a 60.6% aq AN soln at 50°C and 0.2g borax was then added. A gel formed immediately and solidified on standing. Addn of 1 g borax to a soln of 0.8g Xanthomonas in 60% aq AN soln immediately gave a firm, rubbery expl]

340) Th. E. Slykhouse, USP 3326734 (1967) & CA **67**, 66207n (1967) [Inorg nitrate-based expls of high energy, good water-resistance and sensitivity to impact initiation (which set to a hard mass of d > 1) were provided by mixts such as consisting of AN prills 63, a satd aq soln of Mg nitrate 27, a setting agent (such as MgO) 3.6, & metal fuel (such as chopped Al flakes) 10 parts. The resulting slurry set on standing to a very hard expl casting]

341) J.D. Ferguson, USP 3328217(1967) & CA **67**, 83557u (1967) (Aqueous slurry blasting expls which contain a combination of 10–25% propellant grains with 2–15% DNT, 8–25% water, 30–75% inorg oxidizer, and 0.2–5.0% thickener, such as guar gum)

342) M.A. Cook et al, USP 3331717(1967) & CA **67**, 83556t (1967) [Inorg oxidizer blasting slurries contg a proplnt (obtd as surplus from military stocks) together with water, auxiliary sensitizers and thickening agents]

343) H.E. Mager, FrP Addn 88583(1967) to FrP 1430256 (CA **65**, p6992); CA **67**, 83564u (1967) [Granular AN (intended for use in expl

compns) was coated with powd metal (such as Al) suspended in an aq soln of cryst org material (such as glucose, trioxane, sorbitol, PA or MeNH_4 nitrate). The grains could also be made hydrophobic by use of water-sol or compatible substances (such as urea-H_2CO resins, cresol, polyvinyl, etc). Thus 10g powd urea-H_2CO condensate, 80g glucose and 10g Al powd were added to a soln of 40g trioxane in 100g water at 50°, the mass mixed with 1 kg granular AN and dried at 50° to yield Al-coated AN granules]

344) W.L.K. Schwoyer & Th.P. Dowling, USP 3332349(1967) & CA **67**, 83563t(1967) [An improved presplitting blasting compn (for establishing a shear plane in a rock formation before final blasting) and a simple means of application are described. The expl consisted of preferably fine-grained NS (Nitrostarch) 6–25, AN 50–75, Na nitrate 0–10, metal fuel 0–25, carbonaceous fuels (including bulking fuels, such as bagasse) 0–40, antacid (such as ZnO, $CaCO_3$, Al_2O_3) 0–5 and inert bulking agent (such as expanded vermiculite, silica aerosol, fumed silica or silica flour) 0–10%. Thus, a mixt of NS 20, AN 60, $NaNO_3$ 5, bagasse 5, sawdust 2, nut meal 2, oat hulls 2, vermiculite 2, Al_2O_3 1.65, ZnO 0.10 and No 5 fuel oil 0.25% was loaded into cartridges, which were held end-to-end in a continuous column of a desired length by couplers, which also held initiating detonating cord line near or against the cartridge. Proportions of ingredients and diams of cartridges are varied to produce the desired loading densities 0.15–1.5 lb/ft of drill hole required for the specific presplitting operation]

345) A.G. Keenan et al, USP 3336171(1967) & CA **67**, 101577g(1967) [AN expl compns contg metal-excess chloride combustion catalysts are described. In expts, varying amts of metal additive were used with AN. First, a curve showed the max temp obtd with AN alone, then with 0.1% Cr or other metal (a slight improvement), then with NaCl alone (a slightly greater improvement), finally the synergistic improvements obtd by use of compds of Cr, Cu, Ag & Ni with NaCl in a Cl^-/metal atom ratio of 37:1. To show the effect of increasing metal concn at a const chloride content, a run was made at 185.5° in which varying amts of $AgNO_3$ were added to a chge consisting of 7g AN & 570mg NaCl. Optimum results were obtd with ~0.16 mole % of $AgNO_3$]

346) A.N. Barron et al, USP 3336981(1967) & CA **67**, 92494y(1967) [A stable gel of fuming or concd nitric acid can be prepd by the addn of 1–5% by wt of a polymer of N-vinyl-2-pyrrolidone cross-linked in the vinyl group. The prepn of polymer was described in USP 2810716 (CA **52**, 13321). The gelled acid may be used in the fracturing of reservoir strata by pumping the acid and explosive fuel reacting hypergolically with it, separately into the well and forcing them into the formation to react there as described in USP 3266572 (CA **65**, 16755) and USP 3336982 (CA **67**, 101670g). Thus 3.12 parts by wt of N-vinyl-2-pyrrolidone, polymerized by cross-linking with 0.7% by wt of divinylether of ethyleneglycol with catalyst α,α'-azobisisobutyronitrile, was mixed with 156 parts red (or white) fuming nitric acid. A gel stable for >1 year formed in 15 mins]

347) P.E. Woodward et al, USP 3336982(1967) & CA **67**, 101670g(1967) [Fracturing of reservoir strata by injection of fuel (e.g: mixt of crude oil & kerosene) that will not form a hypergolic mixt with nitric acid in the well, is described. This was followed by a solid and/or inert liquid plug (e.g: water, fuming or concd nitric acid), another plug of inert liquid or unreactive fuel, a reactive fuel (e.g: an aromatic or olefinic hydrocarbon, $PhNH_3$, or furfuryl alc), and enough inert liquid to force the previously injected liquids into the formation, where they will mix and explode]

348) G.L. Griffith, USP 3337380(1967) & CA **67**, 92488z(1967) [Inorg nitrate-based NS (Nitrostarch) slurries which are impact-resistant but much more sensitive to initiating agents than are corresponding conventionally prepd slurries, are described. Expl compns of this patent were prepd by adding 2–30% NS to AN slurry previously prepd by cooling aq AN soln saturated at 225–250°F (>80% strong) to 125°F, with rapid agitation to obtain slurry of fine-grained crysts. Other inorg nitrates could be mixed with NS, as well as fuels, thickening agents, gelling agents and antacids. For example, a satd

soln contg 498 lb AN and 102 lb water at 214°F, was passed to Holo-Flite processor and a fine-grained slurry contg 16.19% water at 104°F obtd. To 64 parts of this was added a mixt contg wet NS 22.4, milled $NaNO_3$ 10, ZnO 0.8, bituminous coal 1.5, No 5 fuel oil 0.5 and guar gum 0.8 part. The expl slurry obtd after mixing had the following characteristics: d 1.44, ballistic pendulum value 10.10, sensitivity (in a 1.5-inch pipe) 10g Pentolite, and deton rate 4987m/sec]

349) J. Calzia, FrP 1467939(1967) & CA **67**, 92489a(1967) (Methods of coating expls with polyacrylonitrile to improve their expl characteristics, are described)

350) W.T. Jones & J. Wilby, BritP 1082641 (1967) & CA **67**, 101578h(1967) [Expls suitable for forming into sheets useful for demolishing metal articles or for other purposes, were provided by a mixt of 87–7% particulate HE (such as RDX, HMX, PETN or Tetryl) and 13–3% of viscoelastic binder (such as polyisobutylene or polypropylene having a mol wt of $\geq$5000 and contg 7–15% of its wt of a fluoropolymer). Some plasticizer (such as Et oleate, Bu phthalate, etc) can be incorporated. Method of mixing and of rolling into sheets was described]

351) J.J. Minnick, USP 3338165(1967) & CA **67**, 118754k(1967) [Expls prepd from NMe (Nitromethane) sensitized to initiation by incorporation of spherical resin balloons of finely divided air-entrapped materials. The balloons of diam 2 to 360 microns (such as described in CA **59**, 9731a) and bulk d 0.3 g/cc were made from a polymerized thermosetting resin, either urea-formaldehyde or phenol-formaldehyde. Suitable amts of balloons to sensitize NMe were in the range 1.5 to 10%. Fuels, oxidizers and thickeners could be incorporated]

352) SERADEX, FrP 1479383(1967) & CA **67**, 118759r(1967) [Expl safe for use in presence of natural gas, such as comprising AN (porosity 0.035cc/g) 55.4, NG-NGc mixt 10, NC 0.3, fine dry peat 4.7 & NaCl (sp surface 700 sq.cm/g) 29.6%, had power (by ballistic mortar test) 66% PA. In the French firing test, using a small mortar (1-mm plate) in an atm contg 9% natural gas, only one ignition in 20 firings was observed. In similar tests with the same compn (except that porosity of AN was 0.070cc/g and power of 66% PA), ten ignitions occurred during 20 firings]

353) I. Toth & L. Nemeth, HungP 153585 (1967) & CA **67**, 75039m(1967) [Moisture-proof expls, consisting of AN 63.5, carbazide-HCHO foam 8.5, TNT 9 parts contained also 10 parts of additives which absorbed radiation energy of expln located in the same main region as that of CH_4. Compn of additives was SiO_2 60, P_2O_5 25 & B_2O_3 15 mole %]

354) J.P. Konrat, FrP 1469198(1967) & CA **67**, 75040e, (1967) (Method of coating various HE's to make them more easily and safely compacted without forming agglomerates is described. As coating materials polymeric organic compds, wax or mixt of materials in in a suitable solvent were proposed. Any desired additives, such as graphite, finely divided metals or their oxides, etc may be incorporated)

355) J.F.M. Craig et al, GerP 1244030(1967) & CA **67**, 83560q(1967); Addn to GerP 1232506(See CanadP 729555, CA **65**, 3662b) (Mixts of inorg nitrates with TNT & Al powd slurried in water and stabilized with a self-cross-linked mannogalactan and Zn chromate are described. These mixts remained stable in storage and did not darken, as for example mixt made from AN 37.5, $NaNO_3$ 8.0, TNT 20.0, Al powd 17.0, glycol 0.9, water 16.0 parts to which was added mannogalactan 0.6 & $ZnCrO_4$ 0.1% showed only slight darkening after 33 days storage at 30°, while similar mixt contg no stabilizer darkened with decompn)

356) Karl Porr, GerP 1244031(1967) & CA **67**, 75041f(1967) (Fluid expl mixts suitable for use in charging app, because they have low impact sensitivity, are described. For example, a mixt which contd TNT 12.0, heating oil 4.3, finely divided carbon 0.45, Al powd 2.0 & AN grains 81.25% had impact sensitivity of 35cm with wt of falling hammer 10kg. Its deton velocity was 4130m/sec and power by Trauzl Test 380ml)

357) Dynamit Nobel AG, NethAppl 6611745 (1967) & CA **67**, 55838t(1967) [Four powd expls safe for CH_4-coal dust-air mixts were obtd by

mixing NG, NGc, AN, KNO_3 & $CaCO_3$ in the following percentages by wt: **A** – 5.1, 3.4, 41.1, 46.6 & 3.8; **B** – 5.1, 3.4, 40.6, 45.9 & 5.0; **C** – 5.1, 3.4, 39.4, 44.7 & 7.4; and **D** – 5.1, 3.4, 36.5, 41.4 & 13.6 (D was described in NethAppl 6516262 & CA **65**, 18418g). Expl D had the lowest specific energy. Cartridges made of A, B & C and surrounded by diatomaceous earth showed no salt conversion after being heated in an oven at 130°]

357a) Dynamit Nobel AG, NethAppl 6612512 (1967) & CA **67**, 55837s (1967) [Plastic and gelatinous safety expls prepd from sensitizing components and inorg salts such as ternary mixts of AN, alkali nitrate and alk-earth metal carbonate. E.g: NG 17.4, NGc 11.6, NC 1.0, $NaNO_3$ 27.5, AN 28.8, $CaCO_3$ 10.7 & 50% soln of $Ca(NO_3)_2$ 3.0%. Its props: d 1.58g/cc, excess oxygen 0.85% & gap test 10cm]

358) P.M.L. Chatel de Raguet de Brancion et al, GerP 1240453 (1967) & CA **67**, 55843-R (1967) [Double-salt expl stable toward firedamp: NaCl (passing a 0.5mm sieve but not a 0.315mm sieve) 100 is mixed with fine AN 137 & water 2.5 parts. The mixt is allowed to stand 72 hrs, dried at 70°, and broken into a size which does not pass a 0.5mm sieve. This double-salt 83, glycerol mononitrate 11, NC 0.3 & a hydrocarbon 5.7% forms an expl safe against a dust cloud in an atm of 3% firedamp, safe in a small mortar and 3% firedamp, and safe in a free firing test of 1500gm in a 9% firedamp atm. Power in ballistic mortar test = 54.1, lead block test 41.2]

359) C.J. Kelly & R.F. Knott, USP 3341154 (1969) & CA **70**, 116783g (1969) [Pourable, aqueous slurry compns are described, composed of an inorganic oxidizing salt, water, a thickener, dispersant and a fuel. Their sensitivities could be improved by including in the mixts a chlorinated org compd (such as CCl_4, $CHCl_4$, $C_2H_3Cl_3$, etc) and a material that stops the migration of any air or gas bubbles that have been introduced or occluded into the compn. Suitable materials for this purpose were water-sol salt of lignosulfonic acid or water-sol polyflavonoids. Preferred thickeners were malogalactans (such as guar gum or carob beans). As a dispersant ethylene glycol was used, as a stabilizer NH_4 lignosulfonate, and as a fuel Diesel oil. For example, the expl compn contg granular AN 79.7, Diesel oil 2.4, CCl_4 8.0, water 7.0, guar gum 0.5, ethylene glycol 1.0, and NH_4 lignosulfonate 1.4% was an expl which detonated completely when loaded in a 4-inch diam cartridge and initiated by 160g Pentolite]

360) M.A. Cook & H.E. Farnam, Jr, USP 3341382 (1967) & CA **67**, 118758q (1967) (Boosters which insure complete expln and propagation the full length of slender columns of expls, are described. Suitable boosters for inducing reliable propagation of AN-fuel oil mixts, AN-TNT-water mixts, AN-Al-TNT-water mixts and AN-Al-water mixts were found to be cast or pressed 50/50-Pentolite, pressed Tetryl, pressed RDX and pressed RDX (wax)

361) J.E. Bergwerk, USP 3341383 (1967) & CA **68**, 51491x (1968) [A pourable nonsettling, water-resistant expl slurry or pumpable gels based on AN, an expl sensitizer (TNT, NS, etc), and contg partially hydrolyzed polyacrylamide are described. A further improvement could be obtd by using a suspending agent comprising a mixt of the hydrolyzed polyacrylamide with cross-linked galactomannan gum]

362) D.S. Partridge, USP 3342132 (1967) & CA **67**, 110246a (1967) (Packaged blasting expls which exerted, when detonated, a combination of shattering and heaving effects. For example, a tube of extruded polyethylene film 5 inch in diam, closed at one end, was filled with 25 lbs of a mixt of AN with 6% fuel oil, and the open end sealed. The bulk d of mixt was ~1.0g/cc. This charged, sealed tube was placed inside an extruded polyethylene tube 7 inches in diam, followed by introduction of 25 lbs of aq slurry compn in the larger tube so that the slurry completely surrounded the smaller tube contg AN-FO. Then the external tube was sealed for convenience in handling. The aq slurry compn contained AN 42, $NaNO_3$ 14, hexamethylenetetramine 4, coarse-grained Al 15, paint-grade Al flake 1, water 23 & guar gum 0.6 parts)

363) L. Guricza, HungP 153950(1967) & CA **67**, 110247b(1967) [Industrial expls of low bulk density were prepd by mixing usual AN expls with 2–15% by wt of plastic foam (such as urea-formaldehyde, polystyrene or polyurethane of 10–50kg/m^3 bulk density). The plastic foam of 0.2mm dispersity was homogenized with the expl components preground in an edge runner. Thus, a mixt of 78.5% AN, 14.5 TNT, 5 urea-formaldehyde plastic foam, and 2% PETN was homogenized to have product of d 0.7]

364) A. Desmarais, USP 3344004(1967) & CA **67**, 110245z(1967) [Expl gel compns with improved stability were prepd by addn of a cross-linked carboxylate contg a gelling agent to a conventional compn contg an oxidizer salt, sensitizer, water, and fumaric acid to maintain the pH at 3–5. The gelling agents were the cellulose ether, acrylic acid polymers, or acrylamide copolymers and were cross-linked with $(AcO)_2AlOH$, $(AcO)_2CrOH$, $Al(OH)_3$, or $Fe(OH)_3$. Thus a slurry of AN 50, $NaNO_3$ 15 & water 18 parts was heated to 120°F and treated with 0.8ps carboxymethyl cellulose for 5 mins. After mixing 8ps Al flakes into the slurry, a dry blend of $(AcO)_2AlOH$ 0.16, sugar 5, & fumaric acid 0.1 part was stirred into the slurry for 3mins and after cooling the slurry to RT, it was allowed to stand. An elastic, cohesive, 3-dimensional nonpourable gel with pH 4.9 was obtd, which was stable for several weeks and performed satisfactorily as an explosive]

365) J. Bronstein & G.L. Griffith, USP 3344005(1967) & CA **67**, 118760j(1967) [PETN-based mixts useful as expl sensitizers were described in which the impact sensitivity of the PETN was substantially reduced (without affecting its deton sensitivity) by incorporating 5–55% trimethylolethane (TMeEt). Mixts contg <30% TMeEt were granular solids, while those contg $\geq$ 30% were extrudable. A dry stick powder contg 70:30–PETN/TMeEt 13.5, grained AN 79.7, $NaNO_3$ 2.0, bagasse 3.0, Al flake 1.4, No 5 oil 0.3 & ZnO 0.1% was 60% less sensitive to impact than a similar compn contg PETN 13.5% without TMeEt. Both samples were in 1-cm cartridges and fired with No 1 caps]

366) G.L. Griffith, USP 3344743(1967) & CA **67**, 118752h(1967) [Blasting expl slurries, prepd at the site of work, which are detonable in 2-inch diam or smaller columns are described. For example, formaldehyde (prepd at the site) was mixed in proportion 15:11 with MeOH and dissolved in a 73% aq AN soln contg 12% NaOH. After concentrating the mixture at 60–70° a heavy slurry was obtd. Its compn was AN 65, hexamine 4, HCHO 2, MeHO 2, MeOH 3 & water 23 parts. Finally 0.5 part guar gum was added and the slurry pumped into a borehole, where it was detonated by means of a 1-lb Pentolite booster]

367) G.L. Griffith, USP 3345224(1967) & CA **67**, 118761k(1967) [Dry appearing expl compns contg porous materials capable of releasing absorbed liquid at safe extrusion pressures (such as $\leq$50psi) when transferred into plastic tubes are provided by incorporation into the expl mixt of liquid-contg porous particles, various materials, preferably <6 mesh. These included expanded vermiculite, silica aerogels, alumina, silica, or bentolite and org compds (also serving as fuels) which are preferred for use with oil as they release water in the mixer. They included polyurethane, nylon, cellulose, poly(vinylchloride), and rubber sponges in a sufficient amt to contain 0.5–20% compressible liquid. At extrusion pressures such particles yield their absorbed liquid to transform the mixt into a semi-solid or thixotropic compn which easily extrudes and then reabsorbs the liquid to give a solid self-supporting column. Thus, a mixt contg wet NS (Nitrostarch) (23% water) 17.0, AN (ground prills) 53.0, $NaNO_3$ (granular) 15.0, ZnO 0.6, Al (flaked) 1.75, $Na_2S_2O_3$ 0.3, No 5 oil 0.25, pecan meal 3.0, CM-cellulose 1.3, guar gum 0.3, and No 2 vermiculite (contg 2.25 times its wt of water) 7.0 parts were blended in a paddle mixer, passed thru a No 4 screen, and extruded into 1.25x14-ft plastic tubes at 15–30psi to give dry-appearing self-supporting columns of d 1.41g/cc, which can be detonated by No 16 cap]

368) R.D. McMahon et al, USP 3346429(1967) & CA **68**, 4591v(1968) [Stabilized blasting expls obtd by using AN contg an anticaking agent and subsequent sensitization to produce expl compns having resistance to cryst

degradation. This objective is achieved by combined use of 0.01–1.0% of a C_8–C_{22} aliphatic monoamine and 0.1–5.0% kaolin clay in the final product. These materials are mixed with AN prior to shipment to the site of blasting immediately after addn of sensitizers, such as NG, TNT and NC. The amines, mentioned above, are more effective than are surfactants (such as dimethylnaphthalenesulfonate) in stabilizing the crystal structure of AN]

369) W.E. Gordon, USP 3347722(1967) & CA **68**, 4593x(1968) [Blasting expls were prepd by combining AN, urea, Al, water and a thickener, along with one or more modifying agents (e.g. bulking agents and catalysts) if desired. Bulking agents included wood pulp, perlite, and microballoons such as described in USP 3338615(CA not found). For example: AN 43.5, $NaNO_3$ 14.0, guar gum 1.5 and perlite 5.6 parts were dry-blended in a Hobart food mill, provided with a steam jacket. Then urea 8.7 and boiling water 23.2% were added to the dry components and thoroughly blended for 20 mins. Finally Al flake 1.9 and S (rubbermaker's grade) 1.9, together with 0.5% Na borate were gradually added while blending was contd for 10 mins. The borate was used to impart gelly-like consistency. A deton rate of 9300ft/sec across a 12-inch gap, by using DuPont target-type probes, was detd by a counter-chronograph]

370) A. Berthmann et al, BritP 1090184(1967) & CA **68**, 41780b(1968) [Thickeners (such as homopolymers and copolymers of unsatd acids, ketones & acetals) for liquid expls (such as NG) are discussed. For example, 10g of a 40% soln of poly(vinylacetate) in EtOAc was mixed with 90g NG and after evapn of EtOAc on a water bath, a clear, homogeneous viscous soln was formed. Condensation products of aldehydes and ketones and of diols and dicarboxylic acids were also suitable thickeners]

371) K. Malkovsky et al, CzechP 122929(1967) & CA **68**, 41781c(1968) [Mixed gelled liq expls of decreased sensitivity to impact, such as contg NG or NGc 48–60, TNT or DNT 5–9, an inorg nitrate (such as AN) 30–45, water <6, and starch, dextrin, guar gum or CM-cellulose <2%, can be poured into a borehole and exploded by usual initiators]

372) H.R. Fee, J.D. Ferguson & R.W. Lawrence, USP 3350246(1967) & CA **68**, 14630w(1968) (Tamarind seed powder as a cross-linking thickener (2–4% by wt) for AN expls is described. Such expls had a long shelf life and were stable over a pH range 5.2–8.0. Thus a slurry consisting of water 18.0, tamarind seed powder 4.0, AN 52.7, $NaNO_3$ 13.0, sucrose 5.0 and flaked Al 7.0 parts by wt, had a pH of 5.2 and deton rate 3900m/sec]

373) W.M. Lyerly, USP 3355336(1967) & CA **68**, 51489c(1968) [Thickened, pourable expl compns, comprising an inorg oxidizing salt, fuel, and water, which were thickened with polyacrylamide and cross-linked galactomannan in a ratio 0.1:1 to 10:1. E.g: AN 20–37, $NaNO_3$ 18–21, fuel (hydrocarbon oil, TNT, powd Al or ferrophosphorus) 8–37 and water 10–20 parts. Included in each hundredweight of the foregoing compn are 0.2 to 0.3 parts guar gum, 0.4 to 1.0p polyacrylamide having a mol wt of $5–6 \times 10^6$ and 10 to 30cc of a 5% soln of K tartrate serving as a cross-linking agent]

374) H.R. Fee & J.D. Ferguson, USP 3356544 (1967) & CA **68**, 51492y(1968) [Blasting compns are described consisting of aq slurries of inorg oxidizer salt (such as AN or $NaNO_3$), a nitroparaffin sensitizer contg 1–3 C atoms, and a supplemental sensitizer (such as flaked Al, coal, DNT, Nitromethane) and a cross-linkable thickening agent (such as guar gum) to provide the required plasticity to the final slurry. A typical compn, consisting of water 24, AN 51.5, guar gum 2.0, Nitromethane 22.5 & NC 1.5 parts had d 1.24 and deton rate 2800m/sec. It could not be detonated by No 8 coml blasting cap and required a booster (PETN, RDX or Pentolite)]

375) J.D. Ferguson, USP 3356545(1967) & CA **68**, 51485y(1968) [Nitrocarbonitrate blasting slurry compns contg flaked Al and DNT as sensitizers, are described. Prior-art inorg oxidizer salt expl compns of the aq-slurry type had generally not been detonable unless hazardous material (such as PETN, TNT, etc) was included. In accordance with Ferguson's invention, slurry-type expls are provided that contain a combination of 3–20% flake Al, with 2–15% DNT (as sensitizer), 8–25% water, 30–75% of an inorg oxidizer, and 0.2–5% of a

cross-linked guar gum thickener. Twenty-one expls of such type are listed in patent]

376) H. Sakai, JapanP 17105('67) & CA **68**, 5149w(1968) [Polynitratopolyols of general formula $R\{[CH_2CH(CH_2ONO_2)O]_nH\}_m$ (I) were hardened by the addn of diisocyanates, or diepoxy compns in the presence of amines. Thus, 50g of I($R=OCH_2CH_2O$, $m=2$ & $n=1$), prepd by adding $(CH_2OH)_2$ to glycidyl nitrate, 0.5g Et_3N, and 30g tolylene diisocyanate were mixed and allowed to stand at RT for 4–12 hrs to give a brown elastic, inflammable resin which was useful as a component of expl compns]

377) Wm. Dick & O.A. Gurton, USP 3356546 (1967) & CA **68**, 41782d(1968) [Blasting expls that have a strength up to that of 50% Blasting Gelatin and have a reduced tendency to ignite firedamp were provided by mixts contg an expl sensitizer (such as NG and/or NG gelatinized with NC or MNT) 12–25, a formate (contg no fibrous material) of a metal, such as Ca (preferred), Na, K, Mg, Ba or Pb, as a fuel 20–30, an oxidizer ($AN+NaNO_3$) 30–50 and a flame quencher (such as NH_4Cl or NaCl) 10–30%. Thus a Blasting Explosive that would not ignite at atm pressure, contg 80/20–NG/NGc 20.0, NC 0.3, AN 14.0, $NaNO_3$ 32.0, NH_4Cl 22.2, Ca formate 10.0, guar gum 1.0 and Ca stearate 0.5%, was prepd conventionally. Its strength was 46.7% of BG, deton rate 2400m/sec and gap test value 9–10 inches]

378) W. Zalachowski et al, PolP 52596(1967) & CA **68**, 23257g(1968) [Plastic rock-blasting expls were prepd in which the content of NG was reduced by the addn of aq soln of glycol. Thus, the expl contained NG (with or w/o NGc) 16–18, collod cotton 0.3–0.5, TNT 9–11, AN 69–73, aq soln of glycol (1:1) 1.3–1.7 and ferrite red 0.1% by wt]

379) K.K. Andreev & V.M. Rogozhnikov, TrMoskKhim-Teknol-Inst No **53**, 163–76 (1967) & CA **68**, 31686m(1968) [Effect of addns of some combustibles on the combstn stability of AN and Amm perchlorate and mixtures based on them. The main conclusions were as follows: The addn of small amts of finely dispersed Al to these mixts sharply increased the possibility of the transition of burning into expln. This transition occurred due to the fact that the hot gaseous burning products penetrated deep into the charges and increased the burning surfaces. The main role of Al was not increasing the total amt of the energy (of burning), but the thermal conduction deep into the charge]

380) Ivan Ivanov et al, RodobivMet (Sofia) **24**(4), 4–8(1967)(Bulg) & CA **68**, 41739u (1968) (Use of expl suspensions of AN in admixt with TNT or Dynamite in some Bulgarian mines is discussed)

381) K.K. Andreev & A.P. Glazkova, TrMosk-Khim-TekhnolInst No **53**, 314–21(1967) & CA **68**, 41768d(1968) (The catalytic action of different mineral substances on the combustion of AN, investigated with a manometric bomb is discussed)

382) T. Yoshida, KôgyôKayakuKyôkaishi **27**(5), 323–26(1967) (Japan) & CA **68**, 51456q(1968) [Sheathed and EqS (equivalent to sheathed) expls, such as contg NG 0–27, TNT 0–12, AN 31–75 & NaCl 26–38% are discussed with 8 refs]

383) K.K. Andreev & A.P. Glazkova, TrMosk-Khim-TekhnolInst **53**, 321–28(1967) (Russ) & CA **68**, 51476w(1968) [The effect of some catalytic additives (NH_4Cl, KCl, $BaCl_2$, NaCl & $K_2Cr_2O_7$) on the combustion rates of Dynammons (**Dinamony**, in Russ) (such as contg AN 81 & C 16% or AN 91.4 & C 8.6%, resp) and on their pressure dependency are discussed. Also is described the influence of additives (such as $K_2C_2O_4$, $CaCO_3$, SiO_2, LiCl, NaCl, KCl, $BaCl_2$, NH_4Cl, HgCl, K_2CrO_4, $K_2Cr_2O_7$, $(NH_4)_2O_2O_4$, KNO_3, $KClO_4$ & graphite on Amatol (Ammotol in Russ) contg AN 80 & TNT 20%]

384) W. Cybulski, ArchGorn (Polish) **12**(3), 189–225(1967) (in Engl) & CA **68**, 14612s (1968) [Testing of safety of 5 types Polish mining expls (compns not given in CA) by various methods against coal dust in the presence of methane below the limit of explosibility, is described. It was decided that the most reliable method for classification of expls with respect to safety of their use in gaseous coal mines was the "cannon method with a slot"]

385) N.Ya. Medvedev, RussP 199727(1967) & CA **68**, 88705w(1968); From IzobretProm-Obraztsy, TovarnyeZnaki **44**(15), 194(1967) (Storage-stable, shock, resistant cylindrical or flat charges are composed of AN 58.83, $NaNO_3$ 14.80, KCl 3.76, NH_4Cl 9.32 & TNT 18.29 parts)

386) J.D. Chrisp, USP 3361601(1968) & CA **68**, 51484x(1968) [Gelled aqueous nitric acid prepd by copolymerizing an acrylic monomer or a monovinylpyridine with a difunctional monomer contg $\geqq 2$ terminal unsatd CH_2 groups in 30–98% aq HNO_3, is described. An expl compn was prepd from 70% aq HNO_3 73.2, acrylamide 5.6, N,N'-methylenebisacrylamide 0.4, Ag nitrate 0.4, $(NH_4)_2S_2O_8$ 0.1 & DNT 20.3%. It had O balance −0.7%, density 1.3 and deton velocity 7050m/sec]

386a) G.L. Griffith, USP 3361603(1968) & CA **68**, 51486z(1968) [Inorg oxidizer salt expl compns contg paper sheet particles as pouring density reducers, are described. Such compns comprised mixts of a nitrate, perchlorate or chlorate of NH_3 and alkali- or alkali-earth metal up to 81%, an expl sensitizer, such as NS (Nitrostarch) (preferred), TNT, PETN, Comp B, etc 5–30 & ground paper 4–16%. Up to 30% (preferrably 0.5–20%) conventional carbonaceous fuel or 0.5–15% metal (Al, Al alloys, ferrosilicon and ferrophosphorus) fuels, 0.3–2.0% antacid 0.5–30% liquid slurrying agent and small amts of thickeners (such as guar gum, CMC, psyllium seed mucilage, pregelatinized starch, silica aerogels, Al_2O_3, attapulgite, and bentonite could also be present. Thus a mixt contg granular AN 81.5, petroleum oil 1.5, "Celite" 1.0 & ground parchment paper 16.0% had d=0.350 and ballistic pendulum value 11.9. The d of a control in which bagasse was substituted for ground paper was 0.420]

387) G.L. Griffith, USP 3361604(1968) & CA **68**, 51487a(1968) [Explosive slurries contg an oxidizer salt and particulate vegetable and/or fruit pulp as a major part of the fuel, is described. Such slurries comprised mixts of 50–70% of nitrates, perchlorates or chlorates of NH_3 and alkali- or alk-earth metals, 25–30% of sensitizing expls, such as NS (preferred), TNT, PETN, Comp B, etc, 0.1–30% of finely divided pulpy vegetable matter, and 7–50% suspending medium, such as water or oil and juices present in the pulp. Metallic and carbonaceous fuels, thickeners, antacids, etc could also be present. Thus a mixt contg dry NS 20, AN 44.6, orange pulp 25, guar gum 0.4, paraffin oil (100 Saybolt Universal sec viscosity) 5 and water 5% provided a good expl slurry, d 1.35, ballistic pendulum value 10.00, viscosity 300cp and deton rate >3600m/sec]

388) V.F. Starokozhev et al, VzryvnoyeDelo **1967**(63/20), 136–52(Russ) & CA **69**, 4077c (1968) [Characteristic features of the deton of high-safety expls involving ion-exchange salts, were examined. A $NaNO_3$-NH_4Cl mixt, contg NG as a sensitizer, being exploded in 40-mm diam charges formed NaCl as result of interaction. With increasing NG content, the role of salts participating in expln increased. The extent of explosiveness also increased in case of confinement (such as strong casing) because this hindered the free dispersion of expl products]

389) P.A. Paramonov et al, Ibid **1967**(63/20), 156–65 & CA **69**, 4085d(1968) (Since it was found that mixts of coarsely dispersed AN and TNT of high-d were not suitable when charged mechanically in borehole because they could not ensure complete detonation, it was decided to replace TNT with phlegmatized RDX and then, later, with pure RDX. Such mixts, as for example, contg: RDX 35, coarse AN 38 & inert matter 27% ensured deton only in large diam charges and, if the content of RDX were reduced to below 24%, the critical diam became abnormally high. By replacing about half of RDX with a mixt of finely dispersed AN and TNT, it was possible to reduce crit diam to as low as 24mm)

390) V.I. Chikunov, Ibid **1967**(63/20), 169–77 & CA **69**, 4080y(1968) [Resite-salt coatings (sheaths) for increasing safety of expls in gaseous and dusty coal mines were tested and found to be satisfactory. The sheaths were made of a mixt of 32% phenol-cresol-aniline-HCHO resite, 27% kaolin, 35% KCl, 3% stearin, and 3% lime. The expl was packed in paraffin-coated paper cartridges and these were placed in cylinders pressed

from the resite-salt mixt cured at 180–190° for 2–3 minutes) (Accdg to CondChemDict (1961), p317-R resite is the C-stage resin)]

391) F.M. Gel'fand et al, VzryvnoyeDelo **1967**(63/20), 177–78 & CA **69**, 11862h(1968) (Testing of new expl, water-resistant **Pobedit VP-4** in Karaganda Basin coal mines showed that it was superior to 3 other brands of expls tested under the same conditions. No compns of any of these expls are given in CA)

392) F.M. Gel'fand & V.I. Mamaev, Ibid, **1967**, (63/20), 182–92 & CA **69**, 4089h(1968) [Reasons for partial detonations (failures) in blast-hole charges of safety expls, were investigated in field tests at Karaganda coal mines. It was found that cracks in the coal layers, carried expln products to adjacent charges, thus disrupting them. Fissures created by deton waves had the same effect. Destruction of adjacent charges could be avoided if the distance kept >0.35m, the diam of the fissures kept at <3mm, and the ignition delay was >250m/sec]

393) F.M. Gel'fand & V.S. Alipchenko, Ibid **1967**, (63/20), 209–12 & CA **69**, 11865m (1968) [Reasons for a failure and break in detonations in flooded coal mines were investigated. Expls **Ammonit PZhV-20** and **Uglenit E-6**, coated with water-resistant material, were used in testing. (Compns are not given in CA). It was decided that the excess pressure of CH_4 in flooded coal faces was the cause of failure and break of detonation]

394) P.A. Paramonov, Ibid **1967**; (63/20), 213–17 & CA **69**, 4083b(1968) [Safety props of coarsely dispersed AN expls contg TNT & NaCl were studied with identical compn, but at different degrees of dispersion. They were tested in a methane-air medium (9.5% methane) by expln of freely suspended chges of different wts. The relative increase of the coarser particles of the active component in the Ammonite mixt lead to increased resistance to firedamp. An increase in the NaCl content of the coarsely dispersed expl also increased resistance to firedamp]

395) L.D. Khotina, VzryvnoyeDelo **1967**, (63/20), 217–19 & CA **69**, 4082a(1968) (Effect of sensitizers on the sensitivity of safety expls to channeling effects: Ammonit PZhV-20 samples to which equal amts of RDX or NG were added showed that with 5% sensitizer, the NG was more effective but with 10% RDX is more advantageous. Thus, an Ammonite contg 10% solid sensitizer detonates more stably under channeling effect conditions than one contg the same amt of liq nitroester)

396) A.N. Zakhnenko & M.K. Pesotskii, Ibid **1967**, (63/20), 227–31 & CA **69**, 11860f(1968) (Statistical method for testing safety expls by ignition of a methane-air mixt was conducted as follows: A known wt of expl was placed in an envelope of sand or coal powder surrounded by atm contg 9–9.5% methane and then exploded. If no ignition of methane took place in several tests, the wts of samples were increased until number of ignitions against nonignitions became slightly above 50%. Then the wts were decreased to have ignitions drop below 50%. This allowed calculation of the wt of sample required for 50%' ignitions. The influence of temp and moisture of the gas mixt and of the quality of the envelope were also studied)

397) F.N. Pys & I.T. Kolesnichenko, Ibid **1967**, (63/20), 232–37 & CA **69**, 11861g (1968) (Industrial testing of mining expl **Aquatol** at Kal'makyrsk and Kurgashinkansk mines showed that the expl is effective, inexpensive and easy to use in both dry and wet quarries. The compn of Aquatol is given in the Section which follows these AddnlRefs)

398) M. Gregor et al (Czech), IndChimBelge **1967**, 32 (Spec No,Pt2), 373–76 & CA **71**, 31916y(1969) (Stabilization of AN with dolomite has advantage over stabilization by chalk)

399) O.A. Gurton & Wm. Dicks, IndChimBelge **1967**, 32 (Spec No, Pt 3) 511–14 & CA **70**, 49149q(1969) (An expl **Dynagex**, designed specifically for delay blasting in hard coal) (See in this Vol its brief description)

400) Paul Lingens, IndChimBelge **1967**, 32 (Spec No, Pt 3), 515–18 (Ger); CA **70**, 79647t(1969) [Thermal decompn of inorg salt-based expls contg 8–15% NG–NGc mixts

was studied by a combination of "Thermogravimetry", "Differential Thermal Analysis" (DTA) and by measurement of the temp profile of the heat of localization in a steel tube filled with kieselguhr. Results of tests are listed for German expls **Carbonit B**, **Energit A** and **Nobelit B.** Compns of these expls are not given in CA]

401) C. Fossé, IndChimBelge **1967**, 32(Spec No, Pt 3), 527–31 (Fr); CA **70**, 89333b(1969) (Effect of priming on the pressure exerted in water by industrial expls was determined in Sterrebeek, Belgium. Expls such as AN-fuel oil were confined in Plexiglas and exploded under water using different priming methods. Photographic recording of the shock wave and of the light emission in the water gave new exptl data permitting more accurate understanding of the kinetics of the different priming methods)

402) G. Nenquin & G. Fally, IndChimBelge **1967**, 32(Spec No, Pt 3), 606–09 (Fr) & CA **70**, 49150h(1969) [The combstn of various mixts of NH_4 salts and alk or alk-earth nitrates, and esp NH_4Cl-KNO_3 (which constitutes the base of ion-exchange safety expls) was studied. It was found that combstn depends mainly on the acid linked to the NH_3 mol and that the acid liberated during reaction reacts with the nitrate to form O and N oxides, which support combstn of NH_4. In NH_4Cl-KNO_3 mixts, max combstn pressure was obtd with the 1.5:1 mixt. Concn of N oxides in the burned gas was much lower than with the 1:1 mixt. A new formulation which gave ion-exchange expls greater detonability, a significant reduction in concn of N oxides in deton products, and an appreciable increase in the energetic yield was developed, but not listed in CA]

403) Cléret de Langavant, IndChimBelge salts (such as nitrates of NH_3, Na or Ca) 30–90, a thickener (such as guar gum flour, carob seed, starch, etc) 0–10, conventional fuels 0–40, sol lignosulfonate (such as that of NH_3, Na or Ca) 0.1–25 and a solvent (water preferred) 1–30 parts. For example: a slurry contg AN 47.3, Al powd 30, water 10, $HCONH_2$ 10, Na lignosulfonate 2.0 & guar flour 0.7% by wt could be fired in a 3x24-inch cartridge with only 20g Pentolite initiator. Addn of as little as 0.5% of Na lignosulfonate to expl slurries contg 10% PETN & 20% TNT, which were insensitive, made them sensitive to No 8 cap]

409a) J.D. Chrisp, USP 3372072(1968) & CA **68**, 88704v(1968) [Expl slurries of conventional oxidizers & fuels and water which can be gelled by polymerization, in situ, are described. For example, $NaNO_3$ 14.7, Al powd 3.9, powd coal 5.3, acrylamide 1.9, and methylenebis(acrylamide) 0.2 part were dispersed in 73.8ps 65% aq AN soln held at 54° and 0.2 part $(NH_4)_2S_2O_8$ stirred in. After 2 mins, a medium-to-firm gelled blasting agent was obtd. Its rate of deton in a 3-inch diam column at 85°F was 4500m/sec]

410) E. Reinsalu, TrNauch-IssledInstSlantsev **1968**, No 17, 10–16(Russ) & CA **73**, 5590j (1970) (Relation between the yield of finely divided shale and the specific consumption of expls, was discussed. A series of equations was developed that relates to the sp consumption of expls in kg/m^3 in borehole blasting to the production of fines. The relation between production of fines and the sp consumption of expls was found to be nonlinear. Increasing the latter above 0.4–$0.7kg/m^3$ lead to a sharp increase in the amt of fine shale particles)

411) Ch.H. Grant & Th.E. Slykhouse, USP 3377909(1968) & CA **69**, 20841c(1969) [A 2-component expl of which the 1st component consists of AN 94 & fuel oil 6%, while the 2nd mixt consists of Al (40–100 mesh) 30, formamide 10, water 12, Karaya gum 1, $NaNO_3$ 10 & AN 37%. The 1st mixt can be located in the borehole either adjacent to the 2nd mixt or be surrounded by it]

411a) G.L. Griffith, USP 3378415(1968) & CA **69**, 28937t(1968) [Inorg nitrate expl slurries are described, which were prepd as follows: A blend of 86.24 of AN and 7.84 parts $NaNO_3$ was melted in a steam-jacketed kettle and then poured into a mixing trough in which the melt was blended with 4.3 nut shell meal and 1.62 parts water. After cooling the blend slightly to a plastic consistency, it was squeezed thru a 30-mesh screen and allowed to harden. The resulting mass was composed of particles 49.25% retained on a

No 10 screen, 39.25% on a No 18, 10.25% on a No 35, 1.25 on a No 60, and none thru a No 60 screen. This material (84.6 parts) was mixed with No 5 oil 1.5, DNT 2.5, Jaguar guar gum 5.0; NS 6.1 & Fe oxide 0.3 part and then 11kg of dry mixt was wetted with 5.6kg of water and allowed to stand overnight. A 5-in x 5-lb Pentolite booster was poured into a 5-in x 25-lb "ICC 25G 50" cartridge, and to this was added 9725g of the above material after packing it in an 8 mil polyethylene liner. The resulting cartridge was detonated under 15 to 20ft of water with good expl effect]

412) D.D. Perry et al, USP 3378416(1968) & CA **69**, 11882q(1968) [Liquid expl mixts of polynitro org compds and of aliphatic carboranes, suitable for mining and quarrying, are described. Polynitro compds suitable for use in these expls were: polynitroparaffins such as $C(NO_2)_4$, NG, NGc, DGTeN, PETN & TNT and for carboranes (and their mixts) the following were listed: methylcarboranes, n-propylcarborane, iso-propylcarborane, vinylcarborane, isopropenylacetylene, etc. Mixing of org polynitro compds with carboranes produced very sensitive or spontaneously detonating expls. As an example was cited a homogeneous mixt prepd by adding slowly to 18.4 parts isopropenylcarborane (agitated in a reactor by bubbling a stream of N), 78.4 parts of $C(NO_2)_4$. $C(NO_2)_4$ by itself is not an expl, but an extremely powerful oxidizer and might cause a spontaneous detonation in presence of even small amts of C contg substances]

413) Wm.D. McFerrin, USP 3378417(1968) & CA **69**, 4117r(1968) [Inorg nitrate expls of any desired expl equivalent or strength

1967, 32 (Spec No, Pt 3), 617–20 (Fr) & CA **70**, 69728f(1969) (Improvement of the power of nonfiredamp expls was achieved by two methods. One method involves the use of an intimate mixt of an alkali nitrate and Amm chloride formed from large grains. These grains were obtd by the double decompn, in the presence of water vapor, of AN and an alkali metal chloride of larger grain size. The resulting product was then dried and a sensitizer with a hydrocarbonaceous material added. In the 2nd method, a compn consisting of AN grains, having small pores, was used. The diam of the grains was 1–2mm and the surface area of the NaCl added was >700 sq.cm/g. Finally, a sensitizer and a hydrocarbonaceous material were added)

404) N. Soda & Sh. Endo, JIndExplsSoc, Japan **1967**, 28(2), 154–58(Japan); From CZ **1968**(42), Abstr No 2922; CA **70**, 49151j (1969) (Explodability of AN-metal powd systems was investigated by means of shock-sensitivity tests and the Krupp Ignition Test. The results were dependent on the type of metal. The ignition point decreased in the order of metals: Zn<Mg<Pb<Fe<Cu<Ti-<Sn<Sb<Al. The activation energy of the ignition reaction was calcd from the ignition-delay-time at different temps)

405) Marcel Vereauteren, AustrianP 277027 (1967) & CA **72**, 45655v(1970) (In the process for manuf expls, AN was crystallized from concd or saturated solns in the presence of 0.1–5% by wt C_8–C_{18} primary amino alcs and their salts added in amts 0.4–1.0%. Thus a mixt of 25g n-dodecylamine acetate and 75g triethanolamine acetate was added to 10kg of an aq 80% soln of AN at 65° and the soln allowed to crystallize for 24 hrs without stirring. The crysts formed were filtered off and pressed to obtn a cryst mass contg 3.2% water)

406) R. Deniau, BullTechChambreSyndMines FerFr **1967**, No 88, 143–55(Fr) & CA **72**, 4823e(1970) (A review is given with no refs on blasting with resultant small quantities of noxious gases in the Loraine iron mines)

407) G.L. Griffith, G.A. Lyte & F.B. Wells, USP 3366053(1968) & CA **68**, 70809(1968) [Free-flowing, inorg nitrate based expl mixts sensitized to initiation by incorporation of 1–4% powd Al and 1–6% water, are described. Expl sensitizers, such as NS (preferred), NG, TNT, PETN, RDX, Pentolite or Comp B 5–7, carbonaceous fuels 0.4, stabilizers-antacids (such as $CaCO_3$ or MgO) up to 0.4, and materials (such as rosin, metallic resinates, hexamine, waxes or mineral oil) 0.4% could also be present. A mixt listed in CA contg AN 70.68, $NaNO_3$ 8.91, Al powd 1.57, NS

12.09, CaCO$_3$ 0.2, wood flour 6.54 & C black 0.01% was a blasting expl of d 1.08–1.10, sensitive to a No 10 cap. When 4.45 parts water was mixed with 95.55ps of the above compn, the product had d of 1.48–1.52 and was sensitive to a No 8 cap. If, however, the water was added in bound form, as by addn of 19.6ps of Na borate decahydrate, the d became 1.16 and the expl became sensitive to a No 16 cap]

408) R.B. Clay et al, USP 3367805(1968) & CA **68**, 80091x(1968) [Inorganic nitrate aqueous slurry blasting compns contg Al and having a high-area lyophobic surface are described. These compns are composed of inorg oxidizers, water or aq liquid, a thickener (such as guar gum and starch) and a small amt of particulate Al having a surface of $\geqq 0.5m^2/g$ together with a lyophobic surface capable of forming void reaction sites. To this mixt a phosphate inhibitor was added to prevent premature reaction betw water and Al & other fuel and to bring the O balance to within ±10%. The carbonaceous fuel could be a finely divided coal, gilsonite, flour, or sugar. Molasses imparts not only substantial fuel value, but also plasticity and thixotropic properties. A typical blasting compn that can be pumped into boreholes consists of AN 38.0, NaNO$_3$ 15.0, water 17.0, a phosphate stabilizer 0.1, Al powd 1.1, S 6.0, gilsonite 4.0, guar gum and particulate AN 17.7% by wt. The first three ingredients were combined to make a liq soln and the others were added into the liq cold (or moderately heated) and blended]

409) J.F.M. Craig & E.L. Falconer, USP 3369945(1968) & CA **68**, 88703u(1968) [Storage-stable, sensitive, expl slurries are described, which contained inorganic oxidizer can be prepd by using AN of predetd particle size distribution (as percent of the total compn by wt). The prescribed grain size was given in terms of Taylor Sieve Screen (See Table 1, p A674 in Vol 1 of Encycl), as follows: 78% of AN must pass a 150-mesh screen (0.0041-inch openings), while the retained material must pass a 100 mesh (0.0059-inch openings)]

414) J.W. Francis, USP 3379586(1968) & CA **69**, 4116q(1968) [Gelled Ammonium Nitrate-Nitromethane-Nitropropane-Thickener expl compositions having high rates of deton, but being non-cap-sensitive, are described. They were useful in marine seismic exploration work. For their prepn 800 lbs fertilizer-grade AN prills (previously ground to pass 10-mesh screen of which 22% passed 200-mesh) were intimately mixed with 104 lb MeNO$_2$, 86 lb PrNO$_2$ & 10 lb NC. The resulting expl had rate of deton ca 12000ft/sec]

415) M.A. Cook, USP 3379587(1968) & CA **69**, 4115p(1968) [Inorganic oxidizer salt blasting slurry compns contg formamide are described. They had relatively high d's ($\geqq$1.3) and bulk strength, good water-resistance and good sensitivity to initiation. Their fr p was ca −10°F because the water content was low thru use of extenders to replace all or part of the water. As oxidizers were used AN and/or NH$_4$ClO$_4$ in mixts with alkali or alk-earth nitrates, chlorates or perchlorates; as an inorg sensitizer fine Al (up to 5%) (or when mixed with up to 40% coarse Al, up to 10% ferrosilicon or ferrophosphorus was included); (ferrophosphorus could be used without Al) ; up to 6% thickener, 5% water, 10% of a water extender such as glycol, glycerol, MeOH, EtOH, HCONH$_2$, or HCONMe$_2$, and the conventional fuels. Thus, the expl slurry prepd by mixing AN 17.75, Ba(NO$_8$)$_2$ 19.60, NaNO$_3$ 14.7, TNT 19.6, ferrophosphorus 11.75, water 12.73, ethyleneglycol 2.94, guar gum A 0.294 and guar gum B 0.392%, had d 1.77–1.82, O balance −10.45%, fr p −12°F, crit diam at 15°C 2.5 inches and pH 6.2]

416) D.K. Kuel, USP 3381473(1968) & CA **69**, 20837f(1968) (High-energy fuel systems suitable for use in Dynamites, expl slurries and hybrid rocket systems are described. As fuels Zr, Mg, Si, Ti, Cr and particularly Be, Al & Li alloys and mixts were used. All of these metals, especially Be, Al & Li readily formed protective oxide coatings. When a surface of a base metal was coated with at least one metal (such as Be or Al) whose oxide formed in admixt with that of the base metal, at least one eutectic with mp below the ignition temp of the base metal, the ignitability was improved, and the speed of combustion increased and proceeded to completion. Examples of coated systems and their mp's are given in CA)

417) M.A. Cook, USP 3382117(1968) & CA **69**, 11881p(1968) [Thickened aqueous blasting agents, which can be pumped, are described. They consisted of an inorg oxidizing salt (AN, $NaNO_3$, $Ba(NO_3)_2$, or their mixts), a sensitizer (TNT, RDX/TNT, PETN/TNT, EDNA/TNT, or smokeless proplnt), a thickener (guar gum), water and a polar org solvent (iso-PrOH, a polyhydric alc or an amide). These solvents could replace water to lower the fr p of the mixt. Air or other gas could be introduced to decrease the density. Thus, a typical compn consisted of 75-mm, M-1 smokeless proplnt 25, AN 43, $NaNO_3$ 10, S 2, water 4.5, formamide 10, and cross-linking borated guar gum 0.7 parts. Its d was 1.46 and a crit unconfined diam 5 inches]

418) V.J. Russo, USP 3388014(1968) & CA **69**, 45032c(1968) (Method of prepn of a strong porous prilled AN of bulk d 0.80

419) H.R. Fee, R.W. Lawrence et al, USP 3390028(1968) & CA **69**, 45033d(1968) (Aqueous slurry expls which had a high shock energy and low susceptibility to dust explns, are described. For example, water 17.0, AN 28, and $NaNO_3$ 12.6 parts, were heated to 100°F and treated with 25.0ps smokeless proplnt and 14.0 of Si powd. The pH was adjusted to 4.5–5.0 with HOAc and a dispersion of 0.7ps guar gum in 3 times its and oil retention ca 6.7%, is described. These prills were economical to prep and they produced in combination with other substances, very effective blasting agents) wt of ethyleneglycol, were mixed with the soln. A dispersion contg 0.065ps $NaNO_3$ in 3.5 times its wt in water was added, followed by a dispersion of guar gum cross-linking agent in ethyleneglycol. The resulting expl slurry had an O balance of −18.3, d 1.35, pH 4.7, and deton rate 5500m/sec)

420) R.F. Preckel, USP 3390029(1968) & CA **69**, 45034c(1968) (Expl compns consisting of an inorg oxidizing salt suspended in an org fuel-solvent suspension of the salt, and a thickener, or a cross-linking agent, are described. Thus, systems thickened with 0.5% cellulose acetate and contg NH_4ClO_4 60–70, Al 30–20, and $HCONMe_2$ 10% were tested for crit diam for propagation of deton. In all cases neg results were obtd at 2.6–5 inches diam, including tests in a steel pipe, using a 1-lb Pentolite booster. In another test, 220-lb charges, contg NH_4ClO_4 72, Al 16, and $HCONMe_2$ 12%, with 0.5% added cellulose acetate were cast in a steel pipe and detonated under 12 ft of water with 5-lb Pentolite in direct contact. High-order deton with velocity in the order of calcd values was obtd. Similar systems contg no solvent had crit diam $\geqq$ 20 inches)

421) H.R. Fee, R.W. Lawrence et al, USP 3390030(1968) & CA **69**, 45036g(1968) [Aqueous slurry blasting compns which contain Si alloy and an aeration agent, have decreased susceptibility to dust explns and excellent underwater shock & thrust energies. The compns are comprised of AN 30–75, $NaNO_3$ 5–25, particulate Si alloy (contg 85% Si) 3–15, carbonaceous fuel 3–15, thickening agent (guar gum) 0.4–4, water 10–30, and $NaNO_3$-$NaHCO_3$ aeration mixt 0.04–0.4%. As an example of prepn, a soln AN 52, $NaNO_3$ 15.6 & water 16.5 parts was adjusted to pH 5.4–6.0 with NH_4OH or HOAc. A mixt contg $NaHCO_3$ 0.046, $NaNO_3$ 0.046, ground coal 1.2, natural guar gum 0.4, and cross-linkable guar gum 0.8 part was mixed with Si alloy 8.5 parts. The above soln and the mixt were blended at 140°F to yield the desired compn, which has an O balance of −3.4%, d 1.15, pH 5.2 and deton rate 5250m/sec]

422) A.A. Albert, USP 3390031(1968) & CA **69**, 53324q(1968) [Gelled aq slurry expl compns contg an inorganic nitrite are described. The nitrite serves as a gas-generating substance for adjusting and improving the control of sp gr, thereby providing a means for prolonged energy control of the expl and for imparting increased sensitivity, and underwater shock & thrust energies. Thus, 0.065 part $NaNO_2$ was added to a mixt comprised of 41.7ps AN & 13.0 $NaNO_3$, slurried with water 17 at 100°F & ground smokeless proplnt 25ps. After adjusting the pH to 4.5–5.0 with HOAc, 0.7 part guar gum in 2.1p ethyleneglycol was added and mixed in the sol, followed by 0.08 part cross-linking agent (guar gum) in 0.4p glycol. The re-

sulting compn, tested underwater gave deton rate 5400m/sec, shock energy 0.78, and thrust energy 0.86, relative to confined 60% HP Gel]

423) A.A. Albert & R.W. Lawrence, USP 3390032(1968) & CA **69**, 45031b(1968) [Gelled aq slurry expl compns contg a carbonate or bicarbonate with a nitrite as gas generating agents, are described. Thus, AN 53.5, $NaNO_3$ 16.1 and water 16.9 parts were mixed & heated and the pH adjusted to 5.4–6.0 by NH_4OH or HOAc. Separately blended $NaHCO_3$ 0.025, $NaNO_2$ 0.025, ground coal 1.2, and guar gum 1.2 parts were mixed with Si or Al (amt not shown in CA) and then blended with the above soln at 140°F. The resulting gelled slurry, tested under water, gave deton vel 5550m/sec, shock energy 0.52, and thrust energy 0.74, relative to confined 60% HP Gel]

424) J.J. Minnick & G.W. Bell, USP 3394038 (1968) & CA **69**, 53326(1968) [High-density AN-FO expl compns contained a dense AN (d=1.3–1.7), ground to 8 to 200 mesh and mixed immediately and thoroughly with fuel oil. A typical compn consisted of ground AN 60.2, whole prilled AN 30.0, No 2 fuel oil 2.5, powd coal 4.0, Ca chloride 2.0 and clay 1.3%. This compn had package d 1.11 and exhibited deton rate of 10500ft/sec when primed with Tetryl]

425) J.B. Bronstein, Jr, USP 3395056(1968) & CA **69**, 68701j(1968) {Slurried expls consisting basically of AN as an oxidizer, MeOH as fuel, and 0.1–3% alc thickening agent [such as Me hydroxypropyl guar gum, poly-(oxyethylene)glycols and glycol ethers (Ucar resin C-149 & WSR-301)]. It also contd water and sometimes powd metal, chlorates, perchlorates, other nitrates than AN, and an expl sensitizer (such as NS). These expls were particularly suitable for rock blasting. For example, an expl mixt was prepd from granulated AN 84.30, MeOH 10.0, water 5.0, Ucar resin C-149 0.65 and NH_4OH 0.05% by wt. The pH of the mixt was 7.6. It was pumped into an 8-inch diam borehole and provided every 8 ft of rise with 2-lb Comp B primer for complete deton}

426) J.P. Merryweather et al, USP 3397095 (1968) & CA **69**, 78895q(1968) [Gelled aq AN expl compns which were freeze-resistant and nonhardening were obtd by incorporation of a small amt of H_2NCN, and espec of H_2NCN with urea. A typical expl compn was made as follows: to 5-lb AN and 20-lb flake TNT in a blender, a soln of AN 30, urea 3 & water 15-lb was added and the mass mixed for 1 min. Two lb of buffered 50% aq H_2NCN soln was then added with mixing for 30 sec, after which a premixt of 14-lb Al powder and 1-lb guar gum was added. The mixing was then continued until hydration had advanced to a thick enough consistency to suspend the solids (ca 4 mins). Finally 10-lb $NaNO_3$ was added and mixing contd until the mixt became homogeneous (ca 1 min). This compn had good freeze resistance at 20°F]

427) M.A. Cook, IEC **60**(7), 44–55(1968) & CA **69**, 60433c(1969) [A review is given of slurry expls and slurry blasting agents. The boostering, detonation characteristics, strength, and sensitivity of these materials are described. Included are 33 refs]

428) E.M. Atadan & C.H. Noren, USP 3397097 (1968) & CA **69**, 78899u(1968) [Gelled AN-Fuel aq blasting expls of sufficient deton sensitivity were prepd by incorporating gas bubbles and a crystal-habit modifier. For example, an expl was prepd by placing in a rotary mixer AN "neutral liquor" (normally 80%) at ≧150° and, after starting the agitation, adding a crystal-habit modifier (Na methylnaphthalenesulfonate), followed by a sol fuel (granulated sugar), $NaNO_3$, and guar gum. Finally a cross-linking agent of guar gum was added and the mass mixed for 3 mins and loaded into containers]

429) G.L. Griffith, USP 3399089(1968) & CA **69**, 78897s (1968) (Gelatinized NS-DNT expl sensitizers suitable for incorporation in various formulations, as for example in blasting expls, such as contg AN 54.20, NS 22.00, DNT 4.15, $NaNO_3$ 14.80, nut meal 2.00, flake Al 2.50, DPhA 0.05 & No 5 oil 0.30%. Its d was 1.195 and deton rate >3418m/sec)

430) Ph.W. Fearnow, USP 3400026(1968) & CA **69**, 88493w(1968) [Thickened aq inorg oxidizer salt expls contg dissolved proteinaceous materials, are described. The sensitivity of blasting agents is markedly improved by addn of 0.01–10% (preferably $\sim$3%), proteinaceous material (preferably foamable) and having mol wts of 5 to 200 thousand (preferably $\leqq$100000) (such as powd egg white, lactalbumin, ovoglobulin, lactoglobulin, conjugated protein and mucin). Such materials were added to a mixt held at 150–170°F and having a pH of 3–10. Thus, to a hot aq soln contg 47.9 lb AN and 16.1 lb water, a premix of 15.8 lb $NaNO_3$ and 1 lb guar gum was added, followed by flake Al 2.0, DNT (mp 26°) 11.0, granulated sugar 5.0, and formamide 2.0 lb. After 4–5 min of mixing, 5% aq $Na_2Cr_2O_7$ 100 ml and 5% aq KSb tartrate 200ml were stirred in. The resulting blasting agent had d 1.24, deton rate unconfined in 4-inch column at 60°F 4600m/sec and 3820 in 2-in column at 72°F]

431) H.R. Fee & R.W. Lawrence, USP 3401067(1968) & CA **69**, 98127u(1968) [Slurry expls contg alkanolamine nitrates (such as ethanol- or propanolamine nitrates) as a sensitizer, an oxidizer salt (AN and $NaNO_3$), a thickener (guar gum), generally in cross-linked form an water. Supplementary sensitizers (such as finely divided metal and coal could also be used). A typical compn consisted of AN 44.4, $NaNO_3$ 15.0, ethanolamine nitrate 18.3, guar gum 1.5, Al 5.0, and water 16% by wt. It deton rate was 5000m/sec]

432) Louis McDonald, USP 3403061(1968) & CA **69**, 98129w(1968) [Conditioning particulate materials for use in org expls, is described. Solid pulverulent materials (such as RDX, PETN or AN) suitable for suspension in melted org expls (such as TNT) were treated before dispersion, to make them wet and disperse better, with $\geqq$ 1 liq or solid org Si compd (such as methylchlorosilane of the general formula $R^1R^2R^3R^4Si$, in which the R's are alkyl, aryl, alkoxy, or aryloxy groups, resp, but $\geqq$ 1 of them is a halogen atom or an NH_2, OH, alkoxy, or aryloxy group). Thus, wet RDX was dried and, after being treated with 30 parts Me_2SiCl_2 in 70ps dimethylsiloxane was incorporated into melted TNT for formulation of Comp B or Cyclotol. Expls made in this manner had a more const compn thruout melting and freezing cycles. Also, slurries prepd from such treated expls showed virtually no change in apparent viscosity during 24-hr intervals of heating and agitation]

433) Ph.W. Fearnow, USP 3406051(1968) & CA **69**, 108263b(1968) [Aqueous expl compns free of self-expl sensitizers (such as PETN or TNT) are described. These compns were prepd by the following series of steps in a turbine-type slurry mixer: AN "neutral liquor" (78–80%) at 150–180°F was placed in the mixer and the pH of the liquor adjusted to 4.5–5.0 by addn of nitric acid. KSb tartrate cross-linking agent was added and agitation was begun. A solid carbonaceous material was added (if specified) with mixing and the temp dropped to 140°F. A proteinaceous material (such as egg albumin which causes foaming) was added, and mixed for ca 1 min. The dispersing agent (such as formamide) was then added, followed by the addn of DNT, and mixed for ca 2 mins. A premixed thickening compn of guar gum & $NaNO_3$ was added next and mixed until thickening was observed (4–5 mins). Finally AN prills and carbonaceous fuels (if specified) were added and mixed. A cross-linking agent ($Na_2Cr_2O_7$ in soln) was added while the slurry was pumped into borehole. These compns detonated at ca 5000m/sec at 60°F]

434) R.B. Clay, FrP 1492709(1968) & CA **69**, 4114n(1968) (Sensitive nitrate-base aq slurry expl compns are described. For example, a compn designed for use in cold boreholes was prepd by adding a mixt of chopped Al foil 10, S 4, gilsonite 2, guar gum 0.6 & $NaNO_3$ 12.7 to a soln of AN 38, $NaNO_3$ 15, stabilizer 0.1 and a soln of guar gum 0.2 in ethyleneglycol 0.4 in water 17%)

434a) W.C.G. Baldwin, BritP 1109256(1968) & CA **69**, 4119t(1968) (Expl compns safe to use in coal mines with hazardous atm of firedamp and dust, are described. A typical compn consisted of NG 16.0, NC 0.3, Na CM-cellulose 1.0, Na formate 25.0, kieselguhr 3.0, AN 27.0, NH_4Cl 7.0, $NaNO_3$ 15.1 &

NaCl 5.6%. For prepn of low-freezing mixts, part of NG was replaced with NGc)

435) E. Ludolphy & A. Berthmann, GerP 1273393(1968) & CA **69**, 98130q(1968) [Stabilization of nitric esters of polyhydric alcs (such as mixts of NG with NGc) for use in Dynamites, was achieved by incorporating small amts of acetylacetone, benzoylacetone, or phloroglucinol. Mixt of NG 60 & NGc 40% stabilized with 1% acetylacetone withstood Abel stability test for >300 mins]

436) V. Rank et al, GerP 1274479(1968) & CA **69**, 78894p (1968) [Mining expl.compns are prepd as described below: AN prills were ground, the 0.005–0.125mm fraction pressed (under 200kg/sq.cm) into 1.5–2mm thick pellets having 10% porosity, the pellets broken, and the 0.5–2mm fraction separated. This fraction was treated with 5.5–6% of a nonexpl C-carrier, such as Diesel oil. The oil was completely absorbed by AN, but it did not fill all of the pores. Then the mixt was sprayed with spindle oil (110g oil on 2kg AN) and cartridged. Its deton rate was 2900 m/sec at 20cm from the Gelatine-Donarit primer and 3300m/sec at 80cm from the primer]

437) S. Deli & I. Deli, HungP 154740(1968) & CA **69**, 68698p(1968) (Hung) (Permissible and nonpermissible expls of high-brisance and increased storability. Example of permissible expl: explosive oil 34.0, NC 1.25, rosin 0.80, AN 11.0, sawdust 1.2, and limestone powder 51.75%. Example of nonpermissible expl: expl oil 60.0, NC 3.4, rosin 0.8, AN 16.6, sawdust 0.7 and limestone 18.5%)

438) African Expls & Chem Inds Ltd, BritP 1122708(1968) & CA **69**, 68699q(1968) (Dry expls contg lignosulfonate and AN, as for example, permissible blasting expl consisting of: 90.2% prilled AN, 3% of 50% Ca lignosulfonate soln, and 6.8% sunflower seed husks. Its deton rate was 2000m/sec in a 1-inch steel pipe)

439) D.W. Briden et al, CanP 778710(1968) & CA **69**, 4112k(1968) [Continuous mixing of AN-liquid hydrocarbon (such as fuel oil), using a spiral screw conveyor is described]

440) E.L. Falconer & C.J.N. Kelly, CanP 784637(1968) & CA **69**, 78893n(1968) [Expl compns contg a soluble polyflavonoid sensitizer, are described. For example, a slurry expl compn contg AN 51.5, $NaNO_3$ 18.0, urea 1.2, tamarind flour 0.8, particulate Al 18.0, water 9.0 and polyflavonoid (Na salt of Rayflo-C) 1.5%, detonated at velocity of 3580m/sec when packaged in 2.5-inch diam cartridges and initiated with 100g Pentolite. A similar expl which contd 54% AN but no polyflavonoid failed to detonate when initiated by 100g of Pentolite]

441) R.W. Manderstrom, CanP 789482(1968) & CA **69**, 108262a(1968) [AN-fuel expl slurries were made by coating 40–85% by wt of AN prills (porous, ~0.025–0.1-in diam, bulk d 0.65–0.85) with 2–8% liq hydrocarbon (fuel or mineral oil), 50% of which could be a water-sol fuel (sugar or $HCONH_2$) and blending the coated prills at 30–90°F with a thickened aq oxidizing salt mixt or soln comprising 10–20% water, 0.25–2% guar gum, and one or more salts (NH_4, K, Na, Ca nitrate and/or K_2CrO_4 or $K_2Cr_2O_7$). These slurry compns remained pliable at temps as low as −15°F, their d was 1.2–1.4 and O balance +10 to −10%]

442) P.R. Goffart & L. Waterlot, Explosifs (Belg) **1968**, 21(2), 49–69 & CA **69**, 108245x (1968) (Ion-exchanged safety expls contg NG NGc and $NaNO_3$ (or KNO_3), and/or NH_4Cl showed increased heat and energy of expln by addn of an equim amt of NH_4 oxalate. Addnl advantages were offered by this in reduced toxicity of the product gases and reduced possibility of deflagration so that they were suitable for underground works)

443) G.V. Duganov et al, IzvVysshUchebZaved, GornyiZh **1968**, 11(3), 65–8 (Russ) & CA **69**, 11871k(1968) (Effect of gas liberation from material blasted loose in coal mine atmospheres. Tests were conducted in coal mines of the Krivoi Rog Basin using **Dinoftalit** and **Ammonit-6ZhV**. After removal of the blasted material the concn of CO & NO_2 exceeded the standards. To reduce adsorption of CO and NO_2 on the dust of blasted material, it was recommended to drill

blast holes by hydraulic means and to moisten the blasted material prior to and during its removal)

444) A.N. Khanukaev & V.D. Trofimov, Ibid, **1968**, 11(7), 68–73 & CA **69**, 108244w(1968) [Results of testing protective charges designed at the Institute of Mining at Petrograd for cutting exploratory holes in shafts unsafe because of firedamp and dust. Three types of **Ammonits** were tested. The protective charge (sheath) consisted of a double-walled polyethylene capsule (I.D. 84 & O.D. 150mm), filled with water betw the walls. The expl (1.65kg) was inside the capsule and the opening was covered with a 35-mm thick layer of salt and a polyethylene top. The ensemble was placed in a hole (42-mm diam and 2.2-m long), the open end of which was filled with clay, and the remaining space with water. In the pore perpendicular to this was placed a *vibrigraph* to record the results of expln initiated by an elec detonator. A Cu tube with expls was also used to detect the compressive wave of expln. By this method, safe, min distances were established for expls of different power]

445) J.J. Minnick, USP 3409484(1968) & CA **70**, 21467t(1969) (Aqueous alkylamine expl slurries contg an inorg nitrate oxidizing salt, a lower alkylamine nitrate, water, a non-explosive, finely divided, air-entrapping material as sensitizer, and a thickening agent, are described. For example, a compn composed of AN 46.8, water 16.7, resin balloons 2.0, guar gum 1.2 and methylamine nitrate 33.3% had a deton rate of 21000ft/sec)

446) J.J. Minnick, USP 3409485(1968) & CA **70**, 21469v(1969) [Thickened slurried expl for small boreholes contained 10–90% of an inorg nitrate oxidizing salt (such as AN), 5–50% water, 5–50% of tris(hydroxymethyl) nitromethane (I), a thickener (such as guar gum) and a sensitizer consisting of finely divided non-explosive, air-entrapping material (such as resin balloons, bagasse, woodflour, cork or balsa). For example, a mixt of AN 72.2, water 10, resin balloons 2, (I) 15 & guar gum 0.8% had a deton rate of 16696ft/sec] *Note:* Prepn of "resin balloons" is described in AddnRef

447) D.S. Partridge, USP 3409486(1968) & CA **70**, 21468u(1969) [Aqueous expl slurries contg Amm perchlorate sensitivity stabilizer are described. As an example, a blasting compn, prepd by mixing AN prills 46.2, $NaNO_3$ 10.0, granular Al (94–100% <8-mesh, ≤5% <100-mesh US) 15.0, Al (paint-grade flakes) 1.0, hexamethylenetetramine 3.0, guar gum 0.8 and water 24.0%, was given]

448) M.E. Maes, USP 3419443(1968) & CA **70**, 69763p(1969) [Hydrazine contg liquid, and substantially flameless & smokeless expl compns, having a high deton rate, low fr p, viscosity, and impact sensitivity are provided by mixts of 70–92% of an oxidizer (such as $N_2H_5NO_3$ & its mixts with $N_2H_5ClO_4$ in a 4:1 ratio for increased sensitivity), and including up to 25% of metal salts (such as Na, K, Ca or Al nitrates and perchlorates for reducing foaming during prepn). Also are incorporated 6–15% N_2H_4, and 2–15% NH_3 (as a fr p depressant and viscosity reducer). From 2 to 6% desensitizers (such as oil, glycerol, glycol) or up to 20% water are added, as well as 0.5–5% thickeners (such as Cab-O-Seal or Guartec 503) and 0.5–5% wetting agents (such as Me_2SO_4, $HCONH_2$, EtOH, PrOH & various fatty acids) to aid in adsorption. One such expl, which contd less than 20% water detonated with a velocity of 8000m/sec when initiated with No 8 cap, and only with vel 500 to 2000m/sec when initiated with No 6 cap. These expls with and without minor proportions of perchlorates have impact sensitiveness of 45–85 kg-cm, vs 2kg-cm for NG]

449) J.J. Minnick, USP 3419444(1968) & CA **70**, 79659y(1969) [Thickened slurry AN-Nitroparaffin expl compns sensitized with an air-entrapping material, suitable in small wet or dry borehole blasting operations are described. Thus, a blasting agent having a deton rate of 19650ft/sec was prepd by mixing at RT $MeNO_3$ 17.0 with NC 1.0 until a gel was obtd and then stirring in AN 60.0, guar gum (thickener) 0.6, urea-HCHO resin microballoons (air-entrapping material) 2.0 & water 19.4% until homogeneity was obtd]

450) R.J. Anderson & J.F. Short, CanP 799408(1968) & CA **70**, 79652r(1969) [Water-resistant pelletized blasting compns comprising prills of TNT coated with an oxidant component in a binder are described. The oxidant was a 1:1 mixt of $NaNO_3$ & AN and comprised 50–75% by wt of the compns; the binder was a mixt of Ca lignosulfonate and poly-(ethylene oxide) and comprised 2–10% of the compns. If desired, up to 20% metallic fuel could be included. The final pellets had a diam of 0.25–0.625 in. and a d 1.60g/cc. They were stable in moist atm for up to 6 days]

451) R. Travers & N.M. Jardine, S.AfricanP 66 04953(1968)& CA **70**, 21464q(1969) [Expls composed of nonexplosive ingredients are described. They comprised 50–90% inorg oxidizing salts (such as AN & its mixts with $NaNO_3$), 3–12% water-sol fuel (such as urea, sugar or molasses), 3–7% water, and up to 20% metal fuels (such as Al, Mg, Si, B, ferrosilicon and their mixts). A water-insol fuel (such as S) could be included, and in case up to 2% of a thickener (guar gum) was used, up to 0.5% crosslinking agent and up to 0.1% of a crosslinking retardant were included. For example, the mixt of AN (<16-mesh Brit Std) 71.4, $NaNO_3$ 4.0, water 6.0, molasses 7.0, coarse Al powd 10.0, guar gum 0.5, ethyleneglycol 0.9, Zn chromate 0.2 and tartaric acid 0.01 part was a blasting compn detonated by 20g of Pentolite with velocity 4000m/sec in a 4-inch diam charge]

452) C.J.N. Kelly & R.F. Knott, CanadP 796160(1968) & CA **70**, 30545u(1969) [Aqueous slurry expls that can be pumped, stable in storage, sensitive to initiation in small-diam chges, and do not contain self-expl ingredients are described. They are mixts of inorg nitrates (NH_4, Na, K, Ba, or Ca) 50–88, water 4–20, carbonaceous fuel (such as Diesel oil) 1–20, a chlorinated org compd (C_2HCl_3, CCl_4, $CHCl_3$, C_2Cl_4, or C_2Cl_6) 1–10, a gas-bubble stabilizer (such as NH_4 lignosulfonate) 0.3–10, and a thickener (such as guar gum) 0.2–5 parts. The carbonaceous fuel could be partially or wholly replaced by up to 30% finely divided Al]

453) SandorDeli, BányászKutIntez, Kozlem **1968**, 12(3), 93–101 (Hung) & CA **70**, 69729g (1969) [Incorporation of powdered limestone in Dynamite-type expls (instead of NaCl or AN) increased their disruptive force and water-resistance during storage. Different types of limestones were compared, the optimal type having a minimum of 85% $CaCO_3$, and a maximum of 6% SiO_2]

454) V. Rank et al, SAfricanP 67 00348(1968) & CA **70**, 69760k(1969) (Blasting expls of high deton rate can be prepd from AN of open porosity of ca 10% by wt or greater and only an adequate amt of Diesel or spindle oil to provide a desired O balance of 5.5–6%. AN of desired porosity was prepd by grinding coml product, followed by screening out the fraction having a particle size of 0.05–0.125mm. This fraction was compressed into a mass having porosity of ≧10%, then broken up and the particles of 0.5–2-mm sizes screened out. These particles were impregnated with the oil filler to the desired O balance)

455) G.M. Lukszewski, AustralianInstMining-MetProc **1968**, No 228, 61-70(Eng) & CA **70**, 98402a(1969) (Reactions of ANFO expl with mineral sulfides were investigated. It was found that ANFO expls were unstable in hot sulfide-rich ore body matrixes. When a sulfide-contaminated dry ANFO was heated to 125–140° a very violent exothermic reaction took place, but reactivity was increased on incorporating 1–2% water & traces of SO_2)

456) G.A. Teichmann, SAfricanP 67 00518 (1968) & CA **70**, 69758r(1969) [Flexible explosive tape for seismic prospecting is prepd from a train of individual flat chges (circular or polygonal discs of PETN or Tetryl) sandwiched betwn 2 long strips of flexible polymeric material and reinforced wire or yarn. Segments of the same expl, placed in the sandwich between each disc, or detong fuse cord placed external to the tape assembly, can be used to couple the discs. When used for seismic prospecting, the tape is unrolled from a spool, placed flat on the ground, and detonated]

457) G.A. Teichmann, SAfricanP 67 00519 (1968) & CA **70**, 69759s(1969) (Explosive charges for seismic prospecting: A plastic

expl consisting of PETN 70, DNT 23 & NC 7% is contained in cartridges. Initiating lines of detonating cord are threaded thru the cartridges in numerous geometric designs. When the cord is initiated by a detonator, detonation waves proceed in the chge where it is threaded, and collision of the advancing waves causes an extremely intense shock wave transverse to the direction of deton which is advantageous in seismic prospecting either subterranean or submarine)

458) G.A. Wetterholm & B.P. Enoksson, SAfricanP 68 01174(1968) & CA **70**, 69766s (1969) (Expl charges of low brisance for smooth blasting, contg NG and/or NGc 33–66, kieselguhr 66–33 & NC 0–4%, are packed into thin-walled plastic or paper tubes at one end of which are a number of tongues. When such tubes are joined together, the tongues are directed outward to engage with the walls of the borehole. On firing, these chges direct crack formation betw adjacent boreholes to give smooth rock faces)

459) J. Vacek & A. Skrivanek, CzechP 127990(1968) & CA **70**, 107929g(1969) [Cryst high-brisance expls (such as Pentaerythritol Trinitrate) 60 parts was homogenized with 40 ps of an aq dispersion contg 45% poly(vinyl acetate), 9% DBuPh & 46% water and the resulting paste dried at 70° to give a solid of d 0.92. Its deton vel was 5200m/sec at a chge diam of 28mm. Similar chges were prepd using RDX or Trimethylenetrinitramine in lieu of PE Trinitrate]

460) H. Kimura & H. Tamada, JapanP **68** 19239(1968) & CA **70**, 59435u(1969) [Non-water-resistant slurry expls were changed to water-resistant by addn of carbohydrate syrup. In an example, the water permeation of an expl obtd by mixing AN 55, TNT 25, 3.2% aq soln of CM-cellulose 15 and viscous liq (prepd by mixing with heating 70 parts glucose & 30 ps of water) 5% was only 4mm and even after being covered with w for 24 hrs its expl props were not changed. Starch syrup was also used]

461) Heinz Dehn, BritP 1129777(1968) & CA **70**, 49164r(1969) [Expl compns are described which contain one or more hydrated salts of an inorg O-rich acid (such as Al perchlorate), in which the water of crystn is at least partially replaced by an alkyl sulfoxide (such as Me_2SO)]

462) W.C. Thomison & Th.E. Slykhouse, SAfricanP 68 01234(1968) & CA **70**, 49165s (1969) [Slurry expl compns capable of complete propagation when detonated under pressure (as high as 70kg/cm^2) are described. They contd hollow glass particles ranging in size from 10 to 250μ in a quantity sufficient to provide a d of 1.0 to 1.5g/cc to expl mixts contg AN 10–85, $NaNO_3$ 0–60, water 5–25, gum arabic 0.2–2.5, Al (30–150 mesh particulate) 5–40, and a carbonaceous non-expl fuel (such as C powd or org liqs) $\leqq$20%]

463) D.C. Ascani (Eastern Lab, E.I. duPont de Nemours & Co, Gibbstown, N.J.) AdvanChemSer **1968**, No 78, 565–80(Eng) & CA **70**, 39411e(1969) (Literature on Explosives)

464) S. Deli et al, HungP 155164(1968) & CA **70**, 39435r(1969) (Permissible expls of Dynamite-type contg as cooling agents inorg salts with F content >50% are described. E.g: AN 73.52, TNT 11.47, DNT 2.03, Na_2SiF_6 12.70 and wood dust 0.28%)

465) E.M. Atadan & Ch.H. Noren, SAfricanP 68 02828(1968) & CA **70**, 116784h(1969) (Improvement of Blasting Explosives was achieved by inclusion of 5–60% by vol of small, gas-filled cavities or bubbles of air, which were uniformly dispersed during the prepn, or by the use of a closed-cell rigid foam or cellular low-d fuel. This method permits formulation of compns that are detonable in columns as small as 6 inches, at temps as low as 40°F without incorporating HE's or metallic sensitizers. The gas can be chemically generated in situ by reaction of carbonates or bicarbonates with acidic AN solns)

466) Ph.W. Fearnow, SAfricanP 68 02829 (1968) & CA **70**, 116786k(1969) (Water-contg expl compns consisting of an inorg oxidizing salt, a fuel, a thickener, and a crosslinking agent can be improved by incorporation of a sol proteinaceous material. This makes the expl soft, pliable, and consistently sensitive to deton at low temps. No example of formulation is given in CA)

467) R.J. Mouton, BelgP 714955 (1968) & CA **70**, 116782f(1969) [A stable, easily transportable mixt suitable as a mining expl or a flare proplnt consists of an intimate mixt of finely divided Mg and S in a 1.25–1.33:1 Mg-S ratio. Densities of Mg & S should be substantially identical to assure lack of segregation and, preferably, are bound together with water-sol silicates. Thus, pure Mg powd (d ~0.60) 225.33g and pure S powd (d ~0.54) 174.67g are mixed thoroughly with 190cc aq Na silicate (d 1.37) and the resulting slurry coarse-screened, air-dried for several hrs and then at 80°. Finally the product was screened to obtain particles of 250–707μ]

468) Explosives and Chemical Products Ltd, FrP 1506335(1968) & CA **70**, 21465r(1969) [Ammonium nitrate firedamp-safe expls were prepd by addn of finely ground flame-coolants (such as NaCl, $BaSO_4$, $NaHCO_3$ or metal salts of oxalic or formic acids) to AN-fuel oil mixts in which AN had a sp surface of >70 (preferably ca 2000) cm^2/g]

469) Yu.S. Mets & I.T. Kolesnichenko, GornyiZh **1968**, 144(9), 37–40(1968) (Russ) & CA **70**, 21451h(1969) (Formation of noxious gases during the expln of various Russ plastic and granular expls in an underground mine gallery, was investigated. The amts of CO & NO_2 formed depended upon the compns of expls and conditions of loading)

470) L. Moeling & W. Hofmeister, KaliSteinsalz **1968**(5), 68–80(1968) & CA **70**, 21450g (1969) (Formation of gases formed by various Ger expls used in potash mines)

471) Nobel-Bozel, FrP 1510904(1968) & CA **70**, 69761m(1969) [Improved NG-sensitized, water contg, gelled, or partially gelled expl slurries were provided by incorporation of 3–7% <50μ Zr silicate. Thus, a gelled slurry contg NG 25, DNT 4, NC 1, AN 43, Na nitrate 7.5, charcoal 5, guar gum 0.5, water 12 & Zr silicate 2% had d 1.45, a crit diam 50mm, when fired with the "minimal" booster of 50g **Gomme A** (see Note below) proved to be satisfactory. The "minimal" booster was that amt of Gomme A required to cause deton propagation betw 2 cartridges 0.6-m long and 50-mm diam. The water-resistance of slurry was satisfactory, as judged by the following test. A crit diam plastic tube was filled to a depth of 0.6m with above slurry and a column of water 0.6-m high placed on top of it. After 1 hr without mixing, the slurry was fired satisfactorily with 50g Gomme A booster. A similar gelled slurry in which the 2% Zr silicate was replaced with AN had a d of 1.44, a critical diam of 80mm and required minimal booster of 300g of Gomme A. Its water test was unsatisfactory]

Note: Gomme A is one of the French NG based, gelatinous expls, known as "gommes". It is called "gum A" in CA which is misleading because it is not a gum, but a hard, gelatinous expl contg NG 92–94 & collodion cotton 8–6%. It corresponds to Ger "Sprenggelatine" or Amer "Blasting Gelatin". A list of numerous "gommes" is given in the book of Pepin Lehalleur(1935), pp334–35

472) Fernando S.S. Sobral, Tecnica(Lisbon) **1968**, 43(377), 431–46 (Port) & CA **70**, 13113j(1969) (A review with 28 refs of props and methods of application of AN-FO expls)

473) R.W. VanDolah, C.M. Mason & D.R. Forshey, "Development of Slurry Explosives for Use in Potentially Flammable Gas Atmospheres", USBurMinesReptInvest **7195**, 9pp (1968) & CA **70**, 13126r(1969) [In this rept expts with 23AN-based slurry expls showed that it is possible to have them cap-sensitive and relatively nonincendive. All slurries that used flaked Al were sensitive to No 6 EBC (electric blasting cap) except one contg AN 47.2, NaCl 10, water 30, Al (particle size 12μ) 8, sugar 3.5, guar gum 1.0 & buffer 0.3%. Buffer (consisting of NaOH & KH_2PO_4 in 1:29 ratio) was added to hold the pH at 4.5–5.5]

474) L.V. Dubnov & I.T. Kolesnichenko, GornyiZh **1968**, 143(5), 56–9(Russ) & CA **70**, 13128t(1969) (Energy-producing criterion of the effectiveness of expls and some of their effects. Methods are described for comparing various Russian industrial expls based on simple relationships involving energy release, cost, etc. Tables giving data on these expls made accdg to algebraic criteria are shown, and the relation of application of individual expl types to these criteria is discussed)

475) G.P. Demidyuk & B.D. Rossi, VzryvnoyeDelo **1968**, (65/22), 5–15 (Russ) & CA **70**, 116755z(1969) [Development of Russ industrial expls, such as **Dinamony** (Dynammons) (approved by the Govt in 1934) and **Igdanity** (Igdanites), developed in 1959, and included in the official list of nonpermissible expls in 1966] (See also in this Vol under Dinamony and under Igdanity)

476) B.D. Rossi et al, Ibid **1968**, (65/22), 15–22 (Russ); CA **70**, 116769g(1969) (Laboratory studies of the amount of noxious bases formed during the expln of **Igdanit**)

477) S.A. Mel'nikova, Ibid, **1968**, (65/22), 22–4 (Russ) & CA **70**, 116761y(1969) (Effect of surface active agents on the physicochemical props of **Igdanit**)

478) A.B. Plekhanova et al, GornyiZh **1968**, 144(9), 40–2 (Russ) & CA **70**, 30538u(1969) (Testing of **Igdanit** at a northern Ural bauxite mine, showed that on its expln the formation of toxic gases CO & NO_2 was lower than from expln of **Ammonit 6ZhV**. Compns of these expls are not given in the above CA, but are listed later in this Vol)

479) V.A. Usachev, VzryvnoyeDelo **1968**, (65/2) 24–30 & CA **71**, 31927c(1969) [Effect of degree of dispersion and specific surface of AN on the critical diameter of **Igdanit** (qv)]

480) E.P. Maksimova & A.N. Bugaiskii, Ibid, **1968**, (65/22), 30–41 & CA **71**, 31933b(1969) (Conditions for the safe use of **Igdanit**)

481) M.F. Koshamov, Ibid **1968**, (65/22), 41–5 & CA **71**, 31932a(1969) (Escape of Diesel oil into the atmosphere during the pneumatic loading of blast holes with **Igdanit**)

482) A.M. Faizullin et al, Ibid, **1968**, (65/22), 63–9 & CA **71**, 31934c(1969) (Mechanization of the charging of blast holes and boreholes with **Igdanit**)

483) A.V. Brichkin et al, Ibid, **1968**, (65/22), 75–92 & CA **71**, 31940b(1969) (Aerosol process for charging loose expls, such as **Igdanit**, into boreholes is claimed to be more convenient, safe and rapid than pneumatic charging)

484) M.F. Koshamov, Ibid, **1968**, (65/22), 92–6 & CA **71**, 31946h(1969) (Density of the charging of blast holes with **Igdanit** by means of an ejector pneumatic charger PPZh-2)

485) N.F. Adrianov et al, Ibid, **1968**, (65/22), 109–27 & CA **71**, 31926b(1969) (Testing of **Igdanit** at various mining installations to determine its economic effectiveness, amt of toxic gases produced, etc, in comparison with other Russian expls, showed that detonation energy of Igdanit was similar to those of **Ammonit 6ZhV**, **Detonit 10A**, **Detonit 6A** and **Dinaftalit**, but the amt of toxic gases produced on expln was smaller)

486) E.G. Legastaev et al, Ibid **1968**, (65/22), 130–34 & CA **71**, 40906x(1969) (**Igdanit** 96:4 was more effective than **Ammonit No 6** during the breaking down of ore by borehole charges in the "Kaz" mine)

487) B.P. Poduzov et al, Ibid **1968**, (65/22), 139–42 & CA **71**, 31930y(1969) (Results of industrial testing of **Igdanit**, contg AN 94.75 & Diesel fuel 5.25%, in the sinking of shafts at the Salairskii mine are given and compared with those for **Ammonit 6ZhV** and **Detonit 10A**)

488) D.G. Zil'berg et al, Ibid **1968**, (65/22), 142–45 & CA **71**, 31931z(1969) (Results of the pilot-plant testing of **Igdanit** in mines of the Tekliiskii Combine and Kazakh Gold Trust, showed that amt of toxic gases and concn of Diesel fuel in the air after expln, were below allowable limits)

489) I.L. Zabudkin et al, Ibid **1968**, (65/22), 145–52 & CA **71**, 31908x(1969) (Use of **Igdanit** for blasting rocks in mines of Achpolimetall Combine)

490) V.S. Romanov & A.I. Basystyi, Ibid **1968**, (65/22), 152–55 & CA **71**, 31928d (1969) (Result of testing **Igdanit** in underground mining)

491) L.A. Mamsurov et al, Ibid **1968**, (65/22), 162–68(1969) & CA **71**, 31939h(1969) (Use of **Igdanit** contg 5.5–5.8% Diesel oil in blast holes charges)

492) L.A. Mamsurov et al, Ibid **1968**, (65/22), 182–87 & CA **71**, 31924z(1969) (Effectiveness of **Igdanit** for clean removal of ore veins was higher than that of **Detonit**)

493) Ya.M. Mansurov & I.G. Ushakov, Ibid **1968**, (65/22), 187–95 & CA **71**, 31938g (1969) (Large-scale testing of **Igdanit** in flint, limestone and granite showed that it was more economical to use than **Detonit, Ammonit No 6** and **Skal'nyi** (Rock) **Ammonit**)

494) G.A. Berishvili & V.D. Gabidzashvili, VzryvnoyeDelo **1968**, (65/22), 201–05 & CA **71**, 31937f(1969) (Effectiveness of **Igdanit** in underground cutting of manganese ore was greater than that of **Ammonit No 6**)

495) O.D. Karbelashvili et al, Ibid **1968**, (65/22), 210–15 & CA **71**, 31925a(1969) (Testing of **Igdanit** in cleaning faces of the Chordskii barite deposit showed that it was more economical to use than **Ammonit No 6**)

496) Ya.N. Mansurov, Ibid **1968**, (65/22), 215–22 & CA **71**, 31935d(1969) (Role of stemming during **Igdanit** blasting)

497) G.P. Licheli et al, Ibid **1968**, (65/22), 222–25 & CA **71**, 31929e(1969) (Effect of tamping on the destruction of teschenite blocks by **Ignadit**)

498) V.P. Tambellini, Ibid **1968**, (65/22), 228–35 & CA **71**, 31936e(1969) [Testing of **Igdanit** for use in external charges showed that it can be used effectively for blasting in limestone, rubblestone layer (max 70cm thickness), and reinforced concrete (300–1000cm^2 area)]

499) B.N. Kukib, Ibid **1968** (65/22), 269–76 & CA **71**, 31945g(1969) [Comparison of effectiveness in blasting of several Russian expls, showed that **Anfex** (expl contg porous granulated AN & Diesel oil) is superior to **BA-62** (expl contg nonporous AN & Diesel oil) and to some other mining expls]

500) R W. Coxon, AustralianP 281537(1968) & CA **71**, 5031m(1969) (AN-FO expls contg an aq soln of an anionic surfactant to give better contact between the constuents are described. The surfactant proposed was a Na, NH_4, or org base salt of a sulfonate of an aromatic hydrocarbon which was normally sol in water but not in fuel oil. This addn increased the sensitivity of AN-FO without an increase in the hazards of its use)

501) P.R. Goffart (of Belgium), FrP 1533471 (1968) & CA **71**, 23404q(1969) [Conventional safety expls for use in the presence of coal dust become resistant to aging and at the same time better resistant to water by the simultaneous incorporation of metal soaps (such as described in FrP 1189396) and galactomannose gum]

502) DuPont & Co, FrP 1537625(1968) & CA **72**, 45652s(1970) [Slurried AN expls thickened with galactomannan are described. Thus, 50 parts of 65% aq AN was placed in a mixer at 66–77°, and 30ps TNT (in 3.36–2.38mm diam pellets) added with stirring. Hydrocarbon oil (113g) was added to 50.8kg of the above mixt, followed by a compn contg 91g guar gum, 227g of polyacrylamide, and 20 parts $NaNO_3$ based on the expl compn. Then 907g $HCONH_2$ was added and after stirring for 3.5 mins, 25g $K_2Cr_2O_7$ (in 5% soln) added over 15 secs and 100g of a 5% soln of K antimony tartrate over 30 secs. The final compn, d 1.4, was extruded into polyethylene bags as a viscous gel. It remained stable for >12 weeks at –6.6° or 110°C]

503) P.R. Goffart (Belg), FrP 1540664(1968) & CA **71**, 83182a(1969) (A permissible expl, classified as Ger Class III or Belg Class 4, was prepd from NG, plus NGc 9.5, KNO_3 55.25, NH_4Cl 28, NH_4 oxalate 5 & waterproofing additives 2.25 parts. Other formulations are listed)

504) Hirtenberger Patronen-, Zundhutchen and Metallwarenfabrik AG, FrP 1540861 (1968) & CA **71**, 83183b(1969) (Expl compns contg microcryst AN are described. Such AN was obtd by its crystn from a concd or satd soln contg 0.4–1.0% of the acetate of triethanolamine or dimethylethanolamine, 2-amino-2-methylpropyl lactate, or triethanolamine in addn to 0.2–1.0% of the acetate of dodecylamine, the lactate of tetradecylamine or dodecylamine used in prior formulation. Expls prepd by mixing fuel oil with

such AN were more powerful than those prepd with the ordinary AN)

505) M. Barbaste, FrP 1541657 (1968) & CA **71**, 51826x (1969) [Improved AN-FO expls were prepd by substituting for 50–80% of the fuel a cyclic solid hydrocarbon (such as naphthalene). For example, a mixt contg 94.3% prilled AN (1.2mm diam; 0.14% water; oil retentiveness 4%), 2.8% gas oil, and 2.9% naphthalene (0.1mm) was more powerful than the mixt contg 94.4% AN & 5.6% oil]

506) DuPont & Co, FrP 1544937 (1968) & CA **71**, 103745s (1969) [AN expls are described which comprise a C-contg combustible (such as sugar), a thickening agent (such as guar gum), a crosslinking agent for the gum (such as $K_2Cr_2O_7$ soln), a gas-encapsulating material (such as marrow of sugar cane), and an anionic surfactant as crystal-modifying agent (such as Na methylnaphthalenesulfonate). For example, an expl was prepd by mixing at 65° in a rotating blender the following ingredients: AN 56, water 14, $NaNO_3$ 15, sugar 8, sugar cane marrow 3, Na methylnaphthalenesulfonate 1, stearic acid 3, and guar gum 1% by wt. The gum crosslinking agent (5% aq $K_2Cr_2O_7$ soln) was added last in an amt of 450ml/50kg. The d of the expl was 1.2 and deton rate in 15-cm diam cylinders (initiated by conventional caps) was 4500m/sec at 32° and 4150m/sec at 4°]

507) Schweiz Sprengstoff-Fabrik AG, FrP 1549958 (1968) & CA **72**, 68851v (1970) (Polystyrene foam beads were used as fillers for regulating the charge density of expls used for blasting purposes)

508) Commercials Solvents Corp, FrP 1550911 (1968) & CA **71**, 93220a (1969) [Expl slurries comprising AN, methanol, water, urea nitrate, guar gum or a resin UCAR C-149 or WSR-131 are described. For example, a mixt of AN 67.4, methanol 20.0, water 2.0, urea nitrate 10.0 & WSR-301 resin 0.6 parts was prepd and poured into borehole (200mm diam) in rock. With 0.5kg Pentolite primer per 2.4m long borehole, complete deton and an excellent shattering effect on rock were obtd]

509) R.G. Hall, SAfricanP 68 03385 (1968) & CA **71**, 40929g (1969) [Nitrate-fuel-binder expl compns are described which were prepd from mixts of K, Ba, or org (aliphatic or guanidine) nitrate, a fuel (C, S, sugar, a combustible metal powder, or mixts), and a resinous binder. The binder consisted of a liq polymerizable resin dissolved in an org solvent. A polymerization catalyst or accelerator for the resin was also included]

510) K. Shiino & S. Oinuma, KôgyôKayaku-Kyôkaishi **1968**(5), 347–52 (Japan) & CA **71**, 103734n (1969) [**A compn** is described, contg sym-Dinitrodimethylurea 45, NC 5 & AN 50%, as an excellent expl having the the following props: impact sensitivity (max nonexpln height of 5kg falling hammer) 58cm, heat of expln 1185cal/kg, gas vol 901 l/kg and ballistic mortar value 148 (TNT 100) Also are given props of sym-Dinitrodimethylurea]

511) Ph.W. Fearnow, SAfricanP 68 03478 (1968) & CA **71**, 23401m (1969) [Aqueous expl compns of d 1.0–1.5 contg no self-explosive or metallic sensitizers, yet capable of reliable detonability at low temps, comprise 40–75% inorg oxidizer (such as a nitrate or perchlorate of NH_4, Na, K, Mg, etc or their mixts), 2–40% partially nitrated aromatic sensitizers (such as MNT, DNT, $PhNO_2$, DNB, MNX, DNX, or their blends), 1–30% of a C_{1-4} lower aliphatic monoamide sensitizer dispersant (such as $HCONMe_2$, $AcNMe_2$, propionamide, or acrylamide), 0.2–5% thickener (such as guar gum, quince seed, gums, gelatin, casein, agar, etc) with a crosslinking agent (such as $KSbC_4H_4O_7 \cdot \frac{1}{2}H_2O$), 5–30% water, 2–20% nonexpl carbonaceous fuel (such as carbon, coal, sugar, wood & paper pulp, etc), and $\geqq$ 0.01% of a sol proteinaceous material (such as egg albumin). A detailed description is given of prepn of a mixture which had deton rate of 5100m/sec at 56°F, in 5-inch diam unconfined polyethylene container]

512) M. Novotny et al, SbVedPr, VysSk-Chemickotechnol, Pardubice **1968**, (17) (Pt I), 115–22 (Czech) & CA **71**, 31944f (1969) [Safety measures were made during work in coal mines with charges of expl **STV-40 (Semtinit TV-40)**, which consists of

AN 22.6, nitrate esters 34.9, barites 12, NaCl 27.5, DNT 20, & wood flour 1.0%. The expl has a plastic consistency and can be drawn into charges several meters long. Each chge was wrapped in an inner, thin-walled polyethylene tube and an outer wrapping of vitreous fabric. In use, the desired length of chge was cut from the roll. Since std safety practice has not permitted cutting of chges in operating mines, the exptl work was undertaken to det the handling safety of STV-40 under conditions requiring cutting, and tamping. Tests were conducted for impact, friction, compression, and of special cutting operations. It was found that cutting was safe when conducted with proper equipment and if gritty particles were absent in expl compn]

512a) S. Barska (Bulgarian), GodNauchnoizsledProektInstRudodobivObagat, Rudodobiv **1968**, 7(7), 22–34(Bulg) & CA **73**, 37080j (1970) [Factors affecting the amt and compn of noxious gases during blasting were detd. Accdg to results obtd in Donbas (Russia), the amts of CO involved in blasting with **Ammonit** and **Dinaftalit** varied within the limits 10–40, 40–100, and 100–150 dm^3/kg explosive, depending on the rocks that are blasted, which can be subdivided into three classes on this basis]

513) CanadIndsLtd, BritP 1138920(1969) & CA **70**, 69770p(1969) [Improved meltable expl compns consisting of an inorg O-supplying nitrate salt (or salts), any sol salt of ligninsulfonic acid and a fuel are described. A typical compn consists of 50–90% AN, up to 20% fuel, and 0.5–30% of a sol lignosulfonate. The compn is first prepd by melting the oxidizer, mixed with an optional mp depressant, then the fuel and lignosulfonate are added and the melt cast into a mold where it is allowed to set]

514) NipponKayakuCoLtd, BritP 1141185 (1969) & CA **70**, 89338g(1969) {Gelatinized Dynamites of low NG content are provided by mixts of AN 66–82, DNT and/or DNX 2–8, starch (or other inert filler) 3.2–5.2, NG 8–16, NC 0.4–1.4, an auxiliary plasticizer [comprising a mixt of substantially equal parts of poly(vinylbutyral) resin dissolved in castor oil, or di-Bu(or octyl-) phthalate] 0.1–2 and poly(oxyethylene) stearate [contg 0–50% poly(oxyethylene)-nonylphenyl ether] 0.1–2 parts. For example, the Dynamite prepd by kneading a mixt of AN 75.8, DNT 4.5, nitrogel consisting of NG 14.0, NC 0.7 & auxiliary plasticizer 1.0 part had d 1.25, brisance by Hess Method 14.5mm compression of Pb-block and was water-resistant, nonextrudable and did not lose its plasticity after 3-months storage. Its auxiliary plasticizer was prepd by dissolving a poly(vinylbutyral) resin 5 in castor oil 45 (with agitation at 60°) and stirring in a 75–25 poly(oxyethylene)nonylphenyl ether mixt 50 parts. A similar Dynamite but contg no above auxiliary plasticizer had unsatisfactory water-resistance & extrudability, low brisance and lost its plasticity during storage}

515) R.A. Simpson et al, USP 3423257(1969) & CA **70**, 79657w(1969) {Stable gelled blasting compns contg nitric acid and a "carboxacylic" fuel (such as acetic, propionic, trichloroacetic, nitropropionic, benzoic or azelaic acid; or acetaldehyde) are described. On the basis of 100 parts by wt of anhyd HNO_3, the compn contd: carboxacylic fuel 36–159, a nitrofuel (such as DNT) 0–53, AN 0–745, a gelling agent (such as Gantrez AN–169 resin) 7–37, a crosslinking agent [$SnCl_2$ or $Cr(NO_3)_2$] 3–11, and water 13–230 parts. Small amts of materials to modify density (such as tiny hollow glass balls, or sorbitan monopalmitate) could be added. For example, an expl compn prepd by mixing anhyd HNO_3 13.8, AcOH 10.9, DNT 7.3, AN 64.1, Gantrez AN–169 resin 1.0, $SnCl_2$ 1.0 & water 1.9% had d 1.40 & deton rate (when confined in a 1.5-in steel pipe) 5150 m/sec}

516) G.A. Gross, USP 3423258(1969) & CA **70**, 79658x(1969) [Gelled blasting compns are described which were produced by initially mixing a gelling agent (such as Gantrez AN–169) with either the HNO_3 or "carboxacylic" fuel (AcOH) component before bringing the gelling agent in contact with the crosslinking agent (Cr chromate). E.g: HNO_3 11.8–14.0, AN 70.0–71.5, Gantrez AN–169 10.0, $Cr(NO_3)_2$ 0.5–5.0, AcOH 13.0–13.2 and sorbitan monopalmitate (to modify density) 0.2–5.0 parts]

517) C.J.N. Kelly & R.F. Knott, USP 3431154 (1969) & CA **72**, 4845p(1970) (Correction of CA **71**, 116783g) (Pourable aq slurry expl compns are described, composed of an inorg O-supplying salt, water, thickener & fuel. E.g: granular AN 79.7, Diesel oil 2.4, CCl_4 8.0, water 7.0, guar gum thickener 0.5, ethyleneglycol dispersant 1.0 & NH_4-lignosulfonate stabilizer 1.4%)

518) C. Dunglinson & W.M. Lyerly, USP 3431155(1969) & CA **70**, 116788n(1969) [High-perfomance, water-contg expl compns having densities ca 1.4 were obtd by incorporating in conventional oxidizer fuel-water mixts a sensitizer contg a salt of an inorg oxidizing acid (such as HNO_3, $HClO_3$ or $HClO_4$) and a base (such as $H_2N.NH_2$, $MeNH_2$, Me_2NH, Me_3N, urea, guanidine, aniline, etc). The preferred salts being $MeNH_2.HNO_3$, $Me_3N.HNO_3$ and HNO_3. $H_2NC_2H_4NH_2.HNO_3$. Thus, a mixt of AN 35, water 15 & $MeNH_2.HNO_3$ 30 parts was heated with stirring to 54–60°, and a premix of $NaNO_3$ 15 & guar gum 0.8 part stirred in until thickening was observed (ca 3.5 mins). Then sulfur 2, PhOH–HCHO resin microballoons 1, and carbon powd 2 parts were incorporated and finally ca 1% $K_2Cr_2O_7$ soln 0.1 and 5% KSb tartrate 0.07 part were added, the mixt stirred for several mins, and poured into 5-inch diam cartridges. Its density was 1.35 and deton rate 6300m/sec]

519) M.T. Abegg et al, Explosivstoffe **17**(2), 25–31(1969) & CA **71**, 31919b(1969) [Low-detonation pressure plastic expl compns studied consisted of polyurethane (PU), nitropolyurethane (NPU), and dinitropropyl acrylate plastics to which various expls (PETN, RDX, etc) were added]

520) Ch.H. Grant, USP 3432371(1969) & CA **71**, 23398r(1969) [Dry expl compns contg essentially (by wt) 50–97% particulate AN, 3–50% particulate light metal (Al, Mg, or their alloys, or mixts thereof), ranging from No 4 to No 200 mesh (US Std Sieve) and having a gauge from 0.25 to 40 mils; up to 40% inorg oxidizing salt (particulate alkali metal nitrate, chlorate, perchlorate, or mixts thereof); up to 10% nondetonable fuel (liq hydrocarbons); and up to 2.5% gelling or thickening agent (natural gums or synthetic polymeric materials)]

521) W.S. McEwen & E.W. LaRocca, USP 3437534(1969) & CA **71**, 40926d(1969) [Underwater expl compns contg Al, $KClO_4$ and S or P (no example of compn is given in CA) were sensitive to impact and ignition, their heat of expln and energy were high, but brisance and deton rate low]

522) E. Ludolphy & A. Berthmann, USP 3438823(1969) & CA **70**, 116785j(1969) (Blasting expls were improved by incorporating a wetting agent, such as 2-naphthyloxyacetic acid laurylamide, 2-naphthylstearylamine, or Na benzenesulfonate. The wetting agent renders the water-sol components more readily wettable by the liq nitrate esters, which are also more effectively dispersed. As a result, the sensitiveness of the expl is not diminished when it is used in wet condition. A typical expl formulation of this application is: AN and/or KNO_3 and/or NH_4Cl with liquid nitrate ester 3–20 & wetting agent 0.001–1%)

523) B.P. Enoksson (of Sweden), BritP 1147967(1969) & CA **71**, 23402n(1969) (AN with water-resistant and anticaking props was obtd by coating its particles with 0.05–0.1% of a mixt contg 2–10 moles of an alkylamine and 1 mole of an aliphatic acid. The acid and amine contained 12–20 carbon atoms. Stearylamine and stearic acid were used in one example)

524) DuPont & Co, BritP 1154430(1969) & CA **71**, 40927e(1969) [Water-bearing AN-fuel expls, contg 0.1–5% of an aliphatic acrylic amide crosslinked with a C_{1-5} aliphatic aldehyde as a polymeric gelling agent, are described. E.g: AN 41.77, water 22.50, acrylamide 2.06, $(NH_4)_2S_2O_8$ 0.98, HNO_3 (75%) 0.32, $NaNO_3$ 12.86, TNT 19.28, and HCHO (37% concn) 0.37 part. The deton rate was 2890m/sec]

525) ICIA&NZLtd, BritP 1156596(1969) & CA **71**, 72520h(1969) [A slurried expl compn contg no liquid self-expl sensitizer, but

comprising ≧ 1 inorg oxidizing salt (selected from nitrates, chlorates, perchlorates or their mixts), ≧ 1 liq fuel (water-sol or insol), ≧ 1 insol combustible material in particulate form and water]

526) Th.P. Rudy & T.W. Nakagawa, USP 3442726(1969) & CA **71**, 31965p(1969) (Particulate nitronium perchlorate coated with a complex pyridine salt, suitable as a solid compatible oxidizer in various formulations, is described)

527) J.R. Thornton, USP 3442727(1969) & CA **71**, 31967r(1969) (The storage stability and reliability of blasting compns can be improved by the inclusion of a mono- or diester of H_3PO_4 or its salts as the emulsifier for the HNO_3. This eliminates the need to add an acid-resistant gelatin stabilizer. Typical emulsifiers are: lauryl H phosphate, tetradecyl H phosphate, etc. The blasting compn can be prepd either by mixing all of the ingredients, or by mixing a preblend of HNO_3 and oxidizer salt with the fuel & emulsifier)

528) R.A. Simpson, USP 3442728(1969) & CA **71**, 31969t(1969) (Gelled nitric acid blasting agent, such as compn contg 70% aq HNO_3 17, $PhNO_2$ 10, sorbitan monopalmitate 1.5, AN 70, Gantrez AN-169 1.0, and 30% aq $Cr(NO_3)_2$ 0.5% by wt, had d 1.42 and deton rate 3500m/sec in 1.5-inch diam steel pipe)

529) W.M. Lyerly, USP 3445305(1969) & CA **71**, 31973q(1969) (Galactomannan water-bearing expls were improved by increasing the rate at which the galactomannan can be crosslinked by Cr^{3+}. This acceleration could be effected by a sol reducing agent such as K antimonyl tartrate. This process is particularly suitable for use in situ formulation and mixing)

530) H.G. Knight, Jr, USP 3442729(1969) & CA **71**, 14740u(1969) [Aqueous slurries of inorg oxidizer salts thickened with mixts of partially hydrolyzed acrylamide polymers & copolymers and NH_4 or alkali metal acrylates are described. Thus, a compn was prepd from water 17.9, propellant 34.8, AN 21.9, $NaNO_3$ 12.9, ethylene glycol 10.9, pine oil 0.1, 22% hydrolyzed polyacrylamide (viscosity 4000cp) 0.7, Na acrylate copolymer 0.3, $Al(OAc)_2OH$ 0.003 and paraffin oil 0.5 parts. This slurry had sp gr 1.30 at 60°F and a deton rate 4650m/sec at a min diam of 5 inches. The slurry was stable, easily poured and pumped]

531) J.D. Chrisp, USP 3444014(1969) & CA **69**, 51829a(1969) [Expl compns contg nitric acid gelled by in situ crosslinking of an acrylic polymer (preferably polyacrylamide) with a metal ion having a positive valence of 2–6 (selected from Cr, V, Mn, Ti, Sb, Zr, or Sc). Expl compns were provided by the presence of fuels and/or sensitizers in gelled nitric acid to form mixts having O balances of –25 to +10%. Gelled nitric acid could be used as **Sprengel Type** expls, such as prepd by charging 70–99% nitric acid to a mixing vessel, then the acrylamide polymer (Cyanomer P-250, mol wt 5–6 million was added and dissolved with mixing continued at ~25°]

532) Th.E. Stykhouse et al, USP 3446681 (1969) & CA **71**, 31972p(1969) [Gelled slurry expl compns contg 0.1–5% of a ter-polymer gelling agent (comprising 1–60% acrylonitrile and/or methacrylonitrile, which provides a water-insol yet aq AN soln-sol copolymer); 25–98% acrylamide and/or methacrylamide; and 1–15% acrylic and/or methacrylic acid (which later provide carbonyl crosslinking sites). Crosslinking was achieved by adding ~0.1g polyvalent metal salt such as $Cr(NO_3)_3 \cdot 9H_2O$ per 100g slurry. Up to ~0.25% suspending agent such as guar gum or wheat flour could be included. The slurries could also contain (in addn to the conventional inorg oxidizers, expl and/or metallic sensitizers, and water) solvents for inorg oxidizers, and/or liq fuels, kerosene, glycerol, glycol, etc. Thus, a suspension of Guartec UFD 0.25 in 0.3 propyleneglycol was added to a soln of $HCONH_2$ (Fe-free) 10 in H_2O 6 held at 70–76°F, the mixt stirred (~20 min) to obtain a clear gel; the ter-polymer soln 10 (terpolymer 1, AN 5 & water 4) stirred in, crushed $NaNO_3$ 10, prilled AN 53.45 and particulate Al 10 parts, and finally 0.9ml 1M aq $Cr(NO_3)_3$/lb mixt stirred in]

533) H.F. Bluhm, USP 3447978(1969) & CA **71**, 40928f(1969) [Aqueous emulsion blasting

agent, usually free of self-expl ingredients is described, which includes AN, water, NH_4ClO_3 (or NH_4ClO_4), alkali or alk-earth metal nitrates, Al, Zn, ethylenediamine, carbonaceous fuel and microballoons. Thus, an oil-sol cryst wax (Atlantic 342, mp 121–24°F) 2.4, refined mineral oil (Atlantic Atreol 34) 5.6, oleyl acid phosphate emulsifier 3, AN 100, $NaNO_3$ 16, H_2O 29 and microballoons 2 parts are mixed with heating to above the emulsion temp (114°F), the mixt cooled to 114°F, where gas occlusion occurs, and the mixing is discontinued at 110°F. The product is a blasting emulsion of d 1.15, pH 4, occluded air 14.2% by vol (all at 70°F) and a deton rate after storage for 28 days at 70°F of 16400ft/sec]

534) H.F. Bluhm & N.E. Gehring, USP 3447979(1969) & CA **71**, 62687d(1969) [Gelled HNO_3–AN blasting compns were described which included aq HNO_3 solns of an inorg nitrate ($NaNO_3$), a carbonaceous fuel, a polymer stabilizer, and a gelation agent. The stabilizer, gelled in the presence of ferric ions, is aptly selected from a hydrolyzate of a copolymer of a vinyl monomer and an unsatd acid anhydride, or a water-sol acrylic polymer. Thus a preblend of AN 130, aq HNO_3(60%) 60, $NaNO_3$ 10, $MeOCH:CH_2$–maleic anhydride copolymer hydrolyzate 6, and $Fe_2(SO_4)_3$ 1 part was maintained at 43° and mixed with a blend of paraffin 8, sorbitan monopalmitate 5, and mineral oil 1 part, maintained at 32°. During mixing, sufficient air was introduced to yield a compn having a d of ~0.75 at 43°]

535) J.J. Minnick, USP 3447982(1969) & CA **71**, 51827y(1969) [A blasting agent is described which contained AN made by Stengel Process (See Vol 1 of Encycl, p A315-R). It was melted and 3% of diatomaceous earth and 0.4% of urea were added with stirring. The resulting melt was poured on a cold surface to give a 1/8 to 1/4-inch thick cake and then ground after cooling. After removing the fines passing a 20-mesh screen, the larger particles were mixed with 5 wt% of No 2 fuel oil without runoff. AN prepd by this method had good oil absorbency and the resulting AN-FO expl satisfactory blasting performance]

536) R.J. Armantrout & B.C. Person, USP 3449181(1969) & CA **71**, 51825w(1969) (Slurry expls contg a nitrite and sulfamate and/or sulfamic acid aeration agent are described. The agents are prepd in amts of 0.1–1.6% wt % of the compns and consist of $NaNO_2$ in combination with $H_2N.SO_3NH_4$. Thus, an inorg oxidizer salt expl compn was prepd by mixing water 16.1, a propellant 32.1, AN prills 37.6, $NaNO_3$ 9.7, $HO.CH_2.CH_2.OH$ 3.1, pine oil 0.1, guar gum 0.8, cross-linking agent 0.08, $NaNO_2$ 0.055, fumaric acid 0.10 and $H_2N.SO_3NH_4$ 0.11 part. The comp had d 1.17 and deton rate 5400m/sec at 6° in a 5-inch diam pipe when initiated with 140g Pentolite)

537) H.W. Sheeran & M.H. Oriard, USP 3450582(1969) & CA **71**, 51831v(1969) [Aqueous blasting compns contg AN mixed with fuels that are stable in water and capable of maintaining intimate contact with AN to achieve predictable detonations. The fuels may be solids (such as waxes, nitroaromatic compds, etc) or liquids that are water-stabilized by mixing with an ethylene-vinyl acetate copolymer or other plastics that are sol in the heated fuel and that form a stable gel on cooling. The solid fuels are melted and adsorbed onto AN prills during mixing which is continued during cooling. Then water is added and blending continued]

538) H.W. Sheeran & M.H. Oriard, USP 3453155(1969) & CA **71**, 51830u(1969) (Blasting agents contg a coated AN and a hydrocarbon fuel are described, such as for example: AN 84.56, hydrated Ca nitrate 9.35, No 2 Diesel oil 5.50 and Al stearate 0.5 part; its d was 0.884, deton rate 11560 ft/sec, and OB –0.03%. Another example: AN 17.86, hydrated Ca nitrate 71.45, No 2 Diesel oil 5.94, Al stearate 0.59, and petroleum coke 4.17 parts, had a d of 1.120, deton rate 7004ft/sec and OB +0.05%)

539) R.A. Simpson et al, USP 3454438(1969) & CA **71**, 51824v(1969) [Gelled nitric acid agent, such as contg HNO_3 (42°Bé) 14.0, AN 69.5, $MeNO_3$ 13.0, sorbitan monopalmitate 1.5, water-sol $MeOCH:CH_2$–maleic anhydride copolymer Gantrez AN-169 1.0, and

Cr nitrate (39% aq) 1.0 part; its d was 0.98, deton rate 3490m/sec and sensitivity – No 6 blasting cap]

540) R.W. Lawrence, USP 3455750(1969) & CA **71**, 72518p(1969) [Nonaqueous inorg oxidizer salt blasting compns contg a silicon component of particular size are described. They are claimed to be less sensitive than similar Al-contg mixts. Thus, a blasting agent was prepd from 89% (+20-mesh) AN prills, 7% Si (65% <200 mesh) and 4% No 2 fuel oil; its d was 0.81 and deton rate 3550m/sec]

541) M.A. Cook & W.O. Ursenbach, USP 3457127(1969) & CA **71**, 72519q(1969) [Urea-nitric acid addition product expls were obtd by controlling the reaction temp during prepn in such a manner that self-thickening addn products, rather than nitro compds, were formed. The thickened slurry, capable of supporting granular or particular fuels (such as Al, Mg, TNT, coal, or gilsonite) could be pumped or poured into boreholes. For example, a blasting compn was prepd from concd HNO_3 39, urea 13, AN 26, gilsonite 6.5, water (from nitric acid) 15.5 & guar gum 0.05 part, although strongly acid thickened well and detonated with good power. The urea could be replaced by guanidines, semicarbazides, or methylated carbazides]

542) G.L. Griffith & W.I. Schwoyer, USP 3457128(1969) & CA **71**, 72517(1969) [Particulate self-expl nitrated materials (such as NS, PETN, TNT and their mixts) can be made safe to handle and transport thru formation of a uniform nongelled aq slurry contg 10–50% water and 0.05–10% partially hydrolyzed polyacrylamide suspending agent having 0.1–50% free acid and $\geqq$50% unhydrolyzed amide groups and a mol wt of one to 25 million. Such slurries are esp useful for transporting expl sensitizers used in aq slurry blasting agents. Thus, a mixt was prepd by stirring together 66% dry NS, 39.8% water and 0.2% Polyhall 295 (a coml partially hydrolyzed polyacrylamide of mol wt 3 to 8 million contg 15–30% amide groups hydrolyzed to acid groups). This slurry was blended with dry AN 50, flake Al 30, and water 11 parts to produce a blasting explosive]

543) E. Ludolphy et al, USP 3459608(1969) & CA **72**, 14386d(1970) [Water-contg, nitric ester-free, AN-based expl slurry requiring No 9 cap for initiation is provided by mixts such as follows: An expl slurry contg the water-sol ether Tylose MH-50 0.39, water 7.82, epoxylated p-nonylphenol (having an av of 9 EtO radicals/mol) 0.078, castor oil 0.078, triisopropanolamine 5.39, $NaClO_4$ 19.50, $NaNO_3$ (<0.2mm) 0.59, $Ca(NO_3)_2 \cdot 4H_2O$ 4.4, TNT (<0.2mm) 11.34 and AN (<0.2mm) 50.42 parts had O balance –1.4% and Trauzl block value 415cc]

544) D.P. Moore, USP 3463683(1969) & CA **71**, 114823s(1969); Division of USP 3409708 (CA **70**, 21466s) (A solid propellant used as substitute for Dynamite was prepd by mixing 7–20% starch with 4.05–16% vinyl chloride copolymer, heating to 80°, and mixing with 60–90% AN, and a perchlorate, chlorate or an alkali nitrate)

545) Heinz Dehn, USP 3463684(1969) & CA **71**, 126729d(1969) [An economical and controllable expl was obtd by treating a hydrated salt of a metal (such as Al, Cr, Fe, or Zn) and an oxidizer (such as HNO_3 or $HClO_4$) with a dialkyl sulfoxide to drive off all or part of the water of hydration to produce a corresponding cryst dialkyl sulfoxide solvate. Thus, a soln of $Al(ClO_4)_3 \cdot 6H_2O$ in Me_2SO was distd at 80° and 10mm (to remove water and excess Me_2SO) until appearance of crysts. On cooling the hexa solvate $Al(ClO_4)_3 \cdot 6Me_2SO$ pptd and was recovered by filtration and purified by washing with Me_2SO and C_6H_6. When mixed with an oxidizer, it decompd on detonation as follows: $Al(ClO_4)_3 \cdot 6Me_2SO + 12NaNO_3 \rightarrow AlCl_3 + 12CO + 42H_2O + 6S + 12H_2$. Use of smaller amts of Me_2SO permitted the formation of partial hydrates, such as tetrasolvate dihydrate, $Al(ClO_4)_3 \cdot 4Me_2SO \cdot 2H_2O$]

546) K. Takata & H. Sakamoto (Japan), GerOffen 1906776(1969) & CA **71**, 126723x (1969) [Preventing the caking of AN for use in expls was achieved by coating its particles with a small amt of "active zinc white" (I) mixed with octadecylamine (II) or its acetate. For example 97 parts of (I) and 3 parts of (II) acetate were mixed with a little water and

dried at 100–130° to a water content of 2%. The resulting product (0.5 part) was mixed with 99.5 parts fused porous AN and 6 parts fuel oil. Its deton rate was 2960m/sec. Other examples are given. In some cases, diatomaceous earth or active Al oxide were used instead of "active zinc white"]

547) T. Yamagi et al, JapanP 69 18573(1969) & CA **71**, 114820p(1969) (Porous AN granules, suitable for use in AN-FO expls were prepd by spraying high-density particles with 0.3–3.0 wt % aq soln of higher fatty acid salts, alkylbenzenesulfonates, alkyl amine acetates, etc, followed by rapid drying at 100°)

548) B.V. Pozdnyakov et al, GornyiZh **1969**, 144(5), pp36–9(1969) & CA **71**, 83168a(1969) (Industrial testing and adoption of the use of metallized granulites in mining nonferrous metals. It was claimed that metallized granular expls **AS-4** and **AS-8** were more effective than Russian AN-based expls **Detonit 10A** and **Ammonit No 1**. Compns of AS-4 and AS-8 are not given in CA)

549) F. Rzepecki, Cement-Wapno-Gips **1969**, 5, 148–51(Polish) & CA **71**, 83169b(1969) (New expl **Saletrol** and its application in open-pit mining. Saletrol was prepd by mixing *in situ* AN particles 94–6% with 6–4% mineral oil. Its max expl force was 75–80% of **Gelignite** and deton rate 3500–4000m/sec)

550) W.E. Wales & D.J. Nagy, USP 3465674 (1969) & CA **71**, 103744r(1969) (Expl compns with delayed action are described. They may be used to generate sudden pressure, initiate a mech action, or detonate an expl, particularly where it is desired to have a known short delay betw action and reaction. A series of tests was conducted wherein 1g of an N-halo amide was mixed with a small amt of iso-PrOH and the interval betw mixing and expln measured. Mixts of 1g trichloroisocyanuric acid and 0.05, 0.10, and 0.15ml iso-PrOH exploded after 15, 17, and 6 sec, resp Mixts of 1g dichloroisocyanuric acid, 1,3-dichloro-5,5-dimethylhydantoin, p-$MeC_6H_4SO_2NCl_2$, K dichloroisocyanurate, or N-bromo-5-methyl-2-oxazolidinone with 1ml iso-PrOH had times to expln 11, 41, 20, 200, and 48 sec, resp)

551) J.B. Bronstein, Jr, USP 3465675(1969) & CA **72**, 14388f(1970) [Blasting with thickened, slurried inorganic oxidizer salt-alcohol-water expl mixts. Division of USP 3395056 (CA **69**, 68071). The disclosure is similar, but the claims are different]

552) J.H. Paasch & C.W. Ehmke, USP 3467559 (1969) & CA **71**, 114822r(1969) [Expl slurries prepd in two stages are described. At first, a non-expl mixt consisting of a fuel, water and gelling agent (such as mixt of sugar 13, water 15 & guar gum 1 part) was prepd and transported to the place of blasting. Then to this mixt was added an oxidizer (such as AN in prills 71 parts) and the resulting slurry loaded into boreholes. It was detonated with the velocity of 3700m/sec]

553) W.M. Lyerly, USP 3471346(1969) & CA **72**, 4843m(1970) [Water-bearing expl compns, which contd inorg oxidizers, water & N bases were improved by including 0.2–2% by wt of a salt of a fatty alk sulfate. This salt was added to the hot soln of the blasting compn, as an aq soln or as a paste-like dispersion before addn of gelling agent. It modified the crystn of AN resulting in smaller porous crysts. The N-base salts were derived from amines and strong oxidizing acids. A typical expl consisted of AN 36.8, water 15.0, $MeNH_3^+NO_3^-$ 30.0, $NaNO_3$ 15.0, S 2.0, Na lauryl sulfate 0.25 & guar gum (crosslinked by means of an alkali metal dichromate) 1.0% by wt. Ingredients were blended in a rotary mixer and resulting product packed in polyethylene bags]

554) G.A. Cross & J.P. Rishel, USP 3471347 (1969) & CA **72**, 4842k(1970) [Gelled nitric acid blasting agents of high-strength were prepd by blending in a rotary mixer ingredients by the following sequence of steps: AN liquor (80% AN), AN solid & $MeNH_3^+NO_3^-$ were combined and heated to 120–140°F. Na lauryl sulfate was added with thorough mixing, followed by a premixed blend of $NaNO_3$ & guar gum (crosslinked by means of an alkali metal dichromate). After thoroughly mixing until thickening was observed, sulfur was uniformly incorporated. A typical product contd: water 15.0, AN 36.25, $MeNH_3^+NO_3^-$

30.0, NaNO$_3$ 15.0, S 2.0, Na lauryl sulfate 0.25 & guar gum 1.0%]

555) A. Berthmann & P. Lingens, USP 3472713(1969) & CA **72**, 4840h(1970) (Castable expls prepd from aromatic nitrocompds and nitramines and/or nitric esters and/or powd metals were desensitized by sol polychloronaphthalenes. The use of these compds produced better desensitization to rapid heating, and comparable desensitization to impact than obtd with emulsified insol waxes)

556) Ph.G. Butts, USP 3473981(1969) & CA **72**, 4846q(1970) (Gas-generating AN-based compns contg melamine 4–8%, which acted as a coolant, greatly reducing the flame temp. For example, compn contg AN 68, Guanidinium Nitrate 7.5, C-black 2.0, Na barbiturate 2.0 & melamine 6.5 parts had the calcd flame temp of 1785°F, while similar compns contg no melamine had temps as high as 2960°F)

557) M.A. Cook et al, USP 3473983(1969) & CA **72**, 14389g(1970) [Slurry blasting expls contg sulfur and Na nitrate. It has been suggested (USP's 3249477 & 3282752) that the sensitivities of aq expl slurries contg NaNO$_3$ can be improved by inclusion of S, thus making possible the use of lower proportions of the more-expensive metal-fuel sensitizer. For example, AN 31.5, NaNO$_3$ 13.5, water 15.0, guar gum 0.15 & ethyleneglycol 0.3 part by wt, were compounded as follows: The guar gum was dispersed in the glycol and this dispersion was added to the soln which was heated to 35° to completely dissolve the salts. A mixt of S 7.0, powd gilsonite 6.0 and guar gum 1.0 part was added to 60.45ps of the soln previously described. Then 25.5ps coml particulate NaNO$_3$ was added. This slurry contd 39% NaNO$_3$; it detonated completely with a std booster]

558) N.E. Barr & C.E. Tuttle, Jr, USP 3474729 (1969) & CA **72**, 23053v(1969) [Initiation of AN blasting expls by 3-stage procedure provides a more sustained duration of expln and reduces the amt of nonexploded AN. Detonation of a small HE booster is followed by expl of 20–33% of the total AN–hydrocarbon chge; then deton of a larger, secondary booster explodes the remaining chge. For example, a chge of 94 AN & diesel fuel 6% was put in a cylindrical can, fitted with a small booster well at one end, and filled to ca 2 inches above the well. A 50/50 mixt of the chge & Al was packed in a polyethylene tube closed at both ends. This tube was centered on top of the chge in the can, and the remaining chge poured into can and around the tube. Detonation was more satisfactory than with a similar can filled with the same chge, but with no tube of the Al–AN mixt]

559) G.H. Walker & C.E. Ballard, USP 3475236(1969) & CA **72**, 33911n(1970) [A non-aq cap-sensitive expl comprised of MeNO$_2$ (gelled by 2–8% NC) 10–30 and AN with NaNO$_3$ or LiNO$_3$ 90–70%. The expl had a long shelf life]

560) Ch.H. Grant, USP 3475238(1969) & CA **72**, 45635t(1970) [Gelled slurry expls comprising an intimate mixt of liq and solid phases are described. The liq phase contd a natural gum (such as guar gum) and was maintained at pH 3.0–6.5 by the addn of an acid (such as HNO$_3$) to retard crosslinking. A short time prior to use, the solid-phase component(such as AN) was mixed with the liq phase and the pH raised by adding a base (such as anhyd NH$_3$ satd sith AN) to initiate crosslinking. Deterioration of expl due to usual crosslinking was avoided]

561) H.A. Jessup & L.L. Udy, USP 3485686 (1969) & CA **72**, 68852w(1970) (Aqueous expl slurries contg a Redox crosslinking agent. For example, a slurry contg AN 31.5, NaNO$_3$ 13.5, water 15.0, guar gum 0.19, ethylene glycol 0.31, gilsonite 6.0, S 3.0, paint-grade Al 0.30, coarser Al 1.7 and NH$_4$ClO$_4$ 28.5% lost its crosslinking after 6 hrs. Addn of a small amt of a 50:50 soln of Na$_2$Cr$_2$O$_7$ and K Sb tartrate gave improved crosslinking. A further improvement was achieved by addn of ca 0.25% of borax or Na ligninsulfonate)

562) Y. Takagi & T. Komiya, JapanP 69 28376 (1969) & CA **72**, 33868d(1970) (Prepn of porous, granular AN suitable for use in expls)

563) John McKee, BritP 1171536(1969) & CA **72**, 57360h(1970) [AN-FO expls were

prepd in a device wherein AN was fed from a hopper into the bottom of a tube inclined 30–70° to the horizontal and 1–10% fuel oil was metered continuously into a 2nd opening above the AN opening. A helix mounted eccentrically below the longitudinal axis of the tube and turning at 280rpm carried the mixt to the upper end of the tube where it was discharged into a bin, bags, or cartridges. Al (and other fuels) or oxidizers could be added thru openings provided in the tube]

564) Nitrates et Engrais, BritP 1171976 (1969) & CA **72**, 68849a (1970) (Porous, essentially anhyd AN granules suitable for use in expls are prepd by a process consisting of 4 stages)

565) R.S. Gow, BritP 1173887(1969) & CA **72**, 45654(1970). Addn to BritP 1016462 (CA **64**, 9502d) [Permitted expls having the power (by wt) of 15–50% BG (Blasting Gelatin) and yielding, when fired under poor confinement, fumes low in NH_3, CO & oxides of N (thru use of finely divided oxidizer) are described. Thus, a compn contg 15 parts of 80:20 NG-NGc, NC 0.2, AN (treated with 0.05% octadecylamine) 30, NaCl 32.9, $Ca(O_2CH)_2$ 18.4, Ca stearate 0.5, guar gum 1 & china clay 2 parts provided a permitted expl of d 1.45, deton velocity 2700m/sec, power 31% of BG, propagation 14–15 inches (in 3-inch long cartridges of 1.25-in diam) and min initiator – No 1 cap (80:20–Hg fulminate–$KClO_3$)]

566) U.D. Matzke, Erzmetall **1969**, 22(8), 365–70(Ger) & CA **72**, 23038u(1970) (Review of use of mobile mixers in prepn of AN-carbon and slurry expl compns)

567) Paul Lingens, FrP 1578583(1969) & CA **72**, 113393k(1970) (Powd, gelled, or plastic, antifiredamp expls comprising a sensitizer and a ternary mixt of NH_4Cl, an alkali metal nitrate and a divalent metal oxide are claimed to be safe to handle and more powerful than conventional safety expls. Reaction betw the materials of ternary mixts during the expln proceeds accdg to the equation: $5NH_4Cl + 3KNO_3 + MgO \rightarrow 3KCl + MgCl_2 + 4N_2 + 10H_2O$. For example, a granular expl contg a mixt of NG 5.4, NGc 3.6, KNO_3 45, NH_4Cl 40 & MgO 6% had O bal +0.2%)

568) L.G. Pavlovskii et al, GornyiZh **1969**, 145(9), 35–7(Russ) & CA **72**, 57349m(1970) [A new expl **Granulit AS-8** (compn not given in CA) was tested in a proving ground and in mines and found to have deton vel ca 3050m/sec for samples contg 0.49% moisture, while for samples contg 5% water, the vel was 2450m/sec]

569) R.B. Hoppler, Jr, FrP 1579735(1969) & CA **72**, 113392j(1970) [Bulk loading of boreholes and of containers with crosslinked aqueous expl slurries, which are pumped and mixed simultaneously with injection mixtures, is described. A typical slurry contd granular AN 52, water 16.5, granular $NaNO_3$ 15.6, Al (passing a 1.27mm screen opening) 8.5, ground carbon 1.2, glycol 5 & guar gum 1 part, and its pH was 3–5. A typical injection mixture contd K pyroantimonate (crosslinking agent) 14, glycol 40, $NaNO_2$ (aerating agent) 11 & water 35 parts]

570) V.I. Chikunov et al, GornyiZh **1969**, 12(9), 60–3 & CA **73**, 5578m(1970) [Explosives sheathed with resite salt were tested for use in dusty coal mines using simulated mine drifts and boreholes. It was found that for a borehole with hard walls, the best dispersion on detonation of the sheath was obtd when there was an air gap betw the sheath and borehole walls. For boreholes in soft materials, such as clay, the sheath could fit tightly in the borehole]

Note: Accdg to CondChemDict(1961), p317-R, *resite* is the same as *C-stage resin*, which is the fully cured stage of phenol-formaldehyde resins. The 1st stage or A-stage resin is called *resol*, while 2nd stage or B-stage is known as *resitol*. Resite is infusible and insoluble in all solvents

571) E. Demelenne, AnnMinesBelg **1969**(6), 606–23(Fr/Flemish); CA **72**, 23035r(1970) (Description of research activities and regulations covered in the Belgian National Institute of Mining Industries Annual Rept **1968**, involving the safe use of expls underground. The work was directed to the prevention of hazards from defective expls or unsafe techniques that could result in premature blasts or in the ignition of methane or flammable dusts)

572) I.L. Zabudkin et al, GornyiZh **1969**, 145(8), 41–3(Russ) & CA **72**, 14369a(1970) (Description of pneumatic batch-charging unit which provided cartridges of d 1.15 to 1.2 when using granulated AN-mineral oil expls, which were mixed at the place of application)

573) F.M. Galadzhii et al, RussP 249993 (1969) & CA **72**, 45656w(1970) (A method for determining precautionary measures involving exploding a charge in a methane-air medium whose methane concn is 13–15%, is described)

574) R.I. Semigin, GornyiZh **1969**, 145(8), 63–4(Russ) & CA **72**, 23036s(1970) [Description of spontaneous explns occurring on Aug 2, 1968, in 3 boreholes made in the chalcopyrite deposit of Gaisk combine. The ore in which boreholes were drilled contd FeS_2, CuS, $CuFS_2$, $CuSO_4.5H_2O$, ZnS & other minerals. The content of S in the ore varied from 46 to 54%. Upon prolonged exposure to air the surface of the ore oxidized. At the time of charging, the boreholes (which were 12.5–15m deep and 0.25m in diam) contd columns of water 1–9m high, which became acidic (pH 1) due to dissolved products from oxidation of ore (such as H_2SO_4). Each borehole was charged with several kgs of AN expl compns and after a while, a reddish gas was noticed to evolve from some boreholes. Three chges (out of 55) exploded spontaneously at different intervals of time. The reason for such premature explns was assumed to be due to the formation of hot centers in various parts of the cartridges caused by the chem reaction of AN with the finely pulverized sulfides and S in acid medium in the presence of free Cu]

575) J. Okabe et al, GerOffen 1915456(1969) & CA **72**, 23049y(1970) {Slurry expls contg lignin-chromium complexes having plastic characteristics and high density were prepd as follows: To 5g of guar gum in 150g water, are added with stirring 700g pulverized AN (fertilizer grade) [to which was previously added at 40° a Cr-lignin compn consisting of 120g of a 40% soln of Sangrout-AS (a proprietary soil stabilizer) that contd 25g of 40% aq soln of Na dichromate]. The mass was allowed to stand 4 hrs to obtain 1 kg of a slurry expl of d 1.25, deton rate 3800m/sec. AN could be partially replaced by a perchlorate or by other nitrate. In addn powd Al or TNT could be used as activators}

576) W. Dick & O.A. Gurton, GerOffen 1919395 (1969) & CA **72**, 23047w(1970) [Expls contg an anhydrous oxidizer salt and a fuel suspended in an aq medium, such as contg AN 50–85%; up to 15% of Na, Ba, K or Ca nitrate or their mixts; up to 15% Al, Mg, B, Si or sugar or their mixts; up to 5% of a nonmetallic fuel (such as fuel oil, a carbohydrate or an anhyd lignosulfonate or a mixt of these materials); 0.2–2% guar gum; 5–25% water, and 5–25% iso-$PrNO_3$ or $AmNO_3$. The thickening agent could be a low N% NC dissolved in alcohol. Deton rates for 3-inch unconfined charges were 2800 to 4900m/sec]

577) M. Shinohara & K. Oishi, JapanP 69 05720(1969) & CA **72**, 4844n(1970) [A liq expl prepd from AN 69.9, 1:1-DNT/DNX mixt 10, a nonionic surfactant (compn not given in CA) 0.1, powd Al 10 & water 10%, had a density of 1.45 and a deton rate of 4500m/sec, when initiated by 40g of Dynamite]

578) K. Ito et al, JapanP 69 17518(1969) & CA **72**, 43048x(1970) [AN expls safe against firedamp but with high deton vel were prepd by incorporating water in addn to KCl or NaCl. For example, gel-like expls contd AN 30–70, TNT 10–40, powd Al 5–40, water 10–40, NaCl (or KCl or borax) 5–40 and an adhesive 0.5–2 parts. An expl thus prepd had a deton rate 4000m/sec compared with 3200 for an expl prepd without Al and water]

579) Y. Nomura, JapanP 69 17519(1969) & CA **72**, 14387c(1970) (Plastic expl compns prepd by mixing TNT & AN with acrylamide gel, β-dimethylaminopropionitrile and Amm persulfate. Their densities were 1.36–1.48 and deton velocity 5100–6300m/sec)

580) M. Shinohara & K. Uratsuji, JapanP 69 23038(1969) & CA **72**, 4841j(1970) [Industrial expls with low shock sensitivity were obtd as for example, by kneading TEGDN (Triethyleneglycol Dinitrate) (mixed with some NG+NGc and gelled with 0.7%

Collodion Cotton) 16 parts together with powd AN 81.6, wood powder 1.5, and surfactant (compn not given in CA) 0.2 part by wt. Its apparent d was 1.12, deton rate (measured in a 3-mm diam Fe tube) 5600m/sec and >60cm value of impact test (the wt of hammer is not given in CA]

580a) A.K. Parfenov & I.M. Voskoboinikov, FizGoreniya i Vzryva **1969**, 5(3), 347–54 (Russ) & CA **73**, 5593n(1970) [Low-speed deton in powders of TNT, Tetryl, RDX and PETN was produced on initiation by shock waves. Detonations of low velocity in the above expls took place at diams of charges above critical and below a certain diam, which depended on the dispersity of the expl and the material of enclosure for the charge. Studies of the effect of diam on the velocity of industrial expls consisting of AN and 20–50% of a sensitizer (NG, Tetryl or TNT) showed that at a certain dispersity and in a definite range of values of the diam, these expls detonated with velocities of <2.0km/sec over a large range of initiating pulses. Coating grains of TNT or Tetryl with a thin layer of oil inhibited layer combstn. Grain shattering increased the intensity of combstn of the expls because of a large increase in the sp surface]

580b) S.M. Kogarko et al, Ibid **1969**, 5(3), 379–84 & CA **73**, 5597s(1970) [Shock wave-initiated combustions of finely divided heterogeneous nonpremixed systems (such as of charcoal, coal and aerosols in air enriched with air) is discussed]

580c) J. Papp, PublHungMiningResInst **1969**, 12, 177–83 (in Ger) (The development of low-vel rock-saving expls as loosening materials is discussed. These expls consist of stable plastic foams with waterlike structure. Their hardening with AN was investigated and results reported)

581) N.G. Petrov & S.P. Mal'tsev, VzryvnoyeDelo **1969**, (67/24), pp77–82 & CA **73**, 16943c (1970) [Parameters of blasting drillings in models made of equivalent materials (such as concrete blocks) at the Chiatura manganese deposits were determined using chges of a 3:1 mixt of $C(NO_2)_4$ and oil. The degree of disintegration and the distribution of particles after the expln were compared with the results of an expt carried out in a manganese deposit quarry at 15 times larger linear scale, using **Ammonit No 6** charges. The lab models corresponded to the natural conditions]

582) A.I. Tevzadze et al, Ibid **1969**, (67/24), 149–53(Russ) & CA **73**, 27144u(1970 [Use of chamber charges of **Igdanit** for breaking down agate-contg rocks is discussed. Although Igdanit 98/2 (AN 98 & Diesel oil 2%) was more suitable, because it caused less disentegration of rock surrounding agate, Igdanit 95/5 was recommended because of superior penetrability of the fuel into AN]

583) B.D. Burlutskii, Ibid **1969**, (67/24), pp 186–201 & CA **73**, 16940z(1970) (Industrial efficiency of the expl breakdown of mica-contg rocks in open pit mines of the Aldan mica combine was detd. A new formula for calcn of blasting parameters was applied. Tests were made in vertical blast holes. Granulated TNT was found the most effective expl and the recommended diam for the blast holes was 105mm. The optimum quality of rocks was obtd when the holes were loaded so that there were air gaps between the cartridges)

584) V. Satyendra & N.G. Sinha, Technology **1969**, 6(2–3), 125–8(Engl) & CA **73**, 16941a (1970) [Expln temps (ET) of AN and AN with additives were detd by heating 0.5g samples at 2°/min in sealed glass tubes. The ET of pure AN increased with confinement vol, but additives, at a level of 2%, caused the ET to remain unchanged or even fall with increased confinement vol. Clay, gypsum, KI & $CaHPO_4$ failed to have any significant effect on ET of AN]

585) J. Foltyn et al, CzechP 132327(1969) & CA **73**, 27156z(1970) (Nontoxic plastic expl prepd by replacing NG & NGc with Pentaerythritol Trinitrate. It quickly gels the NC forming a stable and nonhygroscopic gel which easily mixes with the usual fillers)

586) V. Pesata & S. Vitek, CzechP 132339 (1969) & CA **73**, 27155y(1970) (Multi-component waterproof expls by mixing cryst AN 50–80, coated at 40–80° with 3–10 parts of a mixt of wax & paraffin and pressed at >100kg/cm²

with 8–45 parts PETN or RDX in mixt with TNT)

587) A. Berthmann et al, FrP 1581856(1969) & CA **73**, 27154x(1970) (Cast expl charge contg air-filled phenolic resin microspheres or cork flour in various proportions to permit the selection of the deton rate. An adhesive or a binder may be added)

588) R.V. Robinson, CIM(CanadInstMiningMet) Bull **1969**, 62(692), 1317–25 & CA **73**, 57619v(1970) (Water-gel expls. A review with 28 refs. New values, exptl or calcd, are given for deton rate, heat of expln, deton pressure, kinetic energy, density, impulse, and impedance of 6 typical water-gel expls)

589) M. Barbaste, FrAddn 94808(1969) to FrP 1541657(CA **71**, 51826x); CA **73**, 57647c(1970) (Ammonium Nitrate-Based Explosives) (An expl contg AN 100, fuel oil 3, naphthalene 2 and Al 4.2g was superior to that contg AN 100, fuel oil 3 & naphthalene 3g)

590) R.M. Majumdar, IndianP 111824(1969) & CA **73**, 5607v(1970) (Soluble blasting NC is prepd by nitrating with mixed nitric-sulfuric acid winding wastes and spun wastes from cotton mills, as described in the patent)

591) L. Nemeth, PublHungMiningResInst **1969**, (12), 197–201(1969) & CA **73**,16937d(1970) (Hungarian industrial expls with low volume and their fields of application are described)

592) B.P. Enoksson, GerOffen 1808922(1969) & CA **73**, 16968q(1970) (Safety expls prepd by adding to NG-NGc mixt a desensitizer such as an org ester, or an ether, as for example, diethyleneglycol monoethyl ether)

593) H.E. Todd, SAfricanP 69 03621(1969) & CA **73**, 16969r(1970) (AN resistant to sublimation and fuming was prepd by heating it to 200–337°F in the presence of a gas inert to AN, such as air contg up to 3% by vol NH$_3$)

594) G.L. Griffith et al, USP 3489623(1970) & CA **72**, 57361j(1970) [Gelling polyol polynitrate–NC expls prepd by using nitroparaffin solvents. Polynitrates (I) were not good solvents for NC (II) and, as such, were not capable of forming satisfactory gels, but when combined with nitroparaffins (III) in a sufficient amt to dissolve II in I, an acceptable gel was produced. Then III was removed from the resulting compn, and the soln thickened to form a gel. Various inorg oxidizers, org & inorg fuels, and stabilizers could be included. For example, a soln was prepd from I (9.5g), II of 13.2% N(0.5g) in MeNO$_2$ (10g) and allowed to stand in an open container until MeNO$_2$ evapd. A very firm gel of d 1.43 was obtd. As III, PrNO$_2$ & EtNO$_2$ could be used]

595) Laszlo Nagy, BányászKohaszLapok, Bányász **1970**, 103(1), 47–52(Hung) & CA **72**, 68817p(1970) [Expl characteristics of **Paxit** (which is a Hung AN-based and TNT sensitized blasting expl) can be improved by reduction of the particle size of AN, such as 60% particles smaller than 0.1mm]

596) M.F. Drukovannyi et al, GornyiZh **1970**, 145(2), 56–9(Russ) & CA **73**, 27143t(1970) [Investigations of deton failures during blasting by **Granulit AS** and **Trotyl** (TNT)]

597) J.H. Paasch, SAfricanP 68 05568(1970) & CA **73**, 89729x(1970) (Booster compns for initiation of AN-FO and aq slurried expls were developed, such as contg PETN 50, DNT 10, NC 2, di-Bu-phthalate 15, oat hull meal 2 & AN 21%)

598) S.M. Brockbank, SAfricanP 69 04373 (1970) & CA **73**, 47184k(1970) [Expl slurries comprising an aq soln of an oxidizer (such as of AN) in which dry particles were suspended together with a thickener or gelling agent, and were subject to control over aeration, foaming and other gas inclusions]

599) G.H. Walker & C.E. Ballard, SAfricanP 69 05414(1970) & CA **73**, 57648d(1970) (Expl mixts consisting of powd AN, NaNO$_3$ & LiNO$_3$, sensitized with nitroparaffins and gelled with NC or Me vinyl ether, are described. They have a long shelf life and are powerful)

600) L.L. Udy, GerP 1571229(1970) & CA **73**, 57650y(1970) (A method allowing last

minute mixing of components under controlled conditions is described. An aq slurry contg AN-$NaNO_3$ enters a mixing tank thru side ports, while dry, finely divided TNT or Al falls down gradually from a funnel that forms the top of the vessel. Guar gum is added as a thickener)

601) E.M. Atadan & Ch.H. Noren, GerOffen 1813175(1970) & CA **73**, 68169z(1970) (Thickened nitrate slurry expls sensitized for deton in holes at 5° are described. For example, a mixt of 80% aq AN soln 70, $NaNO_3$ 15, granulated sugar 8, sugar cane pulp 3, Na methylnaphthalene sulfonate 1, stearic acid 3, & crosslinked guar gum 1 part were mixed to give a thickened expl with deton rate 4500m/sec at 32° and 4150 at 4.5° in 15-cm columns)

602) A.I. Dyadechkin et al, GornyiZh **1970**, 145(3), 36(Russ); CA **73**, 37076(1970) (Investigations of processes for initiating Russ expls **Igdanit, Granulit** and **Zernogranulit** are discussed, but their compns are not given in CA. They are listed, however, later in this Volume)

603) Nitro Nobel AB (Sweden), BritP 1187661 (1970) & CA **73**, 16967p(1970) [Improved smooth-blasting expl cartridges, such as contg NG 50–55, an absorbent, such as kieselguhr 43.5–49.5, NC 0–1, and antacid, such as chalk 0.5%. Such compns had the following characteristics: Kast app impact sensitivity (2kg) +400cm and confined deton rate 3000–4000m/sec at d 1.20]

604) CanadIndsLtd, BritP 1195830(1970) & CA **73**, 68165v(1970) [A slurry expl of the compn claimed in BritP 1153680 (See CanP 797160 & CA **70**, 30545n), but without the addn of a gas stabilizer was used in boreholes ≧8 inches in diam. Thus, a mixt of prilled AN 54.55, $NaNO_3$ 20.00, guar gum 0.7, $KSb(OH)_6$ 0.05, $ZnCrO_4$ 0.5, $Zn(NO_3)_2$ (buffer) 0.2, C_2HCl_3 8.0, Diesel oil 4.0 and water 12.0% had d 1.44 & detonated at 4000m/sec when initiated by a 1 lb Pentolite chge in an unconfined 8-in diam polyethylene tube]

605) Th.F.R. Lyons, BritP 1205815(1970) & CA **73**, 122078j(1970) [Expls prepd from AN (sprayed at 50–60° with coating of dodecylamine, octadecylamine, propylamine, lauryl sulfate, or hexadecylamine) 80, Al powd 8–12 & DNT 8–12%. AN could be replaced up to 20% with $NaNO_3$ or $Ca(NO_3)_2$]

606) H. Siegert, GerOffen 1805627(1970) & CA **73**, 47182h(1970) (Fine AN granules resistant to moisture and abrasion were obtd by roll-compressing AN contg 2.4% water at 2–25 tons/cm^2 and crushing to 0.5–3mm particle size. The granules were soaked with liq expl, or a C carrier such as Diesel oil)

607) Ph.W. Fearnow, GerOffen 1812976(1970) & CA **73**, 47187p(1970) [Safe, water-contg expls capable of deton in 15-cm diam boreholes at low temp are described. For example, a 78–80% aq AN 61.0, $NaNO_3$ 18.0, DNT(min fr p 26°) 8.0, $HCONH_2$ 4.9, granulated sugar 4.9, and egg albumin 0.2 parts were mixed with small amts guar gum, 5% aq soln tartar emetic, and 5% aq soln Na dichromate. The resulting expl had d 1.3 and detonated at 4500m/sec at 4.5°]

608) Y. Wakazono et al, GerOffen 1920415 (1970) & CA **72**, 91879u(1970) [Stabilities and deton rates of slurried expls were increased by including >0.1 wt % of a hydrochloride of a higher alkylamine, an alkyltrimethylammonium chloride, imidazoline or other surfactant. For example, powd AN 44.53 contg a surfactant 0.5 was mixed thoroughly with CM-cellulose 0.2, locust bean gum 0.2 & guar gum 0.07 part. Water 20 & 35 parts TNT (>60% >30 mesh) were added to the above mixt to give the slurried expl. Deton rate, measured by Dautriche method, was ca 5620m/sec vs 5330 for similar mixt without surfactant]

609) A.G. Keenan & R.F. Siegmund, GerOffen 1935561(1970) & CA **72**, 81005(1970) [Decompn rates of Amm perchlorate expls contg ≧5 wt % AN were increased and the induction times decreased by including ≧ 0.003% of a metal (such as Cr, Cu, Ag, Ni, Co or Ce) and 10 Cl^- ions/metal atom if the metal were added as its chloride. A suitable compn contd ~90 parts by wt NH_4ClO_4 and 10ps AN with added 0.0135% $K_2Cr_2O_7$ and 0.16% NaCl]

610) SERADEX, GerOffen 1940061(1970) & CA **73**, 57646b(1970) (A permissible expl with low defgrn tendency was prepd by addn of 2–10% thermally stable polyethylene, swollen polystyrene, or urea and 1–10% Na bicarbonate to common expl mixts)

611) K. Ehrhardt, KaliSteinsalz **1970**, 5(7), 231–36(Ger) & CA **73**, 68144n(1970) (A review of firing with large amts of explosives in mining, Safety, transportation and storage are stressed)

612) E.M. Atadan & Ch.H. Noren, GerOffen 1940069(1970) & CA **72**, 123587r(1970). Addn to GerOffen 1813175 [Thickened aqueous gel expls contd AN, Na nitrate, a fuel and a thickening agent. Thus, 8.0 parts granulated sugar and 0.6ps guar rubber were added at 66° to 87.4ps soln contg $NaNO_3$ 22.3, AN 45.0 & water 20.1 parts. Addn of 4.0ps bagasse mare (or expanded grain flakes) and 5% aq $K_2Cr_2O_7$ (450ml per 45.4kg rubber) gave a viscous expl which exhibited deton vel up to 5100m/sec]

613) G. Harries, GerOffen 1942400(1970) & CA **73**, 100646r(1970) [Gelatinous expl mixts contg less than usual amts of nitric esters are described. Thus, an extruded expl contg fine AN 73.4, NG 17.5, NC 0.6, gelatinized starch 0.5, wheat flour 7.0, and Na tributylphenol sulfate 1.0 part had a d of 1.40, strength (power) 78% of Blasting Gelatin contg 91.2% NG and could be stored at least 9 months]

614) K.S. Mortensen & L.L. Udy, GerOffen 1945491(1970) & CA **72**, 134822q(1970) [Methods are described for reduction of the d of aq slurry expls by addn of gas-generating materials to avoid a decrease in the deton sensitivity with increasing d and hydrostatic pressure at the lower parts of bores. Thus, a 22-meter bore was filled with a slurry of d 1.4 contg AN 30–50, $NaNO_3$ 10–40, water 10–20, sulfur 0–10, fuel (gilsonite or bituminous coal) 1–10, Al 0.1–8 and guar gum (contg small amt of borax) 0.2–2%; and to reduce the d to 1.25, 0.36% of 35% H_2O_2 in contact with 0.1–0.2% KI or MnO_2 catalyst was mixed with the slurry to be pumped into the bore]

615) L.N. Roberts, GerOffen 1949711(1970) & CA **73**, 79087x(1970) (Liquid TNT expls for the opening of geological formations having cracks in the vicinity of well-bores are described. Expl mixts contg TNT 25.4–47.5, $MeNO_2$ 25.4–47.5, RDX (Hexogen) 2.5–5.0, HMX (Octogen) 0–5.0, NC 1.0–2.0, AN 0–40.0, and/or Al powd 0–9.8% were injected into the formation and detonated]

616) J.F.M. Craig & E.L. Falconer, GerOffen 1950580(1970) & CA **73**, 5606u(1970) (Thickened aqueous nitrate slurry expl with good water-resistance, nonstickiness, and improved flow props without sepn tendencies, such as contg AN 27.63, $NaNO_3$ 21.0, Plantago gum 0.9, guar gum 0.16, fumaric acid 0.01, $HCONH_2$ 1.6, $HOCH_2CH_2OH$ 0.30, $K_4Sb_2O_7$ 0.003, NH_4 lignosulfonate 0.10, TNT pellets 30.0, water 18.3 and $Me_2CH(CH_2)_4OH$ 3.0 parts. The slurry had d 1.45 and deton rate 4500m/sec)

617) Wasag-Chemie AG, FrDemande 2009533 (1970) & CA **73**, 47186n(1970) [ANFO expls with low content of poisonous gas were obtd by incorporating 3–6% KNO_3 or alk-earth nitrate. For example, expl mixt of AN prills 88.7, Diesel oil 6.3 & KNO_3 5% (OB –1.4%) produced 3.36 volumes N oxides and 3.6 vols CO, while a similar mixt without KNO_3 gave 8 and 9 vols, respectively]

618) N. Tomoishi, JapanP 70 09280(1970) & CA **73**, 79082s(1970) [Low-density colloidal Dynamite was prepd by mixing 0.02–3% of a mixt of (30:70)–(80:20) poly(oxyethylene) alkyl allyl ether–polyethyleneglycol monostearate. The setting point was <30°, and thus the mixing proceeded easily]

619) M.W. Arts, Explosifs **1970**, 23(2), 62–73(Fr); CA **73**, 122061y(1970) [Determination of the detonation sensitivities of AN in mixts with oil to which could be added $(NH_4)_2HPO_4$, chalk and $(NH_4)_2SO_4$. TNT & m-DNBz were used as comparative stds]

620) J. Chatel de Raguet de Brancion, GerOffen 2003930(1970) & CA **73**, 89728w(1970) [Sensitized AN, plastic or slurried, expls contd AN 31–85.5 and sensitizers (such as NG gel, DNT, powd Al, cellulose glycol and/or Pentolite) 6–63%. A representative

plastic expl contg AN 61, NG 28, DNT 9.5 & cellulose 1.5% had d 1.30 and deton rate 5200m/sec]

621) A.F. Azarkevich, Fiz-TekhnProblRazrab-PolezIscop **1970**, (3), 64–9 & CA **73**, 89719u (1970) [Correlation between the characteristics (parameters), such as heat of expln, deton rate and Hess brisance, as detd in the laboratory and their mining efficiency, was detd for some Russian industrial expls. **Ammonit 63ZhV** was used as a standard for comparison]

622) B.A. Svetlov et al, Ibid **1970**, (3), 69–79 & CA **73**, 89701g(1970) (Efficiency of water-filled, Al-contg, AN-TNT based expls was investigated. Incorporation of Al increased the heat of expln. The expl recommended contd Al 17–21, water 15, TNT 8–9%, the rest being AN)

623) A.N. Dubyrev et al, GornyiZh **1970**, 145(5), 66–8 & CA **73**, 89711k(1970) [Gases formed during explns of Russian industrial expls and liberated by exploded rock masses, were investigated in an active mine by using special cells with facilities for monitoring of gases. The expls used singly or in combinations were: **Ammonal VA-8; Ammonit No 1 Skal'nyi** (Rock); **Ammonit 6ZhV** and **Detonit VA-8**. Compns of these expls were not given in CA. They are, however, listed later in this Vol]

624) F.G. Gagauz & A.V. Drebnitsa, Vzryv-noyeDelo **1970**, (68/25), 19–23(1970) & CA **73**, 100623f(1970) [Compn of toxic gases CO & NO_2 and their amts released during blasting in mines of various ores, by mixts of AN & DNN (Dinitronaphthalene) was detd. The ore dust after the expln contd 1–7x10^{-4}% adsorbed CO and 1–2x10^{-4}% adsorbed NO_2. The concn of the toxic gases in ores after crushing and transportation were studied, as well as the effects of the cartridge-shell material and of the rock strength on the amts of toxic gases formed]

624a) A.P. Yanov et al, Ibid, **1970**, (68/25), 23–7 & CA **73**, 89704k(1970) [Dust and gas formations during explns employed in sinking of mine shafts in Krivoi Rog Basin were detd for AN–DNN-based expls. Max amts of CO (0.2–0.4%) were formed on the expln of fuse cartridges in granite rocks. An increase in the rock strength caused an increase in CO concn. The concn of CO_2 formed in dry shafts was higher than permitted. The concn of NO_2 was lower in dry shafts (0.0025%) vs 0.082% in wet shafts]

625) A.P. Yanov et al, Ibid **1970**, (68/25), 32–7 & CA **73**, 89707p(1970) (Effect of the mineralogical compn of rocks on the amt of dust and gas produced during blasting operations in the mine shafts of the Krivoi Rog Basin, was studied. No effect of the mineralogical compn of rocks on the amt of gaseous expln products was found, but an increase in rock strength caused an increase in CO formation from 29 to 39 liters per kg expl. Coolin of expln products by water flowing into the shaft caused an increase in the NO_2 concn)

626) P. I. Bolkhovoy & I.A. Ostroushko, Ibid **1970**, (68/25), 37–47 & CA **73**, 89706n(1970) (Entrapment of toxic gases, produced on expln, by rocks was detd in some Caucasian mines)

627) B.D. Rossi & V.A. Usachev, Ibid, **1970** (68/25), 64–7 & CA **73**, 89709r(1970) (Detn of the amt of toxic gases produced during expln of 20g samples of Blasting Explosives in a 50-liter Dolgov Bomb)

628) A.N. Afanasenkov, Ibid, **1970** (68/25), 93–100 & CA **73**, 89720n(1970) [Detonation props of industrial expls, which included: pressure of deton, critical pressure of initiation by shock waves and transmission distance of initiation of detonation (gap) were detd]

629) F.M. Galadzhii et al, Ibid, **1970** (68/25), 100–04 & CA **73**, 89705m(1970) (Detn of the efficiency of expls on a two-pendulum ballistic device, which is described in the paper)

630) V.I. Filatov, Ibid **1970** (68/25), 104–11 & CA **73**, 89708q(1970) (Hydrodynamic Method for detg the relative efficiency of expls consisted of explns carried out in a chamber provided with a brass plate in the bottom and filled with water. The extent of deformation

of brass plate served as a measure of efficiency. Results of detns for various expls were in better agreement with the calcd values of relative heats of explns than the results of detn in the Trauzl Bomb)

631) G.A. Polyak et al, Ibid, **1970** (68/25), 111-115 & CA **73**, 89702h (1970) (Sand Test Method of evaluation of efficiency of AN safety expls provided results proportional to their heats of expln and to the values obtd by using the Trauzl Bomb)

632) V.P. Gorkovenko et al, VzryvnoyeDelo **1970** (68/25), pp116–22 & CA **73**, 89710j (1970) (Evaluation of the permissibility of AN-based expls was conducted by comparing oscillograms of light emitted during explns. The amplitude and time of light emissions, dependence betw their values, and the probability of ignition of methane-air mixts were detd)

633) B.N. Kukib & B.D. Rossi, Ibid **1970** (68/25), 123–27 & CA **73**, 89703j (1970) [Comparison of permissibility of various expls was done by detg the "selectivity index", S= $(V_1-V_2)/V_3$, where V's are volumes of gaseous expln products (V_1, obtd on expln in a steel tube and a sand wad in the Dolgov Bomb; V_2, obtd on expln in a free space in the Dolgov Bomb; and V_3, calculated]

634) L.V. Dubnov & A.I. Romanov, Ibid, **1970** (68/25), 127–31 & CA **73**, 89721p (1970) [Techniques for evaluating the combustion tendency of permissible expls, is discussed. The value $\psi=(S_2-S_1)/S_1$, where S_1 is av (50%) transmission distance in air (gap) of initiation of deton and S_2 is av (50%) transmission distance of combstn, is taken as a basis for evaluation of the combstn tendency. The transmission distance was detd by expln of a composite cartridge consisting of active and passive segments with air gaps between them]

635) F.M. Galadzhii & V.N. Bondarenko, Ibid, **1970** (68/25), 131–34 & CA **73**, 89700f (1970) (Detn of the tendency of various expls to be ignited by an explosive pulse, is described. The expls contg NG had a higher tendency to initiation than those based on AN)

636) S.A. Mel'nikova, Ibid, **1970** (68/25), 173–76 & CA **73**, 79061j (1970) (A method for detg caking tendency of AN expls is discussed)

637) V.P. Gorkovenko, Ibid, **1970** (68/25), 202–09 & CA **73**, 79073q (1970) [Effect of the detonation rate on the permissibility of expls contg AN-TNT-NaCl (such as **Ammonit PZhV-20**) was investigated. It was found that at 2700–3200m/sec deton rate there was no ignition of methane. At higher rates, the frequency of ignition increased, reaching a maximum at 3800–4100m/sec. Above 4500–4800m/sec there was no ignition. As the deton rate of Ammonit PZhV-20 is 3800–4100m/sec, its use in gaseous coal mines is dangerous]

638) K.K. Andreev et al, Ibid **1970** (68/25), 222–31 & CA **73**, 7907 (1970) [Effect of initial temp (–15 to +20°) and viscosity on the deton susceptibility of mixts of NG with NaCl was studied]

639) V.I. Zenin & B.I. Vainshtein, Ibid, **1970** (68/25), 231–35 & CA **73**, 79067r (1970 [Addn $\leq$3–4% nitro ethers (VP-1 or VP-3) to **Ammonit PZV-20** increased its stability of the detonation. No compns of these substances are given in CA of Ref]

640) V.G. Khotin et al, Ibid, **1970** (68/25), 235–43 & CA **73**, 79066q (1970) [The susceptibility to deton of mixts contg AN with finely dispersed RDX (serving as sensitizer) was found to be higher than for mixts with normally dispersed RDX. On the other hand the self-transmission of deton was lower for mixts with finely dispersed RDX than for those contg normally dispersed RDX]

641) V.G. Khotin et al, Ibid, **1970** (68/25), 243–50 & CA **73**, 94064n (1970) [A tendency of AN expls to undergo transition from burning to deton when sensitized with RDX or liquid nitric esters was studied. It was found that a 10% content of a sensitizer (RDX or NG) helped the transition of burning **Ammonit 6ZhV-20** to expln. An increase to 25% of RDX enhanced this tendency, while a 25% NG content reduced the tendency. The same reduction of tendency was observed when 20% NaCl was added to Ammonit]

642) V.F. Starokozhev & V.S. Vacil'eva, Ibid, **1970** (68/25), 250–55 & CA **73**, 79057n (1970) [Stably-detonating permissible expls sensitized with RDX were investigated. For a thorough prepn of Ammonit in a ball mill, a normally dispersed RDX (Gheksoghen, in Russ) can replace finely dispersed RDX in **Gheksamon no 1** (Hexammon no 1). The deton stability with normally dispersed RDX (8–10%) is not worse than that of Gheksamon no 1 with finely dispersed RDX and is much better than that of **Ammonit PZhV-20.** The prepn of **Gheksamon no 5** and **Gheksamon no 6** is simpler and less dangerous than that of **Gheksamon no 1.** No compns of these expls are given in CA of Ref]

643) A.P. Glazkova & P.P. Popova, Ibid, **1970** (68/25), 265–71 & CA **73**, 79063m (1970) [The effectiveness of NH_4 salts (oxalate, citrate, tartrate, carbonate, and fluoride), Na fluoride and urethane as combstn inhibitors for AN permissible expls was studied. The combstn rates of expls with addn of inhibitors were measured as a function of pressure (up to 1000kg/cm^2). In addn to **Ammonit PZhV-20**, the following compns were tested: **ASA-1, ASA-2, ASA-3** and **AF-1**. It was concluded that permissible Ammonit-type expls with decreasing combstn tendency can be obtd by using org NH_4 salts (such as oxalate, or fluoride), urethane or NaF as inhibitors. Compns of the above listed expls are given later in this Volume]

644) N.S. Bakharevich et al, Ibid, **1970** (68/25), 271–79 & CA **73**, 79062k (1970) [Reduction of combustibility of Russ AN-based expls was achieved by replacement of NaCl in the mixt by NaF and incorporating some $Ca(NO_3)_2 \cdot 4H_2O$]

645) S.A. Mel'nikova, Ibid, **1970** (68/25), 279–83 & CA **73**, 79070m (1970)(The effect of storage for 6 months of AN expls on the caking tendency and formation of toxic gases (CO and N oxides) during deton, was studied with **Ammonit 6, Ammonit 6-ZhV** and **Ammonit 6** with 2 parts Na naphthylmethanedisulfonate (I) added. All three specimens showed increased caking tendency with duration of storage, but Ammonit 6 with I had the lowest tendency. The total amt of toxic gases formed on deton was markedly higher for stored than for fresh samples. The presence of I decreased the amt of toxic gases) (Compns of Ammonits are given later in this Vol)

646) V.I. Peshkovskii & G.S. Kal'chick, Fiz-Goreniya i Vzryva **1970**, 6(1), 123–26 (Russ) & CA **73**, 79068s (1970) [Attenuation of deton of long expl chges in boreholes, encountered in expl practice, is esp assocd with the channel effect, i.e. gradual dying out or even stoppage of deton of sheaths with a gap betw the chge and the casing wall. The mechanism of failures has not been yet clarified, but is usually explained by the hydrodynamic theory (increase in density of expl chge under the action of shock waves) which, however, is contradicted by exptl data. The data suggested that the extinguishment of deton in channel effect conditions results from the action of electromagnetic field created during the expln and spreading thru the radial gap]

647) A. Bertham & P. Lingens, USP 3496039 (1970) & CA **72**, 91876r (1970) [Expl compns based on TNT, TNB, Tritonal, etc contained 0.1–15% spray-dried Ca_3PO_4 serving to improve deton capability, thermal stability & shock resistance and to repress exudation. For example, unconfined 50mm x 20cm sticks of cast TNT boostered with 15g Tetryl would not propagate the deton, while similar sticks contg 1% Ca phosphate (of specific surface area 15m^2/g & bulk d 0.03g/ml) detonated at diam of 10mm. Cast TNT detonated under impact of a 5-kg wt falling 10cm, while same TNT contg 2% Ca phosphate required a drop of 20cm for deton. Exudation of TNT containing 2% phosphate held 20 hrs at 70° on a piece of filter paper was much less than for straight cast TNT]

648) D.S. Partridge, USP 3496040 (1970) & CA **72**, 91877s (1970) (Nongassing aq expl slurries contg small proportions of fuel, high propns of water, and no expl sensitizers were provided by mixts, such as the one contg AN 56.7–62.7, water 24, Na nitrate 10, flaked Al 0.5, hexamethylenetetramine 2–8 & guar gum 0.8%. Critical diam of these mixts contg various amts of HMeTeA were tested using 30g Pentolite boosters)

649) J.N. Maycock & Louis Witten, USP 3497405(1970) & CA **72**, 113394m(1970) (Sensitivity of AN was improved by use of a combustion catalyst that caused it to undergo more rapid thermal decompn. The catalyst was prepd by substituting $Cr_2O_3^{--}$ for NO_3^- in the crystal lattice)

650) K.S. Mortensen et al, USP 3507718 (1970) & CA **72**, 37095t(1970) (An expl slurry was produced by coating particulate plant pulp with a finely divided carbonaceous material or powd Al and soaking the resultant product with an aq soln of a strong oxidizing salt. For example, a dry mixt of sugar beet pulp 8.0, gilsonite 2.6, sulfur 4.0, and guar gum 0.3 part was satd with a soln of AN 42, water 15, $NaNO_3$ 15, guar gum 0.25, and $HOCH_2.CH_2OH$ 0.5 part. Subsequently, 13.05 parts AN was added as a supplementary oxidizer. The resulting slurry loaded in 4-inch column detonated at 25° with velocity 21000 ft/sec and with vel 3170 in 5-in column)

651) M.L. Peterson, USP 3507720(1970) & CA **73**, 5610r(1970) [Gelled aqueous expl compn, such as prepd by mixing for 10–20 mins 75% nitric acid – 85.2 parts, mineral oil 11.1, starch 2.5, and polyacrylamide (Cyanomer P-250) 1.2 part with 0.005% crosslinking agent $(HOCH_2NH)_2CO$ to obtain a gel which (20 lb in a cartridge) was detonated with 1 lb TNT. The deton rate was 5000m/sec.

652) N.M. Jardine & G. Harries, USP 3510370 (1970) & CA **73**, 16965m(1970) (Aqueous expl slurry contg ethylene oxide condensate sensitizer, such as a mixt of: fine AN 69, the condensation product of octylphenol with 13 moles ethylene oxide 1.3, $NaNO_3$ 3, molasses 6.8, Al powder 9.7, water 10, guar gum 0.3, Zn chromate 0.2, tartaric acid 0.02, and 2-ethylhexanol 0.05 part. Min Pentolite required for initiating this slurry in 4-inch cardboard tube was 50g, while similar slurry without sensitizer required 150g Pentolite for initiation)

653) H.G. Knight, Jr, USP 3523841(1970) & CA **73**, 79084u(1970) (Continuation-in-part of USP 3442729; CA **71**, 14740u) [Aqueous slurry-type Blasting Explosives contg a densified NC sensitizer, which is more sensitive than TNT or a double-base proplnt, are described. The sensitizer was prepd by adding sufficient NC solvent (MeCOBu-iso) to an agitated NC-water slurry to soften and swell NC fibers without dissolving them. Then the solvent was removed as a min-boiling water-solvent iseotrope to give the residual hard porous 100-2000μ NC particles of d 1.2–1.5 contg some noncolloided NC. Blasting expl was prepd by mixing the above densified NC 10–35 with inorg oxidizer salts (such as AN, $NaNO_3$, and/or $NaClO_4$) 39–56, water 18–31; Hexamethylenetetrammonium Nitrate, and/or ethyleneglycol 2–13; thickeners (guar gum crosslinked with 0.04% K pyroantimonate) 1; paraffin, and/or pine oils <1 and optionally, Al or ferrosilicon 14 parts. The thickener could also be guar gum crosslinked with (1-10):1 blend of a partially hydrolyzed (15–40%) polyacrylamide and a 9:1 by wt acrylamide–Na acrylate copolymer prepd as claimed in USP 3442729]

654) Y. Wakazono & Y. Otsuka, USP 3524777 (1970) & CA **73**, 100647s(1970) [Slurry expls contg an improved thickening agent are described. They contd AN (or its mixts with alk or alk-earth nitrates); at least one member being selected from the group consisting of TNT, Al, smokeless proplnt, or fuels; water, a thickening agent guar gum 0.1–2.0%, borate or borates $\leq$0.3%, Sb compd or compds (for controlling the tendency of the viscosity of the slurry to decrease with time) 0.02–2.0%, and/or <20% hexamethylenetetramine (which acts as a crosslinking agent on guar gum-AN-water solns upon addn of ca 1% Sb compd). The borates promote the crosslinking action of hexamethylenetetramine]

655) Bureau of Mines Information Circular **IC 8371**, April 1968, US Dept of Interior, Pittsburgh, Pa, 15213, "Active List of Permissible Explosives and Blasting Devices Approved before December 1, 1967", lists 83 expls of the granular AN type, 12 gelatinous, and five Cardox blasting devices. One expl has been added since last revision (1965), and 30 have been put on active list. "Permissible Explosive" is one that conforms to the basic specs and to tolerance limits prescribed by current Bureau of Mines schedules: Sched 1H for explosives and Sched 26A for blasting devices

LIST OF SPECIAL NAME DYNAMITES AND THEIR SUBSTITUTES

(Arranged in Alphabetical Order)

A1 & A2 (Monobels) (Brit). See Vol 1 of Encycl, p A1-L

A1 (Roundkol) (Brit). See Vol 1, p A1-L

A-2 and A-3 (Romanian). Permissible expls described by Tat & Hindor (Ref) and described in this Vol as No 315 Additional Reference under DYNAMITE. Properties of three other Romanian expls: **AG, AGC & AGS** are given but no compns are in CA of the Ref
Ref: S. Tat & E. Hindor, RevMinerol (Bucharest, Romania), **17**(11), 490–92(1966) & CA **67**, 55801a(1967)

Abbcites (Brit). See Vol 1, pp A1-L & A1-R

Abelite (Brit). See Vol 1, p A2-R & Giua, Trattato **VI**(1)(1959), 342

Absorbent Materials in Dynamites. Same as Dopes (qv)

Acceptable Dynamites. See Authorized Explosives in Vol 1, p A510-L

Active Base or Active Dope Dynamite. See Class II Dynamites in this Vol, under DYNAMITE

Active Sheath (Aktive Mantelpatrone, in Ger). A type of sheath contg an active ingredient such as NG and/or NGc together with substances used in permissible Dynamites. Eg: NG 11, NGc 1, NaCl 87 & guhr 1%
Refs: 1) PATR **2510**(1958), p Ger 1-R
2) Vol 3 of Encycl, p C450-L

Adams (Explosif). See Vol 1, p A102-L & Daniel(1902), p5

Aerolit (Danish). See Vol 1, p A108-L

AF-T (Russ). See under ASA and AF-1 (Also in AddnlRef 643)

Afror Tyne (Brit). See Vol 1, p A109-L

AG, AGC and AGS. See under A-2 and A-3 (Romanian)

AGB (American Glycerin Bomb). See Vol 1, p A110-L

Ageing and Rejuvenation of Dynamites. See Vol 1, pp A110 to A112

Agesid 2 (Ger). One of the pre-WWI Dynamites: NG 30, vegetable jelly 2, woodmeal 1, AN 36 & KCl 31%
Refs: 1) Naoúm, NG(1928), 411 2) PATR **2510**(1958), p Ger 3-R

Agesid A (Ger). See Wetter-Agesid A in PATR **2510**(1958), p Ger 260, Table

Agriculture and Forestry Explosives. See Vol 1, pp A112-L to A114-L and under "DYNAMITE" in this Vol, Class IX, item (A) and Class X, item (a)

"Agritol". Trademark of one of the DuPont's Blasting Explosives registered in the USPatentOffice
Ref: Blasters' Hdb (1966), p 570

Ajax Powder (Brit). See Vol 1, p A119-L

Akremit (Hungarian). A mining expl consisting of AN and gas oil. Vajda investigated it and found it satisfactory for use in boreholes and chambers. Its exact compn is not given in CA
Ref: L. Vajda, Banyaz Lapok **99**, 594–98 (1966) & CA **68**, 14614u(1968), listed as Addnl Ref 318, under DYNAMITE

Akremite (Amer). See Vol 1, p A119-L

Alba Chemical Co of New York Dynamite. See Vol 1, p A119-R

Albit (Ger). See Vol 1, p A120-L and Gesteins-Albit, described in PATR **2510**(1958), p Ger 69-L

Albite (Ital). See Vol 1, p A120-L

Aldorfit (Swiss). See Vol 1, p A123-R & PATR **2510**(1958), p Ger 3

Alexander's Explosives. See Vol 1, p A127-L

Alkalites (Belg). See Vol 1, p A127-R

Alkalsit I (Ger). A type of Blasting Explosive based on perchlorates such as: K perchlorate 28, AN 25, K (or Na) nitrate 30, Nitrocompounds (TNT) 11.5, resin (such as colophony) 2.5, woodmeal 2.5 & hydrocarbon 0.5%
Refs: 1) Ullmann, **4**(1929), 788 2) Pérez Ara (1945), 218 3) PATR **2510**(1958), Ger 3

Allison Powder (Brit). See Vol 1, p A133-R & Cundill, MP **5**, 281 (1892)

Almatrites (Al'matrity in Russ). See Vol 1, p A140-L and PATR **2145**(1945), p Rus 1

Alsilite (Belg). See Vol 1, p A141-R

Alumatol (Russ). See Vol 1, p A141-R

Aluminum Containing Dynamites (Aluminized Dynamites). The following Dynamites contg Al are described in Vol 1 of Encycl:
French Formula 226 (p A146-L)
Blasting Gelatin (Amer)(p A147, Table)
Pentrinit (Aluminized) (p A147, Table)
LeRoux Nn° 32 (Fr) (p A148-L)
Sevranite (Fr) (p A148-L)
Sofranex (Fr) (p A148-L)
Italian Plastic Explosive (p A148-L)
Aluminum Ophorite (p A155-R)
[See also Aluminized Explosives in PATR **2510**(1958), p Ger 4-R]

Amasite (Belg). See Vol 1, p A157-R

American Ammonium Nitrate Dynamites. See under AMMONIUM NITRATE DYNAMITE in Vol 1, p A355, Table

American Dynamite of 1894. See Vol 1, p A167-L

AMERICAN DYNAMITES AND SUBSTITUTES are described in this Vol under DYNAMITE, Classes 1 to X and accompanying Tables. A brief description is given in Vol 1, p A167. Those which have proper names like Americanite, Apache Powder Co's Dynamites, Atlas Dynamite, Black Diamond, DuPont's Dynamites, etc are described separately in Vols 1,2,3&4

Americanite. See Vol 1, p A168 and under DYNAMITE in this Vol, Class X

Amide (Explosif). See Vol 1, p A168-L

Amidogène (Swiss). The mixture (b) was a Dynamite which was known for its use in blasting at Iron Gates on the Danube
Refs: 1) Vol 1 of Encycl, p A171-R 2) Giua, Trattato **VI**(1)(1959), 340

Amidon (Poudre à l') *(French Starch Powder).* A blasting expl patented in 1888 was prepd by adding about 10 parts of NG to the expl mixture (patented in 1884) consisting of Na nitrate 73, charcoal 12, sulfur 10 & starch 5%
Ref: Daniel (1902), 121

Ammoksil or Ammonxyl (Russ). See Vol 1, p A286-R and PATR **2145**(1955), p Rus 1

AMMONAL (Amer, Austrian, Brit, Ger, Ital, Japan, Span & Russ). Expls used for commercial blasting and for military purposes are described in Vol 1, pp A287 to A292. Additional information on Russ Ammonals (Ammonaly) is given in the book of Yaremenko & Svetlov (1957), p160, where the following types are listed including their properties:
Ammonal No 1, Skal'nyi (Rock), **Pressovannyi** (Pressed)—AN (waterproofed), TNT, RDX, Al (powder) & Ca stearate
Ammonal No 1 ZbV, Skal'nyi, Pressovannyi—AN, TNT, Al & Ca stearate
Ammonal No 1, ZbV, Skal'nyi, Patronirovannyi Poroshok (Cartridged Powder)—AN, TNT, Al & Ca stearate
Ammonal No 2, ZbV, Skal'nyi—AN, TNT &RDX
Ammonal VA-2—AN, TNT, Al & Ca stearate or asphaltite with paraffin
Ammonal VA-4—AN, TNT & Al
Ammonal VA-8—AN, TNT, Al & Ca stearate or asphaltite with paraffin
Note: The amt of noxious gases formed on expln of Ammonal VA-8 and liberated by exploded rock masses was detd by A.N. Dubyrev et al and described in GomyiZh **1970**, 145(5) & CA **73**, 89701g(1970) (Listed as AddnlRef 623 under DYNAMITE)

Ammon-almatrit No 98 (Russ). See item (c), under Almatrits in Vol 1, p A140-L

Ammoncabusit (Ger). See Wetter-Ammoncahusit A, B, D & E described in PATR **2510** (1958), p Ger 260, Table

Ammoncarbonit or Ammonkarbonit (Ger); **Ammoncarbonite** (Belg & Brit). Accdg to Davis (Ref 1), they are permissible Dynamites which may be considered intermediate betw Carbonites and Ammonium Nitrate Dynamites. Formulations of three types are listed in Ref 2
Refs: 1) Davis (1943), 352 2) Encycl, Vol 1, p A293-L

Ammondynamit (Ger). See Vol 1 of Encycl, p A293-R

Ammondyne. See Vol 1, p A293-R

Ammonex (Amer). See Vol 1, pp A293-R to A295-R

Ammongelatine (Ger). Gelatinous AN Dynamites, contg DNClH (Dinitrochlorohydrin), developed after WWI and permitted for use in German mining. Several formulations are listed by Marshall, Naoúm and Stettbacher and three formulations are listed in PATR **2510**, p Ger 5. We are listing here two compns given by Beyling & Drekopf (Ref 1) and one compn given by Stettbacher (Ref 2)
a) *Ammongelatine 1.* DNClH (of which up to 6% of total expl can be replaced by NG or up to 18–22% by NGc) 28–33, CC (Collodion Cotton) 0.7–2, AN 46–56, alkali nitrate 10–15, nitrocompounds of toluene and/or naphthalene (with not more than 5% of total expl of TNT or TNN) 6–15, woodmeal 0–2 & harmless dye 0–5% (Ref 1)
b) *Ammongelatine 2.* DNClH 18–22, NG 3–5, CC 0.5–1.5, AN 54–57, Na nitrate 7–9, DNT 7–9, woodmeal 0.5–2, Al 1.5–3.5 & harmless dye 0–5% (Ref 1)
c) *Deutsche Ammongelatine.* "Nitrochlorin" (which is a mixture of DNClH with 15–20% of NG) 30, CC 3, DNT with TNT 10, AN 45, Na nitrate 10 & woodmeal 2% (Ref 2)
Refs: 1) Beyling & Drekopf (1936), 92
2) Stettbacher (1948), 86

Ammogelatine Dynamit of Nobel (Swed). See Vol 1, p A295-R

Ammongelignite (Brit). See Vol 1, p A295-R and Table on p A368

Ammongelit (Ger). An expl mentioned in Explosivstoffe **1957**, p 168, without giving its composition

Ammongelit 3. A German expl used by Wanzke as initial charge for exploding AN-carbon expls in blasting limestone. Its compn is not given in CA, but probably was given in original Ger paper (See Ref)
Ref: H. Wanzke, Zement-Kalk-Gips **19**(3), 121–23 (1966) & CA **65**, 3659 (1966). It is listed as AddnlRef No 268 under DYNAMITE

Ammonia Dynamites or Ammonium Nitrate Dynamites (AND). Powdery (nongelatinous), "straight" Dynamites contg large amounts of AN as replacement of part of NG. Their description is given in Vol 1 of Encyclopedia, p A357, which includes Table listing American AN Dynamites. European AN Dynamites are listed in Table on p 368. These Dynamites are also described in this Vol under DYNAMITE, Class IV, which includes Table IX, listing permissible, nongelatinous AN Dynamites of Apache Powder Co of Benson, Arizona

Ammonia Gelatin A. See Vol 1, p A306-R

Ammonia Gelatin Dynamites or Ammonium Nitrate Gelatins are described in Vol 1, pp A306-R and A367-R. Amer, Belg, Brit & French AN Gelatins are listed in Table on p A368. They are also described in this Vol, under DYNAMITE, Class V

Ammonia Gelignite (Brit). See Vol 1, p A306-R

Ammoniakkrut (Swed). See Vol 1, pp A306-R & A307-L

Ammonia Nitrate Poudre (French). See Vol 1, p A307-L

Ammonique Dynamite (French). See Vol 1, p A307-R

Table 1

Components and some properties	Designation of German Ammonits									
	No ?	43A	43B *	43C **	No ?	HI	H5	No ?	No ?	No ?
Am nitrate	42.0	46.0	56.0	45.0	46.0	50.0	50.0	55.0	52.0	50.0
Na nitrate	9.8	-	8.0	-	-	-	5.0	5.0	8.0	
Ca nitrate, $4H_2O$	-	8.0	6.0	10.0	8.0	15.0	15.0	10.0	7.0	15.0
Mg nitrate, $6H_2O$	8.4	-	-	-	-	-	-	-	-	10.0
Guanidine nitrate	-	8.0	10.0	15.0	-	-	-	-	-	10.0
PETN	9.8	-	2.0	-	-	10.0	-	-	-	-
PH-Salz	-	-	5.0	-	46.0	-	10.0	10.0		
RDX	30.0	8.0	7.0	-	-	25.0	20.0	20.0	25.0	25.0
Terra-Salz	-	-	-	-	-	-	-	-	8.0	-
TNT	-	30.0	10.0	30.0	-	-	-	--	-	-
"Vultamol" (emulsifier) (added)	-	0.3	0.3	0.5	-	-	-	-	-	-
Density (cast)	-	1.58	1.61	-	-	-	-	1.53	1.50	-
Casting-Temperature	-	104	105	-	-	-	-	108	112	-
Density of Fragments	41m	-	38m	-	39m	-	-	40m	41m	-
Mining Effect	21m³	-	-	-	-	-	-	-	-	-
References	5	5	5	4	5	4	4	5	5	4

*The composition given by Romer (Ref 4, p 22) totals 104

**Ammonit 43C exploded in 1944 on a loading line and its manufacture was discontinued. It was reported that mixtures of TNT with guanidine nitrate were unstable

Most of these mixtures were suitable for loading bombs, grenades and shells

AMMONIT (Ger). A type of AN Dynamite, known since 1884 and used for many years in several European countries including Germany (See AMMONITE). Most Ammonits have been used as commercial expls. Naoúm (Ref 1) lists seven formulations, while Beyling & Drekopf (Ref 2) list four. A typical compn is: Amm nitrate (of which 10% can be replaced with K nitrate) 77 to 87, NG (also gelatinized) 3 to 4, vegetable meal 1 to 6 & nitroderivatives of toluene (and/or naphthalene, and/or diphenylamine) 3 to 18%

Stickland (Ref 3) and Römer (Ref 4) describe Ammonits developed in Germany during WWII for military and civilian uses. Table 1 from p Ger 6 of Ref 5 lists most of these expls

Refs: 1) Naoúm, Expls (1927), 119-21 2) Beyling & Drekopf (1936), 94–5 3) O.W. Stickland et al, "General Summary of Explosives Plants", PB Rept **925**(1945), Appendix 7, p 77 4) G. Römer, "Report on Explosives", PBL Rept **85160**(1945), pp 22–4 5) PATR **2510**(1958), pp G5-R & G6 (Table)

AMMONIT (Russian) (Ammonite). Plural **Ammonity** (Ammonites). Accdg to Bebie (Ref 1) Ammonits have been the main types of Russian industrial expls. Accdg to Gorst (Ref 3), they belong to the group ACBB (Ammiachnaya Selitra Vzryvchatyiye Veshchestva) (Ammonia Saltpeter Explosive Substances) together with Ammonal, Ammontol, Ammotol, Dinamon). All these expls are now practically replaced by **Igdanit** (qv)

Compns of five Russian pre-WWII Ammonity are given in PATR **2145**(1955), p Rus 1-R and in Vol 1 of Encycl, pp A308 to A310

Vaskovskii (Ref 2, pp 25–6) lists the following Ammonits current in 1957, without giving their compns: *Ammonit No 8, Ammonit No 8–PV, Ammonit AP-1, Ammonit AP-2, Ammonit AP-2PV, Ammonit AP-5ZhV, Semyi Ammonit No 1 and Sernyi Ammonit No 2 (for use in sulfur mines); Ammonit No 6, Ammonit No 7, Ammonit No 7-PV, Ammonit V-3, Ammonit No 6ZhV, Ammonit No 7 ZhV, Ammonit, Skal'nyi (Rock) No 1, Ammonit, Skal'nyi No 1 ZhV, Ammonit No 6-PKV, Ammonit No 7-PKV, Ammonit No 9, Ammonit No 10 and Ammonit PZhV-20*

In Table XII are given compns of Ammonits which were current in 1957 and listed in the books of Gorst (Ref 3) & Yaremenko & Svetlov (Ref 4). In Ref 5 are listed Ammonits No 6, No 7, No 9 & No 10 as "modern" Russ permissible explosives of 1962

Gorkovenko (Ref 6) lists $NaNO_3$, TNT & NaCl as components of Ammonit PZhV-20, but it seems that AN must also be included in its compn

Dubyrev (Ref 7) lists among expls investigated, **Ammonit No 1, Ammonit No 1, Skal'nyi** (Rock) and **Ammonit 6ZhV**, without giving their compns

Azarkevich (Ref 8) lists **Ammonit 63ZhV** without giving its compn

Notes:

I) Waterproof-coated AN

II) In Ref 3 it is called "Predokhranitel'nyi (Permissible) without assigning it No 8

III) The values are averages from Refs 3 & 4

IV) TNT may be replaced by other aromatic Nitrocompds, such as TNX (Trinitroxylene) or TNN (Trinitronaphthalene)

V) In Ref 4, p 162 are listed the following permissible Ammonits without giving their compns, but just listing their NaCl or KCl content and properties:

Ammonit No 8, Ugol'nyi (coal). See Table 2
Ammonit PZhV-20, Ugol'nyi
Ammonit AP-1, Porodnyi (rock), NaCl 19.5%
Ammonit P-2, Porodnyi. KCl 15%
Ammonit AP-4ZV, Porodnyi. KCl 15%
Ammonit AP-5ZhV, Porodnyi. NaCl 12%

VI) In Ref 5 are listed Ammonits No 6, 7, 9 & 10 as modern Russ nonpermissible explosives

Refs: 1) Bebie (1943), 23–4 2) Vaskovskii (1957), 24–8 3) Gorst (1957), 104 4) Yaremenko & Svetlov (1957), 156, 158 & 162 5) B.Ya. Svetlov, VzryvnoyeDelo, Sbornik **49/6** (1962) & CA **61**, 6849 (1964)
(See also AddnlRefs 393, 443, 444, 478, 485, 486, 487, 492, 494, 495, 548, 581, 621, 623, 637, 639, 641, 642 & 645)

Ammonite (British). The following Ammonites which passed the Buxton Test are listed in Marshall, Vol **3** (1932), p119: **No 1** – AN 79.5, TNN 5.5 & NaCl 15%; **No 2** – AN 79.5, DNN 5.5 & NaCl 15%

Note: Buxton Test is a gallery test for coal mining expls conducted in GtBritain. It is briefly described by Marshall **3** (1932), 186–87 and is listed in Vol **2** of Encycl, p B394-R

Table 2
Russian Ammonits Used in 1957

Components	No 6 (Powdered)	No 6 (Pressed)	No 6 (I) ZhV	No 7	No 8 (II) (Permissible	No 9	No 10	V–3
Amm Nitrate	79	79	79	81.5	68	87	86	82
TNT	21	21	21	14	10	5	8	16.5
Woodmeal	–	–	–	4.5	–	–	–	–
Pine Bark	–	–	–	–	2	8	6	–
Asphaltit	–	–	–	–	–	–	–	1.0
Paraffin	–	–	–	–	–	–	–	0.5
NaCl	–	–	–	–	20	–	–	–
Density	1.0-1.15	1.25-1.35	1.0-1.15	1.0-1.1	1.0-1.15	0.9	0.9	0.95-1.1
O–Balance	+0.4%	+0.4%	–0.64%	+0.55%	+3.5%	+2.9%	+1.5%	–0.64%
Trauzl Test	370	370	370	350	250	310	320	370
Brisance	15	19	15	14	12	11	12	15
Detonation Velocity	–	3800	–	4070	–	–	–	–
References	3 & 4	3 & 4	4	3 &4	3 & 4	4	4	4

AMMONITE. Title of the description given in Vol 1 of Encycl, pp A306 to A310 of European Ammonites. Included are 28 references. Many Ammonites belonged to so-called *Favier Explosives* (qv)

Ammonit-Goudronit (Rus). See Vol 1, p A310-L

Ammonium Chlorate Explosives. See Cheddites, Vol 2, pp C155 to C161

Ammonium Nitrate Dynamites (AND). See Ammonia Dynamites

Ammonium Nitrate Gelatins (ANG). See Ammonia Gelatin Dynamites

Ammonium Perchlorate Explosives. See under Cheddites

Ammonkarbonit. Same as Ammoncarbonit

Ammon-Nobelit (Ger). A type of permissible expl used after WWI. Two formulations are listed in Refs 1 & 2: a) AN 61.0, Na nitrate 3.0, meal 7.5, glycerin 3.0, Nitrotoluenes 1.0, alkali chlorides 20.5 & NG 4.0%; OB to CO_2 ±0.0%; Trauzl Test 215cc b) AN 78.0, K nitrate 5.0, alkali chloride 8.0, meal 5.0 & NG 4.0%; OB to CO_2 +11.8%; Trauzl Test 200cc
Refs: 1) Naoúm, NG(1928), 434–35 2) PATR **2510**(1958), Ger 5-R

Ammonpek (Russ). See Vol 1, p A382-L

Ammonpentrinits (Swiss & Ger). See Vol 1, p A382

Ammonpulver (Austrian). Mixture of AN 85 with charcoal 15%, proposed in 1890 as a proplnt, but it could also be used as a substitute for BkPdr in blasting operations (Refs 1 & 4). Accdg to Davis (Ref 2), some formulations contained aromatic nitrocompounds. No detailed description is given here because Ammonpulver served primarily as a proplnt. Its modification used during WWII, which may be considered as a sort of Dynamite, consisted of AN 50, NC(12%N) 22, DEGcDN (Diethyleneglycol Dinitrate) 22, hydrocellulose 5 & Centralite 1%
Refs: 1) Marshall **3**(1932), 88 2) Davis (1943), 49 3) CIOS Rept 31–68(1945), p7 4) PATR **2510**(1958), pp 5 & 6

Ammons (Brit). See Vol 1, p A286-R

Ammonsalpetersprengstoffe. Ger for Ammonium Nitrate Explosives

Ammonsemigelatines (Brit). Se Vol 1, pp A382-R & A383-L

Ammonsprenggelatine (Ger). See Vol 1, p A383-L

Ammontal or Russian Mixture. See Vol 1, p A383-L

Ammonxyl (Russ). Same as Ammoksil

Ammonyaku (Japan). See Vol 1, p A383-L

Amonal. Spanish for Ammonal. Two formulations are listed in Table on p A289 of Vol 1 and one on p A292-L

Ammosol' of S.I. Rudakovskii. Russian industrial expl admitted since 1937 for use in coal mines contg large amts of firedamp and coal dust. Its compn and props, which were calcd by Bandurin & Rukin, are given in Vol 3 of Encycl, p C447 & C454-R

Ammotol 80/20. Russ industrial and military expl contg AN 80 & TNT 20%. It corresponds to Amer and Brit 80/20 Amatol described in Vol 1 of Encycl, p A158. There are also Ammotoly 60/40, 50/50 and 40/60 used exclusively for military purposes
Ref: A.G. Gorst, "Powders and Explosives", GosIzdatOboronProm, Moscow(1957), p23

Amvis (Brit). See Vol 1, p A393-R

Anagon (French). See Vol 1, p A400-L

Anasite. See Vol 1, p A401-R

Anbenyaku (Japan). See Vol 1, p A401-R

Anchorite (Brit). See Vol 1, p A401-R

Anderson's (Danish). See Vol 1, p A401-R

André (Explosif). See Vol 1, p A402-L

Anfex. A Russian mining expl consisting of porous, granulated AN and Diesel oil. It is more effective in blasting than **BA-62**(qv), which contains nonporous AN & Diesel oil. *Ref:* B.N. Kukib, VzryvnoyeDelo **1968** (65/22), 269–76 & CA **71**, 31945g(1969)

Angayaku (Japan). See Vol 1, p A402-L

Antacid or Antiacid Components in Dynamites. See Vol 1, pp A460-R to A461-L

Antifreezes and Their Uses in Dynamites. See Vol 1, pp A464-L to A466-L and also in this Vol under DYNAMITE, Historical and Frozen Guhrdynamite under Class I

Antifrost Celladyne (Brit). See Vol 1, p A466-L

Antifrost Gelamonite No 1. (Brit). See Vol 1, p A466-L

Antifrost Penryhn (Brit). See Vol 1, p A466-L

Antifume Blasting Gelatin (SoAfrican). See Vol 1, p A466-L

Antigel de Sûreté (Belg). See Vol 1, p A466-R

Antigrisou d'Arendonck (Belg). See Vol 1, p A466-R

Antigrisou (Explosifs) (Fr). See Vol 1, p A466-R with Table which includes Antigrisou No 1, No 2, No 3 and II. Also *Grisounite roche & Grisounite couche.* Table on p A467-L lists Antigrisou roche, couche and salpetrée

Antigrisouteux (Explosifs) (Fr). See Vol 1, p A367-L, Table, which lists Explosifs antigrisouteux Nno 7, Nno 9 and Nno 69

Antigrisoutines (Fr). Same as Explosifs antigrisouteux [Giua, Trattato **VI**(1)(1959), 342]

Antonite cava (Ital). See Vol 1, p A473-L

Antonite galleria, Extra (Ital). See Vol 1, p A473-L

Apache Powder Co's Dynamites. See Vol 1, p A473-L. Its plant located in Benson, Arizona manufactures many types of Dynamites. We have no info about its current Dynamites, but those manufd betw WWI and WWII are listed in this Vol, under DYNAMITE in Tables IV, VII, VIII, IX and X

APX Explosives (Amer). See Vol 1, p A475-L

Aquatol (Russ). A mining expl contg AN 54, TNT 28 & thickener 3 parts to which water 15 parts was added before use. When tested in dry and wet quarries, it proved to be efficient, easy to use and inexpensive *Ref:* F.N. Pys & I.T. Kolesnichenko, VzryvnoyeDelo **1967**, (63/20), 232–37 & CA **69**, 11861g(1968) (Listed as AddnlRef 397 under DYNAMITE)

Ardeer Powder (Brit). See Vol 1, p A477-R

Argarit (Swiss). See Vol 1, p A477-R

Argentine Dynamites. A list of plants manufacturing expls and ammunition, communicated to us in 1959 by Capitán de Navío, is given in Vol 1 of Encycl, pp A477-L to A480-L. Two large Govt plants: Fabrica Militar de Pólvoras y Explosivos, Villa María, Córdoba and Fábrica Naval de Explosivos, Azul, Provincia Buenos Aires are manufg Dynamites in addn to other expls. Dynamites are also manufd at two private plants: DESA at Sierras Bayas and FADEX at San Vicente. We have, however, no info about compositions of Argentine Dynamites

Argonit (Ger). See Vol 1, p A480-R

Argus (Brit). See Vol 1, p A480-R

Arionite. Belgian SGP Dynamite listed in Vol 2, p B29-R, without giving its compn

Arit. Accdg to info obtd from one of the Ger repts available in the Library of Aberdeen

Proving Ground, Maryland, it is a mining expl manufd by the VEB (Volkseigener Betrieb) Sprengstoffwerke at Gnaschwitz, E. Germany. It contains: NG, NC, TNT, sawdust and inorganic nitrates

Arkite (Brit). See Vol 1, p A480-R

Arlberg Dynamite (Brit). **See** Vol 1, p A480-R

Armstrong's Explosives (Ger). See Vol 1, p A485-R

Arnoudt's Explosive (Guatemala). See Vol 1, p A486-R

Aroclor Explosives (Amer). See Vol 1, p A486-R

AS-4 and AS-8. Russian granular metallized expls claimed to be more effective than AN-based industrial expls **Detonit 10A** and **Ammonit No 1.** Their compn is not given in CA listed below

Ref: B.V. Pozdnyakov et al, GornZh **1969**, 144(5), pp 36–9 & CA **71**, 83168a (1969) (Industrial testing and adoption of the use of metallized granulites in mining nonferrous metals) (Listed as AddnlRef 548, under DYNAMITE)

ASA and AF-1. Russian permissible expls: **ASA-1** (AN 50, TNT 20, NaCl 10, SiO_2 10, & Amm oxalate 10%); **AS-2** (AN 60, TNT 10, NaCl 10, SiO_2 10, & Amm oxalate 10%); **AS-3** (AN 65, TNT 15, NaCl 10, SiO_2 5 & Amm oxalate 5%); and **AF-1** (AN 70, TNT 10, NaCl 12, SiO_2 5 & Amm fluoride 8%) were investigated by Glazkova & Popova from the point of the effectiveness of their ingredients: Amm oxalate and fluoride as combstn inhibitors. Effectiveness of NaCl in the above expls and in Ammonit PZhV-20 was also investigated. It was found that the above salts were effective combstn inhibitors. It was also found that Amm citrate & tartrate, NaF and urethane were effective inhibitors (Compare with Brit ASA described in Vol 1 of Encycl, p A493-R)

Ref: A.P. Glazkova & P.P. Popova, VzryvnoyeDelo **1970**, (68/25), pp265–71 & CA **73**, 79063m (1970), listed as AddnlRef 643

Asbestos Dynamite. An older type of mining expl, which contained as an absorbent a mixture of powdery asbestos, chalk, silica and gypsum. Black Powder and NC could be incorporated

Table 3

Composition (%) and some properties	Astralit 2	Astralit 3	Astralit 4	Astralit O N
Ammonium nitrate	80.0	79.0	68.3	80.0
TNT + DNT	12.0			-
Vegetable meal	3.0			-
TNT + DNT + meal	-	17.0	27.7	20.0
Charcoal	1.0			-
Paraffin oil	-	-	-	-
Nitroglycerin	4.0	4.0	4.0	-
Oxygen Balance,%	-	+2.5	-	+0.3
Trauzl Test, cc	-	390	-	375
Pb Block Crushing, mm	-	16.2	-	16.0
Sensitiveness to Initiation (requires)	-	No 1 Cap	-	No 3 Cap
Velocity of Detonation m/sec	-	5400	-	4900
Density of Cartridge	-	1.09	-	1.03
Heat of Explosion, kcal/kg	-	957	-	1006
Temperature of Explosion, °C	-	2170	-	2220

Refs: 1) Daniel(1902), 31 2) Federoff & Clift **4**(1946), p6 3) Vol 1 of Encycl, p A494-L (just mentioned)

Asphalines (Belg). See Vol 1, p A496-L

Astrolit (Ger). A type of mining expl similar to Ammonit and Donarit. Compn of Astralit 1 is listed in Vol 1, p A497, while compn of Astralit 1A is given on p A498-L. Compns of Astralits 2, 3, 4 and ON are listed in Table 3 given here
Refs: 1) Naoum, NG(1928), 423 & 426
2) PATR **2510**(1958), Ger 10

Atlantic Dynamite Co of New Jersey patented in 1900 Dynamites using an absorbent which was prepd by heating a mixture of sulfur and rosin until the foaming ceased. Then the cooled mass was pulverized, mixed with other ingredients and finally with NG
Refs: 1) Daniel(1902), 32 2) Clift & Fedoroff **4**(1946), 6

Atlas Dynamite (Amer). See Vol 1, p A478-R

Atlas Powder Co of Wilmington, Delaware. See Vol 1, p A498-R. The name was changed to Atlas Chemical Industries

Atlas Powders. Naoúm, NG(1928), p 284 gave for one of the compositions: NG 75, wood fiber 21, Na nitrate 2 & Mg carbonate 2%. Two compns are given in Vol 1, p A499-L. Similar expls were *Etna* and *Hecla*

Audemar Explosives (Brit). See Vol 1, p A507

"Aunt Jemima" Powder (Amer). See Vol 1, p A507-R. Could be used as a Blasting Explosive

Austin Powder Co of Cleveland, Ohio (Amer). See Vol 1, p A507-R

Austin-Red-D-Gel (Amer). A gelatinous permissible Dynamite, manufd by Austin Powder Co

Austin-Red-Diamond (Amer). A nongelatinous permissible Dynamite manufd by Austin Powder Co
Ref: Bebie (1943), 30

Austrian Military Dynamite of Siersch & Hess. See DYNAMITE in this Vol, under Class X and also as "Camphorated Blasting Gelatin" in Vol 2, p C23-R

Authorized or Acceptable Explosives. See Vol 1, p A510-L

Avigliana Dynamite Plant at Avigliana, near Torino. See Vol 1, p A516-R

Avigliana 3 or Nitramite (Ital). See Vol 1, p A516

Axite. A Brit sporting proplnt, which could be used as a blasting expl. See Vol 1, p A516-R

BA-62. A Russian mining expl consisting of solid (non-porous) granular AN and Diesel oil. It is less effective in blasting than **Anfex** (qv), which contains porous AN & Diesel oil
Ref: B.N. Kukib, VzryvnoyeDelo **1968**, (65/22), 269–76 & CA **71**, 31945g (1969)

Baelinite (Belg). See Vol 2, p B4-L

Baldwyn's Dynamite of Hercules Powder Co (Amer). See MVD (Medium Velocity Dynamite) in this Vol under DYNAMITE Class X

Barbarit (Ger). See Vol 2, p B19

Baryte Dynamites of Nobel (Swed). See Vol 2, p B24-L

Base or Dope in Dynamites. See Dope or Base

Bautzener Sicherheitspulver (Ger). See Vol 2, p B26-L

Bavarit (Ger). See Vol 2, p B26-L

Bayon (Poudre) (Fr). See Vol 2, p B26-L

Beet Pulp Explosive of C.D. Pratt (Amer). See Vol 2, p B27-R

Belgian Ammonium Nitrate Gelatin Dynamite. See Vol 2, p A368, Table

Belgian Dynamites and Their Substitutes. Information about Belgian explosives, industrial and military, was communicated to us in 1959 by Drs L. Deffet and the late F. Lebrun. Composition of current industrial expls is given in Vol 2 of Encycl, p B29, while older industrial expls are only listed on p B30 without giving their compositions

Bellenite (Australian). See Vol 2, p B32-R

Bellites (Swed, Brit & Russ). See Vol 2, pp B32-R & B33-L

Bender Explosive (Fr). See Vol 2, p B33-L

Benedite (Brit). See Vol 2, p B33-R

Beneké Explosives (Brit). See Vol 2, p B33-R

Benite (Amer). See under "Black Powder Modifications" in Vol 2, p B173-R

Bennet Powder (Brit). See Vol 2, p B33-R

Bent & Talley Explosives (Amer). See Vol 2, p B33-R

Berclavite B (Fr). See Vol 2, p B101-R

Berg Explosive or Nitrolkrut (Swed). See Vol 2, p B101-R

Bergé Explosive (Fr). See Vol 2, p B101-R and Fedoroff & Clift Vol 4(1946), p9

Bergenström Explosive or Salite (Swed). See Vol 2, p B102-L

Bergmann Explosives (Ger). See Vol 2, p B102-L

Berg-Roburite Explosive. See Vol 2, p B103-R

Bettanin's Explosive (Amer). See Vol 2, p B109-R

Bicarbit or Bikarbit (Ger). A type of permissible expl contg large amounts of Na bicarbonate & of Na chloride and small amts of NG. They were patented before WWII by WASAG. These expls, although they contained a small amt of NG and large amts of inert ingredients, were very easy to initiate. Mixtures contg as little as 5% NG and as much as 95% of Na bicarbonate could still be initiated by ordinary blasting caps

Beyling & Drekopf (Ref 1) and PATR **2510** (Ref 2) give properties for Bikarbit contg Na bicarbonate 50, NaCl 35 & NG 15%, as follows: density 1.35, detonation velocity 2500m/sec, heat of explosion 162kcal/kg, temperature of expln 400°C and Trauzl Test value 30cc

When a more brisant Bikarbit was desired, the amt of NG was increased, while the amts of Na bicarbonate and of NaCl were decreased and some oxidizers and fuels were incorporated. One of such expls contd NG (slightly gelatinized) 30.0, Na bicarbonate 40.0, Na chloride 12.5, Na nitrate 13.0 & woodmeal 4.5%. This expl had density 1.4, detonation velocity 4000m/sec, temp of expln 1400° and Trauzl Test value 124cc

Refs: 1) Beyling & Drekopf (1936), 145–46
2) PATR **2510**(1958), pp Ger 11 & Ger 12
3) PATR **2700**, Vol 2(1962), p B111-R (Gives compn of original Bikarbit, but no properties)

Bichel Explosives (Ger). See Vol 2, p B112-L

Bielefeld Explosive (Ger). See Vol 2, p B113-L

Bielefeld's Gelatinization Method. See Vol 2, p B113-L

Björkmann, (C.G.) Explosives (Swed). Accdg to Daniel (Ref 1, pp 72–3), C.G. Björkmann patented in 1880 two expl mixts which can serve for blasting purposes. We listed these expls in Vol 2 of Encycl, p B165-L (Ref 3) but failed to mention that the nitrated mixture of glycerine and glucose (or sugar) was known as *glükodine*. Daniel (Ref 1, p 712) also stated that the same scientist patented in 1867 a Dynamite known as *Seranin* (spelled Séranine in Fr). Its compn is given in this Vol under DYNAMITE, Historical. Daniel also stated on p396 that C.G.B. invented the so-called *Kraft* (Poudre), but does not say when. Its compn is: NG 55.36, K chlorate

16.96, K nitrate 15.18 & pulverized cork 12.50%

Gody (Ref 2) listed on p398, under Glukodine, two similar compns of Dynamites invented by Björkmann. Although his initials are not given, we can assume that they are C.G.

Refs: 1) Daniel (1902), 72–3, 396 & 713 2) Gody (1907), 398 3) Vol 3 of Encycl, p B165-L

Björkmann (L.A.) Dynamite (Swed). Accdg to Daniel (Ref, p 785), L.A. Björkmann patented in 1875 in England a Dynamite known as *Vigorine* or *Vigorite*. Its compn was: Nitroline (nitrated mixt of stearin & sugar) 40, cellulose 22, K nitrate 22 & K chlorate 10%. Some formulations contained powdered tannin or coal

Black Carlit (Japan). See Kuro Carlit

Black Dynamite. See Vol 2, p B165-L

Black Powder (BkPdr) or Gunpowder. A detailed description is given in Vol 2, pp B165-R to B179-R. BkPdr and some of its modifications listed on pp B174-R to B176-L are used for some blasting operations which do not require strong expls like Dynamites

Blainite (Ital). See Vol 2, p B179-R

Blake Explosive. See Vol 2, p B179-R

Blanche Dynamite de Diller (Fr). See Vol 2, p B179-R

Blanche Dynamite de Paulilles (Fr). See Vol 2, p B179-R

Blanketed or Covered Explosives. See Enveloped Explosives, known as *Sheathed Explosives*

Blasting Explosives consisting of Dynamites and Their Substitutes are listed in Vol 2, p B202 to B211. Ammonium Nitrate Blasting Explosives are listed in Vol 1, p A341. See also in this Vol, under DYNAMITE, Class IX

Blasting Gelatin (BG) (Amer & Brit). See Vol 2, p B211-R and in this Vol, under DYNAMITE, Class III, Table V

Blasting Gelatin with Aluminum (Amer). See Vol 1, p A147

Blasting Gelatine Antifume (South African). See Vol 1, p A466-L

Blasting Oil or Explosive Oil. See Vol 2, p B212-L

Blasting Powder "A" (Amer). See Vol 2, p B212-L

Blasting Powder "B" (Amer). See Vol 2, p B212-R

Blastite (Brit). See Vol 2, p B214-R

Blomen Explosive (Amer). See Vol 2, p B216-L

Bobbinite (Brit). See Vol 2, p B218-L

Bofors Dynamites are manufd by the Aktiebolaget Bofors at Karlskoga, province of Värmland, Sweden. Activities of Bofors Industries are described in Vol 2 of Encycl, pp B218-L to B220-L, but we have no information about composition of their dynamites

Boghead Dynamite. See Vol 2, p B220-L

Bolivian Dynamites. No info at our disposal

Bolovon "O" (Austrian). See Vol 2, p B221-R

Bolton Powder (Brit). See Vol 2, p B221-R

Bombs, Dynamite (Makeshift). See Vol 2, p B239-R

Bomlit (Ger). See Vol 2, p B241-L

Boritines of Turpin (Fr). See Vol 2, p B250-R

Bostaph's Explosive (Amer). See Vol 2, p B257-R

Boyd Powders (Brit). See Vol 2, p B259-L

Brain Powders (Brit). See Vol 2, p B260-L

Bram's Powder. See Vol 2, p B260-R

Brank's Powders (Ger). See Vol 2, p B261-L

Brazilian Dynamites. Industrial Explosives in Brazil are described by Borges (Ref 2). The following plants manufg Dynamites and other industrial expls are listed in Ref 2: a) Fábrica Presidente Vargas at Piquete, São Paulo State; b) Fábrica de Estrela at Vila Inhomerin, State of Rio de Janeiro c) Companhia Dinamites do Brasil at Rio de Janeiro; and d) DuPont do Brasil Sa Industrias Químicas (Duperial)
Refs: 1) Vol 2 of Encycl(1962), pp B261 & B262 2) W.D. Borges, RevQuímInd (Rio de Janeiro), **32**(378), pp 13–16, 19–20, 23–4 & 26(1963) (Port) (Industrial Explosives in Brazil)

Brigg's Explosive (Amer). See Vol 2, p B265-L

Britanite (Brit). See Vol 2, p B300-R

British Ammonals. See Vol 1, pp A289 to B291

British Ammonium Nitrate Dynamites. See Vol 2, p B300-R

British Commercial Explosives, Advances in. See Vol 2, p B301-L

British Commercial Explosives, Including Dynamites. See Vol 3, pp C437 to C438 and C450, C452 & C453

British Dynamites. See under British Commercial Explosives in Vol 3, pp C437, C438 & C450

Britonites (Brit). See Vol 2, p B302-L

Broberg & Wildrick's Explosives (Amer). See Vol 2, p B303-L

Brockite. See Vol 2, p B303-L

Bronolithe (Ger). See Vol 2, p B317-L

Bronstein's Explosives (Amer). See Vol 2, p B317 and in this Vol, under DYNAMITE, Class VIII, Nitrostarch Dynamites

Brown's Powder. See Vol 2, p B318-R

Buck's Explosive (Amer). See Vol 2, p B320-L

Budenberg's Explosive (Fr). See Vol 2, p B320-R

Buehert's Explosive (Amer). See Vol 2, p B320-R

Bulgarian Dynamites. No information at our disposal

Bulldog Brand Powder (Brit). See Vol 2, p B324-L

Bulldog, Special (Brit). See Vol 2, p B324-L

Burrows & Hoyt's Explosive (Amer). See Vol 2, p B362-R

Burstenberger Explosives. See Vol 2, p B364-L

Burton's Explosive (Fr). See Vol 2, p B365-L

Buse's Explosive (Brit). See Vol 2, p B365-L

"C" (Explosifs) (Fr). See Vol 2, p C1-L

Cabuecite (Brit & Fr). See under Carboazotine in Vol 2, p C51-R

Cahücit (Ger). Composition of this blasting expl is given under Carboazotine. It may be added that Cahüsit was manufd before WWII by the Deutsche Cahüsit Werke AG at Gnaschwitz, located now in East Germany. A permissible expl of this type was called *Wetterammoncahüsit*
Refs: 1) Ullmann, Enzyklopädie, v **4**(1929), 780 2) Thorpe's Dictionary **4**(1940), 463 3) PATR **2510**(1958), p Ger 23-R (Cahüsit)

Calcinit (Calcinite). A type of mining explosive contg large amounts of technical calcium nitrate [$Ca(NO_3)_2 \cdot 4H_2O$], such as:

Calcinit 1. NG 15–20, Ca nitrate 32–36, Am nitrate 32–34, wood meal 13–17, liquid hydrocarbon (with flash point not lower than 30°) 0–2% (Ref 2)

Calcinit 2. NG 15–20, Ca nitrate 60–70, Am nitrate 0–15, charcoal and/or vegetable meal 6–15, liquid hydrocarbon (with flash point not less than 30°) 0–8% (Ref 2)

Marshall (Ref 1) gives for a Calcinite: NG 20, Ca nitrate 66 and charcoal 14%. Stickland (Ref 3) gives for Calcinit 1 manufd at the Krümmel Fabrik of D A -G the following composition: NGc (Nitroglycol) 6.0, DNT 4.8, TNT 7.2, Ca nitrate (tech) 38.0, Am Nitrate 35.5, wood meal 8.0, caput mortum dye (Fe_2O_3) 0.5%

Refs: 1) Marshall **3**(1932), 109 2) Beyling & Drekopf (1936), 99 3) Stickland, PBRept **925**(1945), 69 4) PATR **2510**(1958), Ger 23

Callenburg's Dynamite. See Vol 2, p C8-R

Cambrites (Brit). See Vol 2, p C13-L

Camphorated Blasting Gelatin (Austrian). See Vol 2, p C23-R

Canadian Dynamites. See Canadian Patents listed as AddnlRefs under DYNAMITE

Cannel Explosives (Belg & Australian). See Vol 2, p C25-R

Canopus (Ital). See Vol 2, p C39-R

Capexco Powder. See Vol 2, p C39-R

Carbite (Fr). See Vol 2, p C51-R

Carboazotine (Brit, Fr & Ger). See Vol 2, p C51-R (Known also as *Cabuesite* or *Cahüsit*

Carbocarbonite (Brit). See Carbon Carbonite in Vol 2, p C60-L. Its Ger name is *Kohlencarbonit*

Carbodynamites (Brit). See Vol 2, p C52-L and Giua, Trattato **VI**(1)(1959), 343

Carbogelatine (Brit). See Vol 2, p C53-R and Giua, Trattato **VI**(1)(1959), 343

Carbon Carbonite. Same as Carbocarbonit or Kohlencarbonit

Carbonit (Ger). A permissible Dynamite invented in 1885 by Bichel & Schmidt and modified many times since then. Naoúm (Ref 1), Davis (Ref 2) and PATR 2510 (Ref 3) list the following Ger Carbonits (See Table 4)

Table 4

Composition (%) and properties	Carbonit	Carbonit 1	Carbonit II	Carbonit Extra
NG	25.0	25.0	30.0	35.0
Collodion cotton	-	-	-	0.3
K nitrate	30.5	-	-	25.5
Na nitrate	-	30.5	24.5	-
Ba nitrate	4.0	-	-	4
Spent tan bark	40.0	-	-	-
Meal	-	39.5	40.5	34.7
K bichromate	-	5.0	5.0	-
Na carbonate	0.5	-	-	0.5
Density	-	-	1.10	1.20
Heat of Explosion, kcal/kg	576	536	602	-
Temperature of Explosion, °C	1874	1666	1639	-
Velocity of Detonation, m/sec	2443	3042	3850	4070
Trauzl Test (10g sample)	235 cc	240 cc	258 cc	-

(See also Kohlencarbonit under Kohlensprengstoffe)

Refs: 1) Naoúm, NG (1928), 401 2) Davis (1943), 353 3) PATR **2510**(1958), pp Ger 25 & Ger 26

Carbonit B. One of the Ger expls studied by Lingens from the point of view of its thermal decompn. Its compn is not given in CA of the Ref

Ref: Paul Lingens, IndChimBelge **1967**, 32 (Spec No, Pt 3), 515–18 (Ger); CA **70**, 79647t (1969) (See also AddnlRef 400, under DYNAMITE)

Carbonite (Brit). See under Carbonites in Vol 2, p 61-R and in Giua, Trattato **VI**(1)(1959), 343 (Table)

Carbonite, Extra (Brit) is similar in compn to Ger Carbonit, Extra, listed in Table XIV. Accdg to Naoúm (Ref), its composition was: NG 34–36, CC 0.1–0.5, K nitrate 24–27, Ba nitrate 3–5 and wood meal 31–35%
Ref: Naoúm, NG(1928), 402

Carbonites, French Modifications. Accdg to Naoúm (Ref 2), Carbonites were not allowed in French coal mines on acct of their rather high temperature of explosion, which ran betw 2400 & 3000°C. On suggestion of Commission Francaise du Grisou (the French Firedamp Commission), AN was incorporated in Carbonites and the percentage of NG was lowered slightly. The resulting permissible Dynamites became known as *Grisoutines* (qv)
Refs: 1) H. Dautriche, MP **15**, 120–22(1909–1910) 2) Naoúm, NG(1928), 402–04

Carlit or Karitto. A Japanese military expl suitable for demolition purposes. See Vol 2, p C68-R (See also Kuro-Carlit later in this Vol and in AddnRefs 228 & 319)

Carlsonites (Swed & Brit). See Vol 2, p C69-L

Car-Prill Explosive (Amer). See Vol 2, p C69-R

Carrière (Poudre de) (Fr). See Vol 2, p C69-R

Casteau (Explosifs de) (Belg). See Vol 2, p 83-R

Castellanos (Poudres) (Fr). See Vol 2, p 83-R

Cast Explosives of Winning (Amer). See Vol 2, p C84-L

Castro Powder, patented in 1883 in France is listed in Daniel, p 183 under the name *DeCastro.* The same expl was patented in 1884 in the USA under the name of *Castro Powder* (See Daniel, p 118). This expl is listed in Vol 2, p C88-R

Catactines (Fr). See Vol 2, p C88-R

Cauvet & Baron (Explosifs) (Fr). See Vol 2, p C91-R

Ceipek's Explosives (Austrian). See Vol 2, p C93-L

Celladyne Antifrost (Brit). See Antifrost Celladyne in Vol 1, p A466-L

Cellamite (Fr). See Vol 2, p C93-L

Celmonite (Brit). See Vol 2, p C125-R

Celtite or Zeltit (Ger). See Vol 2, p C125-R

Centralite RII (Belg). See Vol 2, p C440-R

Chakatsuyaku. Japanese for TNT

Chakoor Powder. See Vol 2, p C146-L

Champion Powder (Amer). See Vol 2, p C148-L

Chapman (Brit). See Vol 2, p C148-R

Charbonneux (Fr). See Vol 2, p C149-R

Charbrite 418 (Belgian). A permissible expl described by Nanquin & Demelenne (Ref) and listed in this Vol under Additional References on Dynamites as No 330
Ref: G. Nanquin & E.A. Demelenne, RevInd-Miner **49**(1), 57–64(1967) & CA **66**, 97066z (1969)

Cheddites. See Vol 2, pp C155 to C161

Chemische Fabrik's Dynamites (Ger). See Vol 2, p C178-R

Chidite (Canad). See Vol 2, p C179-L

Chlorate Explosives. See Vol 2, pp C202 to C209

Chloratit (Austrian & German). In addn to brief info given in Vol 2, p C209-R, we are including Table 5, which is the same as in Ref 3
Refs: 1) Beyling & Drekopf(1936), 97
2) Weichelt(1953) 3) PATR **2510**(1958), Ger 28

Chlorat-Rivalit (Ger). See Vol 2, p C209-R

Chloratzit (Ger). See Vol 2, p C209-R

Chrome-Ammonites (Ger). See Vol 3, p C298-L

Table 5

Components and some properties	Chloratit 1	Chloratit 2	Chloratit 3
Na chlorate and/or K chlorate	70-72	73-75	83-91
Vegetable meal	1-2	1-2	0-4
TNT and DNT	18-20	18-20	-
Paraffin	3-4	3-4	-
Nitroglycerin	3-4	-	-
Liquid hydrocarbons (flash point not less than 30°)	-	-	5-12
Oxygen Balance	+3.0%	+1.9%	-
Lead Block Expansion	290cc	280 cc	-
Lead Block Crushing	20 mm	19.5 mm	-
Sensitivity to Initiation (requires at least)	No 3 Cap	No 1 Cap	-
Gap Test (using 25 mm cartridges)	8 cm	8 cm	-
Velocity of Detonatiom	5000m/sec	4300m/sec	-
Density of Charge	1.57	1.46	-
Heat of Explosion	1250 cal/g	1280 cal/g	-
Temperature of Explosion	3645°	3700°	-

Claessen's Explosives (Ger). See Vol 3, p C325-R

Clarites (Australian). See Vol 3, p C326

Clark's Explosive (Amer). See Vol 3, p C327-L

Clement or Fuchs Powder. See Vol 3, p C329

Cliffite and Supercliffite (Brit). See Vol 3, p C329-R

Cloramite (Ital) . See Vol 3, p C329-R

Cloratita (Spanish). See Vol 3, p C329-R

Clydite (Brit). See Vol 3, p C351-L

Coad Explosives. See Vol 3, p C352-L & Giua, Trattato VI(1) (1959), 340

Coal Carbonites. See Vol 3, p C357-R and under Kohlen-Sprengstoffe

Coalites (Brit & Amer). See Vol 3, p C360-R

Coal Mining Dynamites, Nonpermissible (Amer). See Vol 3, pp C437-L to C444-R

Coal Mining Dynamites, Permissible (Amer). See Vol 3, pp C444 to C459-R

Cochise No 1 (Amer). See Vol 3, p C388-L

Coconut Fiber Dynamite. See Vol 3, p C389-L

Colinite antigrisouteuse (Belg). See Vol 3, p C393-L

Colliery Cheddite (Brit). See under CHEDDITES in Vol 2, p C158-L, item C

Colliery Explosives. Same as Coal Mine Explosives, described in Vol 3, pp C437 to C460, under COMMERCIAL OR INDUSTRIAL EXPLOSIVES

Colliery Steelites (Brit). See under Colophony–Starch Nitrated Mixtures in Vol 3, p C404-L, where *Steelites* No 3, No 5 and No 7 are described

Colloidal Dynamites and Other Colloidal Explosives. See Vol 3, p C395-R

Cologne Powder (Kölnpulver, in Ger). See Vol 3, p C403-L

Cologne Rottweiler Safety Powder (Köln–Rottweiler–Sicherheits–Sprengpulver). See Vol 3, p C403-R

Colombia (Columbia) Powder (Fr). See Vol 3, p C403-R & C420-R

Colophony, Nitrated and expls contg it: *Mining Explosive* and *Silesia A* (Ger). See Vol 3, p C404-L

Colophony-Starch Nitrated Mixture, called by Davis *Oxidized Rosin* and by its inventor E. Steele Résidée. Colliery expls contg it are known as *Steelites* (Brit). See Vol 3, p C404-L

Columbia Powder. See Vol 3, p C420-R and also Colombia Powder

Comet Powder (Amer). See Vol 3, p C433-R

Commercial and Industrial Explosives. Description given in Vol 3, pp C434 to C460 includes Dynamites, American and foreign

Commercial and Industrial Explosives, American. See Vol 3, p C444 and C455 & C456

Commercial and Industrial Explosives, Belgian. See Vol 2, p B29 and Vol 3, pp C437, C450, C452 & C453

Commercial and Industrial Explosives, British. See Vol 3, pp C437-R & C438-L and C450

Commercial and Industrial Explosives, French. See pp C438 and C450-R & C451-L

Commercial and Industrial Explosives, German. See PATR **2510**(1958), pp Ger 29–30 and Vol 3, pp C438 & C451

Commercial and Industrial Explosives, Italian. See Vol 3, pp C438-R to C440 and C451-L to C454-L

Commercial and Industrial Explosives, Japanese. See pp C441-L and C454-L

Commercial and Industrial Explosives, Russian. See Vol 3, pp C441 & C454

Commercial and Industrial Explosives, Spanish. See Vol 3, pp C441-R to C442-L and C454-R to C455-L

Commercial and Industrial Explosives, Swedish. See Vol 3, pp C442 to C443-R and C455-L

Commercial and Industrial Explosives, Swiss. See Vol 3, pp C443 to C444 and C455-L

Commercial and Industrial Explosives Prepared from Surplus Military Propellants. See Vol 3, pp C459-R to C460-L

Compex (Brit). See Vol 3, p C462-L

Compositions EL-387A and EL-387R of DuPont Co. See Vol 3, p C488-R

Compositions, Explosive (Brit). See Vol 3, p C489-R

Cooppalite and Nitrocooppalite (Belg). See Vol 3, p C512-R

Core Explosives or Core Charges. In order to lower the temperature of explosion to the level of "permissible" explosives, thus avoiding the ignition of firedamp and dust in coal mines, R.W. Lawrence of Hercules Powder Co proposed [USP 2565380(1951); CA **46**, 1752(1952)] to place inside of an AN Dynamite cartridge a "core", which can be either hollow or consist of an "inert" material, similar to that used for sheaths (See under Enveloped or Sheathed Explosives)

Cork Powder Explosives (Brit & Amer). See Vol 3, p C541-R

Cornaro Explosives (Brit & Amer). See Vol 3, p C542-L

Corncob Cellulose Dynamite (Amer). See Vol 3, p C542-L

Cornet Powder (Fr). See Vol 3, p C542-R

Cornil or Poudre Blanche (Fr). See Vol 3, p C543-L

Cornish Powder (Brit). See Vol 3, p C543-L

Cornstalk Explosives (Brit & Canadian). See Vol 3, p C543-L

Coronite or Permittite (Brit). See Vol 3, p C543-R

Cosilit (Ger). See Vol 3, p C545-R

Cotter Powder (Fr). See Vol 3, p C545-R

Coyote Tunneling Explosives (Amer). See Vol 3, p C551-L and in this Vol under DYNAMITE, Class IX, Item D

Cratering Explosives (Amer). See Vol 3, p C553-R and in Table on p D57, under Demolition Explosives. Also in this Vol under DYNAMITE, Class IX

Cratiti (Ital). See Vol 3, p C555-L

Cremonita (Ital). See Vol 3, p C556-L

Cremonita (listed by Pérez Ara). See Vol 3, p C556-L

Cronite (Amer). See Vol 3, p C561-R

CSE (Explosifs de) (Fr). Explosives developed at the laboratory of CSE (Commission des Substances Explosives). See Vol 3, pp C570 to C572 with Tables on pp C571 & C572
Note: Several expls are based on NEPD, which means 2-Nitro-2-ethyl-1.3-propanediol Dinitrate

Cugnite (Fr). See Vol 3, p C573-L

Curtis & André Powders (Brit). See Vol 3, p C578-L

Curtisite (Brit). See Vol 3, p C578-R

Cyclonite-Nitromethane Mixtures (Canad). See Vol 3, p C601-R

Cyclonite Mixtures, Binary and Ternary. Numerous Amer, Fr, Ger, Ital, Japan, Russ, Span, Swed & Swiss are listed in Vol 3, pp C626 to C630. Some of the mixtures are suitable for demolition purposes

Cyclotrimethylenetrinitrosamine or R-Salt (Ger name **R-Salz**). Its mixtures with other ingredients were used in Germany during WWII as substitute explosives for military and commercial purposes. Table on p C632 of Vol 3 (from Table 57, p Ger 170 of PATR 2510) lists 8 of such expl mixtures. Refs on R-Salt are given on pp C631 & C632 of Vol 3

Czech Dynamites. No information on their compositions at our disposal. Two Czech pre-WWII arms and munitions plants are listed, however, in Vol 3, p C638. They are Czekoslovenska Sbrojovka Akciova Spolecnost v Brne (formerly called Brünn) and Skoda Werke at Pilsen

Dahmen Explosives (Austrian & Ger). Six formulations are given in Vol 3, p D1-R under "Dahmen, Johann, von"; two of these formulations are given in PATR **2510**(1958), p G32-R and three formulations in Giua, Trattato **VI**(1)(1959) under "Explosivo von Dahmen"

Dahmenit A (Ger). See Vol 3, p D1-R

Dainamito. Japanese for Dynamite

Danish Dynamites. No information at our disposal

DAP-1. A Czech mining expl described by J. Dusek & R. Mecir in Rudy (Prague) **14** (3), 72–4(1966) & CA **65**, 5292(1966). It is listed as Addnl Ref No 264 for DYNAMITE

Dapremont Powder (Ital). See Vol 3, p D16-R

Davey's Powder 1 (Brit). See Vol 3, p D17-R

Davey's Powder 2 (Brit). See Vol 3, p D17-R

Davey's Powder 3 (Brit). See Vol 3, p D18-L

Davey & Watson's Powder (Brit). See Vol 3, p D18-L

Davies & Curtis' Safety Explosive (Brit). See Vol 3, p D18-L

Davies' Powder (Brit). See Vol 3, p D18-L

Davies' Safety Explosive (Brit). See Vol 3, p D18

Dawson & Karstairs' Powders (Brit). See Vol 3, p D18-R

Dean Explosive (Brit). See Vol 3, p D21-L

DeCastro (Explosive) (Fr). See Vol 3, p D37-L, where it is erroneously spelled "DeCustro Explosive" (Comp with "Castro Powder", listed in this Vol)

Defraiteur's Explosives (Fr). See Vol 3, p D40-R

Deissler & Kuhnt's (Brit). See Vol 3, p D49-R

Dekammon 1 and Dekammon 2. East German AN-fuel expls developed for use in potash mines. Their exact compns are not given in CA
Ref: J. Kaemmler & J. Ruhmannseder, Rudy (Prague) **14**, 139–41 (1966) & CA **67**, 4302 (1967)

Delattre (Explosif) (Belg). See Vol 3, p D49-R

Delborbe (Explosif) (Fr). See Vol 3, p D54-R

Delvigne (Explosif) (Belg). See Vol 3, p D55-R

DeMacar (Explosifs) or *Macarites* (Belg). See Vol 3, p D55-R

Demétriade, Jonesen & Williams' Explosive. See Vol 3, p D55-R

Demolition Dynamite (Amer). See Vol 3, p D57, Table and Items (d) & (e) on p D58-L. Also in this Vol under DYNAMITE, Class IX, Item d

Denaby Powders (Brit). See Vol 3, p D62-R

Denespex (Brit). See Vol 3, p D63-L

Densites (Belg). See Vol 3, p D64-R & Gody (1907), 701

DeTerrés Explosives (Brit). See Vol 3, p D102-L

Detonit (Ger). In addn to info about Brit Detonites given on p D1109-L, it may be stated that the Germans used the same three formulations, plus *Detonit 3* of compn: AN 82.7, NC (colloided by NG) aromatic nitrocompds 1.0, vegetable meal 4.3 & NaCl 8.0%; Trauzl Test value 225cc
Ref: PATR **2510** (1958), p Ger 35-R

Detonit 10A (Russ). This industrial expl was investigated from the point of view of formation of toxic gases during expln in mines, but its compn was not given in CA of Ref
Ref: A.N. Dubyrev, et al, GornyiZh **1970**, 145 (5), 66–8 & CA **73**, 89711k (1970) (Listed as AddnlRef 548, under DYNAMITE)

Diaspan Explosive. See in this Vol, p D1153-L

Diaspan Gelatin. See in this Vol, p D1153-L

Dinaftalit (Dinaphthalite). Russian mining expls described in this Vol. One of such expls is **Zernënyi Dinaftalit** (Granular Dinaphthalite). There are also: **Dinaftalit No 1**, which contains noncoated AN 88 & DNN 12%, and **Dinaftalit No 1 ZhV**, which contains waterproofed AN 88 & DNN 12%
Refs: 1) Yaremenko & Svetlov (1957), 157 2) N.F. Adrianov et al, VzryvnoyeDelo **1968** (65/22), 109–27 & CA **71**, 31926b (1969) (Testing of Dinaftalit at various Russ mining installations) (Listed as AddnlRef 485 under DYNAMITE)

Dinamit. Russian for Dynamite

Dinamita. Spanish for Dynamite

Dinamitas de Galdácano. Popular Spanish Dynamites manufd by la Sociedad Anónima Española de la Dinamita in its Fábrica de Galdácano (Vizcaya). They are listed on pp 397–98 of the book by Vivas, Feigenspan & Ladreda, Vol 2 (1946) and also in Vol 3 of Encycl, under COMMERCIAL or INDUSTRIAL EXPLOSIVES. Their nonpermissible formulations known in Spanish as "explosivos industriales de no seguridad" are listed on pp C441 & C442, while their permissible varieties, known as "explosivos de seguridad contra el grisú" are on p C455-L

Dinamite. Italian for Dynamite

Dinamon. Russian for Dynammon. See under DYNAMMONS in this Section on DYNAMITE

Diorrexine (Austrian). A mining expl patented in 1881 by Pancera contd Na nitrate, sulfur & sawdust mixed with water and ca 1.5% Picric Acid
Ref: Daniel (1902), 206

Ditching Dynamites of DuPont Co. See in BlastersHdb (1968), p 33 and in this Vol under DYNAMITE, Class IX, Item (Ad)

Dittman's Dynamite. See in this Vol

Dittmar's Dynamites. See in this Vol under Dittmar's Explosives

Divine's Explosive Rack-a-Rock. See Divine's Explosives in this Vol

Domergue Explosive. See in this Vol

Donar. See in this Vol

Donarits. See in this Vol

Dorfit. See in this Vol

Double Effect Powders. See in this Vol

Douglas Powder. See in this Vol

Dualines. See in this Vol

Dualine Stumping Powder. See in this Vol

DuBois-Raymond Explosives. See in this Vol

Dulitz Explosive. See in this Vol

Dumorite. See in this Vol

Duobel. See in this Vol

Duplexite. See in this Vol

DuPont's Dynamites. See in this Vol

Dutch Dynamites. No information at our disposal

Duxita B. See in this Vol

Duxite. See in this Vol

Dynamagnite. See in this Vol

Dynamit. German & Swiss for Dynamite

Dynamit 1 (Ger). NG 61.0–63.5, CC 1.5–4.0, Na nitrate and/or K perchlorate 25–29 & soda and/or chalk and/or ocher 0–2%
Refs: 1) Naoúm, NG 1928), 334 2) Beyling & Drekopf (1936), 90–91

Dynamit 2 (Ger). NG 56–61, NC (from surplus smokeless proplnts) 4.9–9.0, alkali nitrate and/or K perchlorate 25–30, woodmeal 3–8, nitrocompounds of toluene and/or naphthalene and/or diphenylamine 0–4%
Ref: Naoúm, NG (1928), 334

Dynamit 3 (Ger). NG 34–39, CC 0.5–3.0, alkali nitrate and/or AN and/or K perchlorate 44–54, woodmeal 1–6, nitrocompds of toluene and/or naphthalene and/or diphenylamine 6–10 & antacid with ocher 0–5%
Ref: Beyling & Drekopf (1936), 91
Note: A slightly different formulation is given in Naoúm, NG (1928), 334. He cites NC (from surplus smokeless proplnt) 1–6% instead of CC

Dynamit 4 (Ger). NG 36.0–39.5, NC (from surplus smokeless proplnt) 0.5–4, alkali nitrate and/or K perchlorate 40–50, vegetable meal and/or solid hydrocarbons 1–7 & inorganic inert salts 7–12%
Ref: Naoúm, NG (1928), 334

Dynamit 5 (Ger). NG 16–22; CC 0.5–2, K perchlorate and/or AN and/or alkali nitrate 50–74, woodmeal and/or solid hydrocarbons 1–6, nitrocompds of toluene and/or naphthalene and/or diphenylamine 2–12, alkali chloride 0–12 & antacid with some ocher 0–5%
Ref: Beyling & Drekopf (1936), 91

Dynamit "F" (Swiss). See Vol 3, p C443, under "Swiss Commercial Explosives of Nonpermissible Type"

Dynamit "N" (Swiss & Ger). A current Dynamite based on Stettbacher's patents. It contains NG and/or NG with either PETN (Ref 1) or RDX (Refs 2 & 3). For example: RDX 70 & NGc (gelatinized with CC) 30% (Refs 2 & 3). Its density is 1.54, detonation velocity 8200m/sec, temperature of

explosion 4170°C, volume of gases at NPT 746 l/kg and specific pressure 12538kg/cm². It is suitable for use in demolition of reinforced concrete and steel constructions
Refs: 1) A. Stettbacher, private communication, Zurich (1953) 2) F. Weichelt, "Handbuch der gewerblichen Sprengtechnik, C. Marhold, Halle-Saale (1953), 375
3) PATR **2510**(1958), p Ger 39

Dynamite. Belgian, British and French for Dynamite

DYNAMITE. See section in this Vol prior to the "LIST OF SPECIAL NAME DYNAMITES AND THEIR SUBSTITUTES"

Dynamite O (Fr). See Vol 1, p A368, Table

Dynamite No O (Ital). NG 70, Na nitrate 25 & woodmeal 5%
Ref: Giua, Trattato **VI**(1) (1959), 340

Dynamite No 1. European name for Guhrdynamite contg NG 75 & kieselguhr 25%. Its Amer name is *Giant Powder No 1*

Dynamite No 1 (Ital). NG 70, Na nitrate 18, woodmeal 8 & charcoal 4%
Ref: Giua, Trattato **VI**(1) (1959), 340

Dynamite II (Brit). NG 18, K nitrate 71, charcoal 10 & paraffin 1%
Ref: Thorpe (1917), 437

Dynamite No 3 (Brit). NG 50, K nitrate 33.1, guhr 7, woodmeal 9.7 & chalk 0.2%
Ref: Marshall **2**(1917), 491

Dynamite No 3 (Ital). NG 25, Na nitrate 54, woodmeal 19% & Na carbonate with some ocher 2%
Ref: Giua, Trattato **VI**(1) (1959), 340

Dynamite, Ageing and Rejuvenation. See Vol 1, pp A110 to A112

Dynamite, Baryte. See Baryte (Dynamites) in Vol 1, p B24-L

Dynamite-Grisoutites. Belgian and French permissible expls, such as NG 44–42, Mg sulfate 44–46 & cellulose, woodmeal or kieselguhr 12%
Refs: 1) Gody (1907), pp 701 & 713
2) Clift & Fedoroff, Vol **2**(1943), p D14
Note: Other French permissible expls are listed in Vol 3 of Encycl, p C450

Dynamite of Trauzl & Abel (Brit & Ger), also known as:
Guncotton Dynamite (Ger), is described in this Vol, under DYNAMITE, Class II and in Naoum, NG (1928), 282

Dynamite de Vonges. An older French Dynamite which contained randanite, a siliceous material found in Auvergne, France. E.g: NG 75, randanite 20.8, pulverized quartz 3.8 & Mg carbonate 0.4%
Refs: 1) Marshall **1**(1917), 360 2) Clift & Fedoroff **2**(1943), p U-V2

DYNAMONS: Dynammon Sprengstoffe (Austrian & German), **Dinamoni** (Italian) and **Dinamony** (Russian). They are mechanical mixtures of AN (Ammonium Nitrate) with various combustible, but nonexplosive substances, such as sawdust, lignite, bark flour, peat, coal dust, charcoal, etc. Dynammons are inexpensive and very suitable for blasting hard ores on lower mine levels and also for excavation work, ditching, blasting wood logs, etc. They are unsuitable, however, for reinforced concrete, steel construction and hard rocks

Following are examples:
A) *Dynammon of E. von Geldern* consisted of AN mixed with naturally carbonized celluloses, such as peat, fallen leaves, rotten trees, etc (Ref 1)
B) *Austrian Dynammon:* AN 87–88 & charcoal 13–12% (Refs 2a & 6)
C) *German Dynammon:* AN 90 & red charcoal 10%; its density was 1.01 and detonation velocity 4100–4210m/sec (Refs 2b & 12)
D) *German Dynammon:* AN 95.5 & charcoal 4.5%; its density was 0.865 and detonation velocity 3380m/sec (Refs 2b & 12)
E) *Dynammon I:* AN 88 & lignite 12% (Ref 5)
F) *Italian Dinamon,* described by Belgrano

Ref 9a, p 163) and in Vol 3 of Encycl, p C440-L has quite a different compn. No Dinamons are listed in Giua, Trattato **VI**(1) (1959), Alphabetical Index, p 845

G) **Russian Dinamony,** described in Vol 3 of Encycl, p C441-R and in Refs 3, 4, 6, 7, 8, 9, 10 & 11 are based on coated AN. They have been prepd by heating AN crysts to 55–65°C and stirring them for 10–15 mins with 1.5–2% of paraffin. Then a pulverized combustible material [such as woodflour, pine bark, peat (called in Russ "torf"), etc]was gradually added with stirring and the thoroughly blended mixture extruded (while still hot) into paper cartridges. Dinamony were introduced in Russia beginning in 1934, but now they are practically replaced by **Igdanit** (qv)

The following formulations are described in Vol 3, p C441-R: Dinamon "D", Dinamon "K", Dinamon "SSh" and Dinamon "T"

To these may be added the following:

a) *Dinamon* (listed by Gorst, Ref 10): AN 88–90 & organic flour (such as pine bark) 12–10%; its d 0.9, deton rate 2500m/sec, strength (by Trauzl Test (called "fugasnost") 330cc (vs 225 for straight AN) and brisance by Pb cylinder compression 11mm (Ref 10) (Compare with Dinamon "T")

b) *Dinamon "DM"* (listed in Ref 11): AN 85 & woodflour 15%; d 0.9, deton rate 2800 m/sec, strength 320cc, brisance 10–13mm & gap test 3–4cm

c) *Dinamon "T"* (listed in Ref 11): AN 88 & turf 12%; d 1.0, deton rate 3000, brisance 13–16mm, and gap test 3–4cm

d) *Dinamon "K"* (listed in Ref 11) – same compn as given in Vol 3; its d 1.0, brisance 11–14 & gap test 3–5cm

e) *Dinamon "SSh"* (listed in Ref 11) – same compn as given in Vol 3; its d is 1.1–1.15, strength 340cc & brisance 16–17mm

Refs: 1) Daniel (1902), 231 & 793
2a) Marshall **1**(1917), 392 2b) Marshall **2** (1917), 493 3) P. Radevich, Tekhnika i Voöruzheniye **8**(10), 126(1939) 4) M. Onukhin, Ibid **8**(11), 59–61(1939)
5) CondChemDict (1942), 282(Not found in later editions) 6) Clift & Fedoroff **2**(1943), p D14 7) I.A. Mukhin, Ugol' (coal) **20**, (No 1/2), 27(1945) 8) Fedoroff et al, PATR **2145**(1955), pp Rus 5 & Rus 6 9) V. Mertsalov, GornyiZh **120**(No 7/8)(1946), p 39
9a) Belgrano (1952), 163 10) Gorst (1957), 104 11) Yaremenko & Svetlov (1957), 149
12) Fedoroff et al, PATR **2510**(1958), p Ger 39 13) Giua, Trattato **VI**(1) (1959) – not listed in Alphabetical Index

Other Refs on Dynammons are given in the following Addnl Refs listed under DYNAMITE: 383 & 475

Dynamogène (1882). French expl prepd by boiling with agitation 17 parts of K ferrocyanide and 17p of charcoal in 150p of water. After cooling, the mass was thoroughly blended with 70p of K chlorate, 30p of K carbonate, 10p of starch in 50p of water and spread on pieces of filter paper to dry. Then the paper with adhering material was cut and rolled in the shape of cartridges

Ref: Daniel, Dict (1902), 232

Dynamoites (Fr & Brit). Explosives patented in 1891 by Moschek & Brunner EnglP 5843 & FrP 212265): a) K chlorate 20 to 40, AN 40 to 10 & residues from the manuf of malt (residui di malto, in Ital) 40 to 50%; b) K chlorate 60 to 30 & malt residues 40 to 70% and c) AN 30–60 & malt residue 70–40%

Refs: Daniel, Dict (1902), 232 2) Giua, Trattato **VI**(1) (1959), 395

Dynastite. French expl prepd by thoroughly blending 94 parts of pulverized K chlorate with 6ps of pulverized Ba nitrate and then soaking the resulting mass for at least 2 hours in liquid Nitrotoluenes

Refs: 1) G. Rauzieres, FrP 465718 and CA **8**, 3629(1914) 2) Clift & Fedoroff **2**(1943), p D14

Dynobels. A series of British mining explosives, of which the older type passed the Rotherham Gallery Test and was on the British Permitted List: K chlorate 27, NG 32.5, Collod Cotton 0.7, Amm oxalate 29.5 & woodmeal 10.3%. Its swing by Ballistic Pendulum was 2.61 inches compared with 3.27 for "Standard 60% Gelignite" (Power ca 80% of Gelignite) (Ref 1, p 385)

The newer types include: a) *Dynobel No 3* NG 15.0, NC 0.5, Di- & Tri- nitrotoluenes 1.5, AN 53.0, woodmeal 5.0 & Na chloride 25.0%. Power 76.4% of Gelignite (Ref 2, p 120); b) *Polar Dynobel* NG (with NGc) 15.0, NC 0.5, Di- & Tri- nitrotoluenes 1.0, AN 53.0, woodmeal 5.5 & Na chloride 25.0%. Power 77.3% (Ref 2, p 120); c) *Polar Dynobel No 2* NG with NGc 15.0, NC 0.5, Di- & Tri- nitrotoluenes 1.0, AN 62.5, woodmeal 5.0 & Na chloride 16.0%. Power 83.4% (Ref 2, p 120)

Refs: 1) Marshall **1**(1917), 385 2) Marshall **3**(1932), 120 3) Clift & Fedoroff **2** (1943), p D14

Substitute Dynamites Listed in Additional References Without Their Compositions

Gheksamony (Hexammons). Russian permissible expls based on 8–10% normally dispersed or finely dispersed RDX (Gheksoghen in Rus) and AN (Ammiachnaya Selitra, in Russ). In CA **73** of Ref are listed **Gheksamony no 1, no 5** and **no 6** (spelled by the abstractor as hexamon no 1, hexamon no 5 and hexamon no 6, without giving their compns)

Ref: V.F. Starkozhev & V.F. Vasil'eva, VzryvnoyeDelo **1970**, 68/25, pp 250–55 & CA **73**, 79057n (1970) (Stable-detonating permissible expls, including Gheksamony no 1, no 2 & no 3 and Ammonit PZhV-20) (Listed as AddnlRef 642 under DYNAMITE)

Gomme A. French Dynamite contg NG 92–94 and collodion cotton 8–6%. It is practically identical with Amer "Blasting Gelatin" or Ger "Sprenggelatine"

Ref: Pepin Lehalleur (1935), 334–35

Note: It is called Gum A in CA **70**, 69761m, AddnlRef 471, which is not correct, because the explosive is not gum but a hard substance. The word Gomme A signifies a specific expl and should not be translated into English

Granulity and Zernogranulity (Granulated Granulites). According to Svetlov (Ref), Granulits are Russ nonpermissible expls consisting of AN mixed at the plant with petroleum products, Al powd and carbon black, while Granulated Granulits are mixts of AN with granulated or flaked TNT

Refs: 1) B.Ya. Svetlov, VzryvnoyeDelo, Sbornik **49/6**(1962); RefZhKhim **1963**, Abstr No 20N/390; CA **61**, 6849(1964) (Modern Russ nonpermissible expls, which include Granulits and Granulated Granulits) 2) L.G. Pavlovskii et al, GornyiZh **1969**, 145(9), 35–7 & CA **72**, 57349m (1970) (Props of **Granulit AS-8**: deton rate 3050 m/sec for samples contg 0.49% moisture, while for samples contg 5% water, vel was 2450m/sec. No compn given in CA) 3) M.F. Drukovannyi et al, GornyiZh **1970**, 145(2), 56–9 & CA **73**, 21143t (1970) (Investigations of deton failures during blasting by **Granulit**) 4) N.I. Dyadechkin et al, GornyiZh **1970**, 145(3), 36(Russ) & CA **73**, 37076n (1970) [Description of initiation of Granulit & Zernogranulit is given, using 2 lines of detonating cord placed along the whole length of the cartridges or by two donor charges (min wt 0.4kg each) connected by two lines of detonating cord. Compns of **Granulit AS-8** and of **Zernogranulit** were not given in GornyiZh and in CA]

Note: Above papers are listed in AddnlRefs 114, 568, 596 and 602 for DYNAMITE

IGDANITY (Igdanites). Accdg to Khablov (Ref 1), they are nonpermissible, free-flowing expls, consisting of granular AN 95–97 & Diesel oil 5–3%, and their ingredients are combined *in situ*, just before use. This information was given in Encycl, Vol 3, p C441-R, where it was stated that these expls were developed in 1961, but actually it was in 1959, as stated by Demidyuk & Rossi (Ref 4). These expls were extensively tested in 1963-1964 and in 1966 included in the Official List of Russian Explosives. They practically replaced **Dinamony**, introduced in 1934. It is stated in Ref 4, that the term "Igdanit" was derived from IGDAN, which is probably an abbr for "Institut Gornago Dela, Akademiya Nauk", where Igdanity were developed. CA of Ref 4 does not give Russ meaning of IGDAN, but its Engl equivalent: "Institute of Mining, Academy of Sciences, USSR, is given

Svetlov (Ref 2) described modern Russ nonpermissible expls, which included Igdanit

Usachev (Ref 3) studied the influence of specific surfaces of AN in Igdanit on their expl props. Sp surfaces were calcd by the formula developed by Usachev

Mel'nikova (Ref 6) investigated the effect of surface active agents on the physicochemical and explosive properties of *Igdanit*. To insure full utilization of the expl props of Igdanit, the Diesel oil must be uniformly distributed on granules or crysts of AN. Since there is no chem bonding betw the oil and the nitrate, the oil tends to exude, producing a stoichiometric imbalance along the charge. Surfactants can provide bridging between the ingredients of Igdanits and thus improve their expl props. The following surfactants were tested: Na dodecylsulfate (I), Na dodecylbenzenesulfonate (II), Na dinaphthyl methanedisulfonate (III) and OP-7, which is a mixt of alkylated phenol derivatives of poly(oxyethylene) (IV). The 0.5–1.0mm fraction of AN was used for coating, which was done at 160°. The amt of surfactant was 0.2% by wt to AN and the amt of Diesel 5.5%. First, the oil-retaining capacity of the control and surfactant-contg samples was detd by pouring excess Diesel oil onto 100g nitrate in a buret and then draining for 24 hrs. Next the samples were tested in a *Dolgov Bomb* (qv), to det the pressure & volume of gases produced on expln and also the amt of toxic gases, such as CO. Finally, heat of expln, extent of detonation, and brisance were detd. The oil retaining capacities were: control 19.6, I 43.1, II 35.5 and III 51.1%. Brisances were: control 21.6, I 26.2, II 26.7, III 27.4 and IV 26.4mm. The heats of expln and the extents of deton were consistently higher for surfactant-modified Igdanits than for the control. The amt of CO was lower for the modified expls than for the nonmodified used for the control. In general, the use of surfactants improved not only the expl props of *Igdanit*, but also its physicochemical props

Plekhanova et al (Ref 7) stated that testing of Igdanit at a Northern Ural bauxite mine showed that on its expln the formation of toxic gases CO & NO_2 was lower than from expln of *Ammonit 6ZhV* and *Detonit 1011*

Rossi et al (Ref 5) described laboratory detn of the amt of noxious gases formed during the expln of *Igdanit* and of other Russ mining expls, such as Ammonit No 6. A special technique used in these detns consisted of the following: A block mining material (marble, granite, or rock salt, was placed in the *Dolgov Bomb* (qv) and an expl chge was inserted in the cylindrical bore of the block. After closing the bore and exploding the chge (initiated by an electric detonator), the pressure of the gas in the closed bomb was continuously measured and corrected for temp effects. Gas samples were withdrawn for analysis and granulometric measurements were performed on the solids. Many parameters (methodical changes in nature of mineral block, charge size, its O-balance, etc) were detd. It was found that for Igdanit contg AN 94–95 & Diesel oil 6–5%, the increased wt and diam of the chge resulted in decreased volume of noxious gases (CO & NO_2) for unit wt of the chge. Igdanit with finely powdered AN gave less toxic gases than that with granular AN. Igdanit with negative O-balance (such as 4.5%) produced more toxic gases than with +0.7% balance. Expln in a marble block of a 20g, 22-mm diam chge with O-balance of –4.49% yielded 54.9 l/kg of toxic gases, while expln of identical chge in granite gave 49.8 l/kg. Expln in rock salt produced 68.2 l/kg of toxic gases. Data for other types of charges were also given and compared with *Ammonit No 6*

Refs: 1) W.S. Khablov, PromStroitel' **39**, No 4, 55(1961) & CA **55**, p 20433(1961) and Explosivst **1964**, 47 2) B.Ya. Svetlov, VzryvnoyeDelo, Sbornik **49/6** (1962); RefZhKhim **1963**, Abstr No 20N/390; CA **61**, 6849(1964) 3) V.A. Usachev, VzryvnoyeDelo, **1966** (60/17), 197–201 & CA **67**, 23700a (1967) 4) G.P. Demidyuk & B.D. Rossi, Ibid **1968**(65/22), 5–15 & CA **70**, 116755z(1969) 5) B.D. Rossi, et al, Ibid **1968**(65/22), 15–22 & CA **70**, 116769g(1969) 6) S.A. Mel'nikova, Ibid **1968**(65/22), 22–

4(1969) 7) A.B. Plekhanova et al, Gornyi-Zh **1968**, 144(9), 40–2 & CA **70**, 30538u(1969)

Investigation of Igdanit in laboratories and in mines was described in the following AddnlRefs, listed under DYNAMITE: 479–98, 582 & 602

Iponit. A new Polish safety expl consisting of granulated porous AN 94 & fuel oil 6%. Iponit with min water content made of fuchsine-coated AN with apparent d 1.01–1.1gm/cc and 1.4mm diam contg 5.5% fuel oil has the best props. The expl can be prepd by manual or mech mixing of both components or by pouring diesel fuel into plastic bags contg AN; then the mixt is poured into borehole
Ref: H. Dabrowski, Cement-WapnoGips **20**, 267–74(1965) (Polish); CA **64**, 12453(1966)

Komex. A new Yugoslavian mining expl described in Ref.
Ref: F. Meznar & S. Uran, Rudasko-Met-Sbornik **1965**, (3–4), 327–37 (in Slovenian) & CA **65**, 6987(1966) listed as No 237 under AddnlRefs for DYNAMITE

Kuro Carlit (Black Carlit). A Japanese industrial expl described in the following Refs:
Refs: 1) K. Ito, KôgyôKayakuKyôkaishi **26** (3), 140–41(1965) & CA **64**, 17344(1966); listed as AddnlRef No 228 under DYNAMITE 2) K. Sakamoto & H. Yoshitoni, Ibid **27**(6), 377–81(1966) & CA **66**, 117543h(1967); listed as AddnlRef No 319 under DYNAMITE

Mansit. An expl mixture consisting of AN 72, Amm picrate 23 & petroleum asphalt 5%
Refs: 1) Blinov, v **2**(1949) 2) PATR **2145** (1955), p Russ 11-L

Metanit and Miporit. Russian permissible expls prepd by enclosing a core of **Ammonit** or **Pobedit** in a rigid casing (sheath) of compressed KCl and ca 1.5% asphalt binder. The purpose of the sheath was to protect expls from dynamic compression and to make them safer for use in gaseous and dusty coal mines. Detonation rate of **Metanits** were 4620 to 4900m/sec. **Miporits** had a higher resistance to dynamic compression and lower deton rates than Metanits. Their core consisted of an Ammonit and microporous rubber. For example, **Miporit No 1** contd 4.5% rubber and its deton rate was 3570m/sec
Ref: N.S. Bakharevich, VzryvnoyeDelo **1966** (160/17), 143–56 & CA **67**, 13424z(1967) (Listed as AddnlRef 310, under DYNAMITE)

Miporit. See above under Metanit and Miporit

Nitrol. A new Yugoslavian mining expl described in Ref:
Ref: F. Metznar & S. Uran, Rudarsko-Met-Zbornik **1965**, (3–4), 327–37 (in Slovenian) & CA **65**, 6987(1966), listed as No 237 under AddnlRefs for DYNAMITE

Nobelit B. One of the Ger expls studied by Lingens from the point of view of its thermal decomp. Its compn is not given in CA of the Ref
Ref: P. Lingens, IndChimBelge **1967**, 32 (Spec No, Pt 3), 515–18 (Ger); CA **70**, 79647t (1969) (See also AddnlRef 400, under DYNAMITE)

Paxit. A Hungarian blasting expl contg AN (of which 60% of grains are smaller than 0.1mm), sensitized with TNT. Exact compn is not given in CA of Ref
Ref: Laszlo Nagy, BanyaszKohaszLapok, Banyasz **1970**, 103(1), 47–52(Hung) & CA **72**, 68817p(1970)

Permon Extra. A Czech expl used for large diam boreholes in surface blasting. It is described by K. Malkowsky & R. Mecir in Rudy (Prague) **12**(9), 345–49(1964) but its compn is not listed in CA **62**, 8924(1965)

Permon Extra 9 and Permon Extra 18. Czech slurry expls described by K. Malkovsky & V. Pesata in Rudy **14**(4), 141–44 & CA **65**, 5293(1966). Its CA is listed as AddnlRef No 265, under DYNAMITE

Pobedity (Pobedites). Accdg to Vaskovskii (Ref 1, p30). They are gray, powdery industrial expls contg the same ingredients as Ammonits with the addn of 8–10% NG. They are not affected by low temps as are the Dynamites and are considered as safe as Ammonits. Vaskovskii lists **Pobedit PU-2, Pobedit No 8 v Obolochke** (sheathed) and **Pobedit VP-1** as permitted for blasting coal in all kinds of mines, while **Pobedit No 6** & **Pobedit VP-2** are permitted for blasting rocks in coal mines. Compns of these expls are not given in Ref 1, pp 25–6 (See also Ref 2)

Bakharevich (Ref 3) studied Ammonits and Pobedits and found that incorporation of fluorides (such as NaF or KF) in lieu of NaCl made the expls safer for use in gaseous mines and also made them more water-resistant

Bakharevich (Ref 4) enclosed in a rigid casing (sheath) of compressed KCl and asphalt binder (1.5%), Ammonits and Pobedits to obtain expls, known as **Metanits**, which did not ignite methane-air mixts and had deton rates of 4620-4900m/sec. The sheath protected the expls from dynamic compression. Another expl described by Bakharevich was **Miporit**

Gel'fand et al (Ref 5) described field tests of water-resistant **Pobedit VP-4** in Karaganda Basin coal mines and stated that the expl proved to be more effective in comparison with three other brands of safety expls. No compns of these expls are given in CA of Ref 5

Refs: 1) Vaskovskii (1957), 25 & 30
2) Vol 3 of Encycl (1966), pp C441 & C454
3) N.S. Bakharevich et al, VzryvnoyeDelo **1966**(60/17), 126–36 & CA **67**, 13423y (1967) (Gallery tests of Pobedits and Ammonits) 4) N.S. Bakharevich et al, Ibid, **1966**(60/17), 143–56 & CA **67**, 13424z (1967)(Development of Metanits and Miporits from Ammonits and Pobedits)
5) F.M. Gel'fand et al, Ibid, (63/20), 177–78 & CA **69**, 11862h (1969) (Results of testing water-resistant Pobedit VP-4)

Russkaya Smes' (Russian Mixture). Blinov (Ref 1) gives its compn as AN 50, TNT 38 & TNX (Trinitroxylene, called in Russ *Ksilil*) 12% (See also Ref 2). Gorst (Ref 3) gives a different compn: PA (Picric Acid) 51.5 & DNN (Dinitronaphthalene) 48.5%

Refs: 1) Blinov, Vol **2**(1949), pp64–5
2) PATR **2145**(1955), p Russ 20-R
3) Gorst (1957), 99
Note: Shilling (1946), p90, calls the compn given by Gorst **Russkii Splav** (Russian Alloy or Fusion)

Russkii Koktel' (Russian Cocktail). An incendiary liquid used during WWII against tanks. It consisted of K chlorate 80 & Nitrobenzene (or Nitrotoluene, or Nitroxylene) 20%
Refs: 1) A. Stettbacher, Protar **10**, 160 (1944) 2) PATR **2145**(1955), p Russ 20-R

Saletrol. A new Polish expl consisting of AN 94–96 & 6–4% mineral oil was tried and found satisfactory in open-pit mining. Mixing of ingredients was conducted *in situ.* Its max expl force was 75–80% that of Gelignite, and its rate of deton 3500–4000 m/sec
Ref: F. Rzepecki, Cement-Wapno-Gips **1969**, (5), 148–51 & CA **71**, 83169b (1969) (Listed as AddnlRef 549, under DYNAMITE)

Securite or Sekurit. A gelatinized O-balanced safety explosive patented recently in Sweden by Wetterholm. It is described in this Vol of Encycl, p D1255-L

Semtinit TV-40 (STV-40). A Czechoslovakian mining expl described by M. Novotny et al in CA **71**, 31944f (1969) (See Addnl-Ref 512 under DYNAMITE)

Skal'nyi Ammonit No 1 (Rock Ammonite No 1). See under Ammonity and in Addnl-Ref 623 listed under DYNAMITE

Sonntag's Explosives (E. Ger). Industrial expls with water-sol salts (AN and $NaNO_3$) having tendency to cake and harden were improved by incorporating 1–5% of a mixt of high-mol-wt fatty alcs and fatty acid-glycerol esters. E.g: AN 60, $NaNO_3$ 10, DNT 15, NGc 10, sol NC 0.3, fatty alc/ester mixt 1.7, woodmeal 2 & iron oxide 1%
Ref: D. Sonntag, Ger (East)P 42947 (1966) & CA **65**, 3662–63 (1966)

Uglenit E-6 (Russ). A coal-mining expl tested together with **Ammonit PZhV-20** in flooded coal faces. Their compns were not given in CA of Ref

Ref: F.M. Gel'fand & V.S. Alipchenko, VzryvnoyeDelo **1967**, (63/20), 209–12 & CA **69**, 11865m (1968) (Reasons for a break in explosions in flooded coal faces)

Zernogranulity (Granulated Granulites). See in this section under Granulity and under AddnlRefs 114 & 602, for DYNAMITE

Dynamitron. An accelerator developed & manufd by Radiation Dynamics Inc, Westbury, NY for the Explosives Division, Feltman Research Laboratories, Picatinny Arsenal, Dover, New Jersey 07801. This 3 MEV particle accelerator is capable of operating in the electron mode at a voltage of ½–3 MEV, producing a beam current from 50 microamps to 10 milliamps; and in the positive ion mode in the same voltage range, producing a beam current to 2 milliamps. X-rays, gamma rays & neutrons, in addn to electrons, can be generated by accelerating either deuterium ions or protons onto the appropriate target. This facility is used to study radiation damage to single crystals and for gross irradiation of expl samples

Ref: Expl Lab Newsletter, No 4, pp 3–4 (Jan 1969)

E

E (Abbr). In such usage as M53E4, designates an experimental item, whereas M53 without attached E designates a standardized item. In jato unit nomenclature, designates an extruded double-base solid proplnt
Ref: Glossary of Ord (1959), p104-L

"E" (Explosive)(Japan). Blend of AN (Ammonium Nitrate) 40 and TNAns (Trinitroanisole) 60%
Ref: R.A. Cooley et al, "Japanese Explosives", PBL Rept **53045** (1945)

E-4 Great Enzian. One of the German guided missiles used during WWII. See under Guided Missile (Gesteuerte Geschoss) in PATR **2510** (1958), p Ger 84-R

E-4 HEXA. See under Ersatzsprengstoffe (German Substitute Explosives) in PATR **2510** (1958), p Ger 44 (Table)

E-7. A solvent-extruded composite proplnt developed in USA during WWII. It was described as Unit No 593 in conf Propellant Manual SPIA/M2(1962)

E-8 Rocket Launcher (Amer). A light-weight, portable, dependable launcher which fires 16 riot-control-agent canisters in each of four volleys, producing a rapid build-up of a large chemical cloud. Highly effective against ambush and in defensive operations, it is a versatile method for projecting riot-control agents. It was manufd at the Edgewood Arsenal, Maryland, as of 1968 and probably later
Ref: Anon, Ordnance, **53** (July-August 1968), p30-L

E-19 (Incendiary Bomb)(Amer). It was described in OSRD **4784** (PBL5828)(1945) and OSRD**4748a** (PBL12095)(1945)

E-24 (Cartridge)(Amer). A riot-control cartridge, known as *Handy-Andy*, which is intended mainly for dispersing mobs, controlling riots, and delivering riot-control agents at ranges longer than those of hand-thrown grenades. The Handy Andy can be fired from the hand or from a flare pistol or M79 grenade launcher. Burning time is about 14 seconds. It was manufd at the Edgewood Arsenal, Maryland as of 1968 and probably later
Ref: Anon, Ordn **53**, (July-August 1968), p30-L

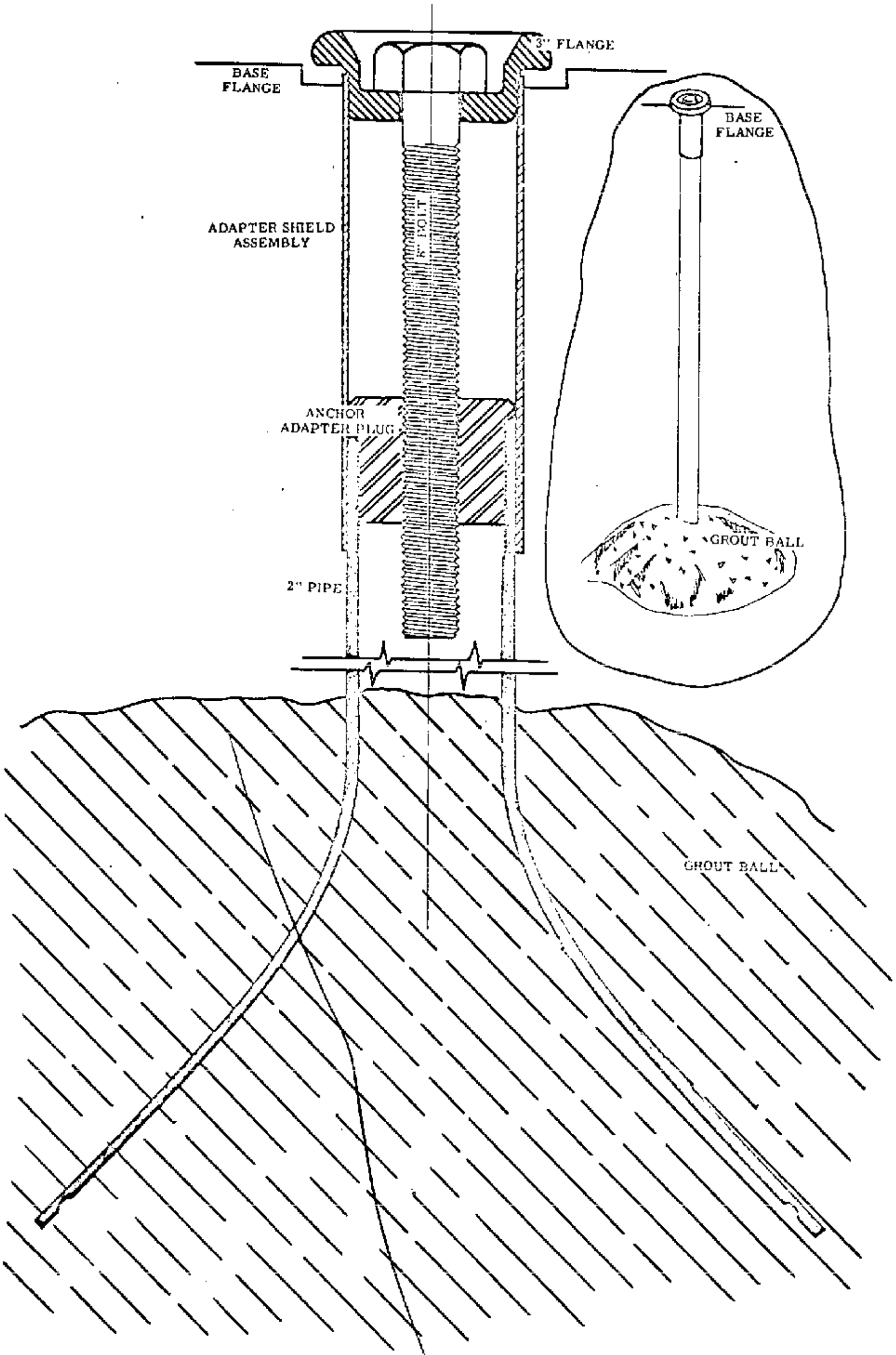
Eagle Duck and Eagle Rifle Powders (Amer). Black sporting powders manufd by the DuPont Co at the end of the 19th century. Their compns are not given
Ref: Daniel (1902), 210 (under DuPont de Nemours)

Eales patented in 1887 (FrP 184326) a safety fuse contg as a core a mixture of BkPdr and NC, previously treated with a soln of K chromate
Ref: Daniel (1902), 433 (under Mèches de sûreté)

Earle & Reid proposed at the end of the 19th century to make covers of safety fuses of NC mixed with castor oil
Ref: Daniel (1902), 432 (under Mèches de sûreté)

Earle & Reid proposed in 1895 (FrP 251985) an expl prepd by mixing 1 part of nitrated linoleine + ricinoleine with 9 parts NC
Ref: Daniel (1902), 670 (under Reid & Earle)

Earth Anchor EAW-20 – The earth anchor was initially designed to anchor the catapult shuttle truck to the earth in the installation of short airfields for tactical support (SATS). The anchor consists of two primary components, the anchor tube assembly and the explosive cartridge assembly. The anchor tube assembly consists of the following subassemblies: 1) Anchor tube – a steel tube,



Soil cross-section portraying EAW-20 Earth Anchor principle. Note cutaway showing tension take-up capability

Fig E.1

2" dia and 56" or 138" long, which has internal threads at the top end and 6 machined external vertical grooves 8 inches in length at the bottom end 2) Ogive – a pointed steel cap to aid in penetrating the soil. The cap is assembled to the anchor with a snap ring 3) Anchor adapter – a threaded closure plug for the anchor tube with a threaded hole in the center for the take-up bolt 4) Adapter-assembly – a hollow sleeve which fits over the anchor between the take-up bolt and the anchor adapter which transmits the restraining force of the anchor to the component to be anchored (See Fig E1) 5) Take-up bolt – a bolt which threads into the anchor adapter. When tightened this bolt holds the adapter assembly to the anchor adapter and the item to be anchored

The "explosive cartridge assembly" consists of approx 0.1 lb of RDX as mechanical firing device and a firing lanyard. An auxiliary charge of 0.1 lb of RDX is also available to supplement the main charge for use in areas where the formation of a satisfactory camouflet is difficult. This charge is contained in a small canister which is taped to the main charge

To install, the anchor is driven into the ground with a sledge hammer or a pneumatic tool. The explosive charge is pushed to the bottom of the anchor with a push rod and then exploded. A rapidly curing cement (grouting material) is then poured into the anchor and tamped to fill the camouflet enclosing the anchor tines (See Fig E1)

Ref: Anon, Landing Force Information Letter No 20, Instructions for Installation of EAW-20 Earth Anchor, Marine Corps Landing Force Development Center, Quantico, Virginia

Earth Cratering. See Vol 3 of Encycl, pp C552 to C555, which includes "Crater, Cratering and Cratering Effect"; "Cratering Charge or Cratering Explosive"; and "Cratering Effect Tests"

Addnl Ref: J.A. Dennis, "Earth Cratering with Aluminized Explosives" (U), Engineering Research and Development Laboratories Technical Report **1762-TR**, 18 Dec 1963(Conf). PicArsn Library Accession No C104641

Earth Crustal Studies. In recent years, there has been a vast increase in seismic studies devoted to: 1) the earth's compn and origin, and 2) the detection of nuclear explns. This work can be conducted on both land and water, but the latter is preferred for reasons of convenience and economy. Special expls and techniques are required in either case and, since DuPont Co's experience in this field is outstanding, it is advisable to consult the Co prior to undertaking any seismic earth crustal studies

Ref: Blasters' Hdb (1966), 358

Earth Displacement Test. See "Cratering Effect Tests" in Vol 3, pp C554 & C555-L

Note 1: This test was developed in France under the name *Essai dans la terre* by the Commission des Substances Explosives and described in MP**15**, 229–31(1909–1910).

Note 2: The Ger method developed before WWII for testing the efficiency of bombs and land mines on expln under ground was conducted by burying a test item in earth at a predetermined depth and then exploding it. The volume, in cubic meters, of the resulting crater, gave an approx idea of the power (strength) of the item

Ref: 1) O.W. Stickland, PB Rept **925** (1945), Appendix 7 2) Fedoroff et al, PATR 2510(1958), p Ger 39-R

Earthquake Bomb. A British aeroplane bomb, weighing 6 tons (12000 lbs) developed during WWII by Commodore Huskinson. The bomb sank the German Superbattleship Tirpitz and destroyed submarine pens at Brest, France

The British later developed a 22000 lb bomb (11 tons). Both bombs resembled a torpedo in appearance

Ref: 1) Anon, Army Ordn **28**, (May–June 1945) 2) H. Scullin & C.H. Morgan, ArmyOrdn **29**, No 152, 248–49(1945)

Earthquake Powder (Brit). Expl mixture patented in 1898–1899 by Curtis & Durnford (Engl P 17878) and manufd by Curtis & Harvey, Ltd. Its compn was KNO_3 78–81 & charcoal(contg ca 56% volatiles) 22–19%

mixed with 1/3 by wt pure sulfur. It was authorized for use for blasting coal in gaseous and dusty mines. It is actually a sulfurless BkPdr
Refs: 1) Daniel (1902), 233 2) Clift & Fedoroff **2** (1943), p E1

Earth Test. Same as "Earth Displacement Test"

Ease of Ignition by Flash Test (British). A portion of 3g of an explosive is placed in a short 3/8 inch diam test tube, and a piece of safety fuse is inserted in the middle of it. After igniting the upper end of the fuse, it is observed whether the expl deflagrates when the fuse burns down to it
Ref: Marshall **2**(1917), p434

Eastern Laboratory of the DuPont Co. A scientific research laboratory established in 1902 at Gibbstown, New Jersey. It was called "Eastern" because of its connection with "Eastern Dynamite Co." A detailed description of its activities is given in the ref
Ref: Blasters' Hdb (1966), pp7–11

Ébarbage des poudres (French). Trimming, scraping and cleaning of propellants. It is one of the operations of *lissage* or *plombaginage* (glazing)
Ref: Pepin Lehalleur (1935), p 306

Ebeling, Franz (1881–1942). German scientist specializing in mining explosives
Ref: Dr Rontz, SS **36**, 12–13(1942)

E–Boat (Enemy Boat). British high-speed motorboat 60–100 ft long, mounting two or four torpedo tubes, AA Guns, MG's (Machine Guns) and equipped with Depth Charges and Smokeproducing Apparatus. Such boats were also known as "Mosquito Boats" or "PT-Boats"
Ref: "Webster's New Collegiate Dictionary", Merriam Co, Springfield, Mass (1953), 550 (under "Motor Torpedo Boat")

EBW (Exploding Bridge Wire) or **EW** (Exploding Wire), also known as **EEW** (Electrically Exploding Wire). See Exploding Bridge Wire (EBW) or Exploding Wire (EW) in Vol 6

EBWR Core Alloy: U(uranium) 93.5, Zr (zirconium) 5.07 & Nb(niobium) 1.5%

F.G. Baird et al described in AtomicEnergyCommissionRept CF-58-10-43(1958) & CA **54**, 2028(1960) an explosion which occurred while dissolving the alloy in boiling concd nitric acid

EC Blank Fire Powder. See under EC Powders (EC Propellants) in this Volume

EC Dynamite. A Dynamite manufd in England betw 1883 and 1886. It was a mixture of Dynamite No 1 (NG 75 & kieselguhr 25%) and Na carbonate in quantity not exceeding 3%
Ref: Daniel (1902), 234

Echo. A Norwegian Ammonal type explosive proposed by Berger (Ref 1). Accdg to the late Dr Kostevich (Ref 2), it was similar to the mixture contg AN 86, stearic acid 8, and Al 6% used in Russia before WWI as a filler for projectiles
Refs: 1) N.W. Berger, SS **1**, 150 & 169(1906) 2) M.M. Kostevich, Buenos Aires, private communication (1954)

Echo. A 100 ft aluminum coated inflated sphere, 0.0005" thick and 150 lbs in wt, designed to serve as a communication satellite by reflecting radio waves. The spheres before inflation are packed in a 28 in diam container, together with 4 lbs of water held in a plastic bag. Upon ejection, the container opens and the sphere begins to inflate due to the presence of residual air inside and the water which vaporizes. The carrier vehicle is the *Thor-Delta*
Ref: F.I. Ordway & R.C. Wakeford, "International Missile and Spacecraft Guide", McGraw-Hill, NY (1960)

(Ref 1) it consisted of AN 75, ferro silicon (of 95% purity) 16, Al (pdr) 2 and "ipposino" (dried, finely powdered horse manure) 7%. A similar expl AN 75–80, Al 2–3, ferrosilicon 14–16 & ipposino 5–7% is listed in the book of Mangini (Ref 2) under the name of **Escho.** It was claimed that Escho was used for military purposes both in Italy and in France

Refs: 1) R. Molina, "Esplodenti", Hoepli, Milano (1930), 342 2) A. Mangini, "Esplosivi", Pàtron, Bologna (1947), 225

Éclair. A French detonating cord ("cordeau détonant"), consisting of a 5mm diam core of PETN (9.9g per meter) and two white threads serving as pilots. The core is covered with a thin cellulosic film and fabric. Its external diam is 5.5mm and detonation rate 6355 to 6745m/sec. Earlier French cords are listed in Encycl, Vol 3, p C530-L

Refs: 1) A. LeRoux, MP **32**, 203–04(1950)
2) M. Dutour, MAF **24**, 578 & 583(1950)

Eclipse (Powder). A mixture of PA (Picric Acid), litharge (yellow lead monoxide, PbO), sulfur & charcoal. It was considered as very dangerous to handle (Ref, p230). A similar mixt exploded on June 22, 1887 at the plant of Roberts, Dale & Co at Cornbrook, near Manchester causing considerable damage (Ref, p622)

Ref: Daniel (1902), 230 & 622

EC-NACO. Navy single-base proplnt manufd at the Naval Propellant Plant, Indian Head, Maryland. Its compn and props are given in conf Propellant Manual SPIA/M2 (1969), Unit No 1062

Economics in Commercial Explosives. Accdg to the French book of Vennin, Burlot & Lécorché (Ref 1, pp 198–200) it is required to know in estimating the economics of an explosive, not only the cost to the manufacturer, but also the cost to the consumer of explosive in blasting or other operations

The cost to the manufacturer ("prix de revient" in French) includes the following:

1) Cost of primary materials, which consist of substances (such as toluene, cellulose, glycerin, glycol, etc), acids for nitration (such as nitric & sulfuric), alkalies (such as soda ash, NaOH, etc) serving for neutralization of acids and of other materials
2) Cost for grinding the materials, screening them, nitration, neutralization & washing, centrifuging, crystallization, flaking, cartridging, handling, storing in magazines, and transportation
3) Cost of power, heating of buildings and of lighting
4) Cost of buildings and installations and of their maintenance
5) Amortization of buildings and installations
6) Interest on the invested capital and payment on patents
7) Cost of management
8) Laboratory costs for chemical and physical analyses of primary materials, acids and final products
9) Insurance against fire and accidents

The cost to the consumer of explosive usually includes the following:

1) Storing of expl in a magazine
2) Handling and transportation from magazine to the place of blasting
3) Drilling of boreholes
4) Cost of blasting caps and fuses and cost of labor for fitting cartridges with them
5) If blasting is done by means of electric caps, the cost of such caps as well as wiring and of blasting machines
6) Cost of labor for loading the cartridges into boreholes, tamping and blasting
7) Removal of blasted materials (such as ore, coal, rocks, etc) and loading for transporting to places of storing. Cost of transportation and storing
8) Total cost of ton of explosive and cost per cubic meter (or per ton) of blasted material
9) Cost of management in blasting operations
10) Cost of accident insurance

Berliner and staff (Ref 3, p7), discussed the "economy of commercial explosives", as follows:

It is easier to select an explosive to give good execution than it is to select one to give utmost economy. Good execution often can be obtd from several different expls, but a study of costs usually shows that a certain one is decidedly more economical. Economy is not only the cost of the expl per 100 pounds. The true basis for comparison is the cost of an expl per ton or cubic yard of properly blasted material. To this may be added the costs of drilling, handling and loading the broken ore or rock, and some other factors

It must be remembered that each mining operation has its peculiar blasting problems; an explosive suitable for one mine is not necessarily best adapted for another mine. All of the factors that go to make up explosives' economy must be considered as a whole. Operations with larger equipment can handle larger material more economically than can smaller equipment. The breakage necessary to give the best economy must also be studied. See also Refs 2 & 4

Refs: 1) Vennin, Burlot & Lécorché (1932), 198–200 2) C. Taylor, "Chemical Engineering Economics", McGraw-Hill, NY (1948) 3) J.J. Berliner and Staff, "Explosives", Pamphlet, NY, p7 4) Collective, ChemEngrg **60**, No 1, 167–200(1953) (Symposium on Cost Estimation)(Eight papers with numerous references)

Economic Mobilization. The process of preparing for and carrying out such changes in the organization and functioning of the national economy as are necessary to provide for the most effective use of resources in a national emergency

Ref: Glossary of Ord (1959), p 104-L

Economic War Potential. That segment of the economic capacity of a nation which can be used for purposes of conducting war

Ref: Glossary of Ord (1959), p 104-L

E–Cord. See under Detonating Cords in Vol 3 of Encycl, p D104-L

EC Powders (EC Propellants) (Brit and Amer). Accdg to Marshall 1 (Ref 2, pp 39 & 47), the early attempt (beginning ca 1852) of an Austrian Officer von Lenk to produce a "smokeless powder" was unsuccessful because it was too violent in its action and either damaged the gun barrel or burst it open. Von Lenk's powder consisted of NC prepd by nitrating unpulped cotton by the Schönbein process, but purified more thoroughly. The dried material was loaded unpressed in the guns and that is why it burned so quickly. F. Abel of Waltham Abbey improved in 1865 the purification of NC because he pulped it previously to washing. This material, although more stable than von Lenk's powder was too-fast-burning for use as a proplnt, even in unrifled weapons (Ref 2, p41)

A slower-burning, nearly smokeless proplnt, was prepd in 1864 by Captain and later Major E. Schultze of Prussian Artillery by simply impregnating small grains of wood with saltpeter and then drying the product. This was not as successful as the product prepd by him in 1865 by nitrating purified grains of wood (lignose) with nitric acid, and impregnating the stabilized product with saltpeter alone or in mixture with Ba nitrate. Accdg to Cundill's "Dictionary of Explosives", as quoted in Ref 2, p 47, Schultze's product contd ca 20% of unnitrated lignin. Schultze's product was still too violent for rifles, but was found to be quite suitable for shotguns. F. Volkmann acquired in 1870 the Austrian rights of Schultze's invention and in 1871 made the product still slower burning by partly gelatinizing the grains with ether-alcohol mixture. This became known as **Collodin**, which is described in Vol 3, p C394-L of our Encycl. A company was formed in England in 1868 to work the Schultze's invention and in 1869 a factory was established at Eyeworth, in the New Forest. After Griffiths improved the process, the powder achieved great success, and by 1881 became very popular with sportsmen on account of its light recoil and absence of smoke as compared with Black Powder. In 1883 Schultze started a factory in partnership with Voltz and Lichtenberger at Hetzbach in

Hesse-Darmstadt, Germany (Ref 2, p48)

The next successful smokeless proplnt was invented at the Explosives Co of Stowmarket in England and was protected by Engl patent No 619 taken out in 1882 by W.F. Reid and D. Johnson. This was called **EC** (Explosives Company) **Powder**, and consisted of Nitrocotton mixed with the nitrates of K and Ba with the addn of coloring-matter and small quantities of some organic compds. The NC, which consisted of soluble and insoluble types, was gelatinized to the extent of 50–80% by ether-alcohol to which a small amt of camphor was added to facilitate the gelatinization

A separate company was formed in England to utilize the invention and a factory was constructed at Green, near Dartford in Kent. The powder manufd there was too quick for use in rifled weapons, but was suitable for use in shotguns. It became very popular with sportsmen for the same reason as was mentioned above under Schultze's Powder (Ref 2, p 48)

The compn of one of the earliest British sporting EC Powders was given by Daniel (Ref 1, p 235) as follows: NC (soluble) 27.95, NC (insoluble) 28.32, unnitrated cellulose 3.15 and nitrates of K & Ba 37.80 parts. An improved British sporting EC Powder was described by Marshall (Ref 2, p 327) among the 30-grain powders as consisting of NC 79.0, K nitrate 4.5, Ba nitrate 7.5, camphor 4.1, woodmeal 3.8 and volatiles 1.1%. The same powder was described in Barnett (Ref 3, p 86). This formula was further modified and the powder became 36 grain while manufd by the Imperial Chemical Industries Ltd at the time of publication (1932) of Marshall's Vol 3 (Ref 4, p 96)

Note 1: 30-grain powder signifies that the charge required for an ordinary 12-bore sporting cartridge is 30 grains, and this quantity occupies the same space in the cartridge as 82 grains of Black Sporting Powder. The same discussion applies to 36-grain powder, etc (Ref 2, p 326)

Note 2: EC Powder, as well as Schultze Powder belongs to the type of "Bulk Powders" because they are loaded by bulk or because, for the same bulk they have about the same power as BlkPdr(Ref 5, p289). A detailed discussion on this subject is given in Vol 2 of Encycl, p B322)

In the USA, the EC Powders began to be manufd near the end of the 19th century by the "EC and Schultze Gunpowder Co, Ltd at Oakland, New Jersey and this continued until the plant was destroyed in August 1900 by fire caused by lightning (Ref 1, p 235)

Davis in the section describing "Bulk Powder" (Ref 5, p 289) lists the following powders with compns similar to EC Powders:

Table E1

Components	Percentage		
	No 1	No 2	No 3
NC	84.0	87.0	89.0
(%N in NC)	(13.15)	(12.90)	(12.90)
K nitrate	7.5	6.0	6.0
Ba nitrate	7.5	2.0	3.0
Starch	–	–	1.0
Paraffin oil	–	4.0	–
Diphenylamine	1.0	1.0	1.0

Manuf of Bulk Powders, which included EC Powders, by British and American methods, was described by Marshall (Ref 2, pp 324–26) and Davis (Ref 5, pp 289–92)

Accdg to Davis (Ref 5, p 289), bulk powders used for sporting were also suitable for use as bursting charges in hand grenades and as proplnts for blank fire cartridges. Later, when dense colloided propellants were invented and used in artillery projectiles, EC Powders were used occasionally in the igniter charges which set fire to the slower burning colloided proplnts. Accdg to Marshall (Ref 2, p 334), some fast-burning bulk powders were suitable for use as proplnts for trench howitzers

Bain (Ref 6) examined at Picatinny Arsenal, the Amer "EC Blank Fire Powder" as a bursting charge for fragmentation hand grenades

Composition and properties of American *EC Blank Fire Powder* are given by Tomlinson and Sheffield in Ref 9, as follows: NC(13.25%N) 80.0, Ba nitrate 8.0, K nitrate 8.0, Starch 3.0, DPhA (Diphenylamine) 0.75) & Aurine 0.25%. It is manufd by partially gelatinizing NC using either ethanol with acetone or butyl acetate with benzene, followed by incorporation of other ingredients. The process is controlled so that the final dry product passes a No 12

US Std Sieve and is retained on a No 50 Sieve
Properties of EC Blank Fire Powder:
Oxygen Balance to CO_2 +5%, to CO 25%
Density, loose, used for loading 0.40g/cc
Brisance by Sand Crushing in 200g Bomb 46.8g (vs 48g for TNT)
Explosion Temperature, dec in 5 mins at 200°
Friction Pendulum Test – snaps by steel shoe
Heat Test at 100°, % Loss – 2.0 after 1st 48 hrs, 0.2 after 2nd 48 hrs and no expln in 100 hrs
Heat Test at 120° – salmon pink after 150 mins, red fumes at 300+ and then explodes
Hygroscopicity – 6.2% gain at 30°C and 90% RH
Impact Sensitivity, BurMinesApparatus, 2kg wt 19cm (comparable to PETN)
Initiation Sensitivity – minimum detonating chge 0.22g Hg
International Test, 75°C – % Loss in 48 hrs 1.8%
Loading – loose
Storage – wet
Velocity of Detonation – not detd
Used in Grenades, Caliber .30 Blank

US Military Specification requirements and tests are discussed in Specification MIL-P-20466. Calorimetric method for determination of Aurine is given in MIL-STD-286A (1960)

As was already mentioned, EC Powders and other quick-burning formulations were unsuitable for use as proplnts in rifled weapons. In order to obtain proplnts suitable for such purpose it was required to prepare proplnts considerably slower burning than EC or Schultze's Powders. This was achieved by using formulations with NC in which the cellulosic structure was completely destroyed by treating pulped NC with colloiding (gelatinizing) agents. Historical development of such proplnts is described in Vol 3 of our Encycl, pp C398 to C403, under the title "Colloiding Agents and Colloidal Propellants"

EC Powders also are described in Refs 7&8

Refs on EC Powders: 1) Daniel (1902), 234–35 2) Marshall **1**(1917), 47–8, 324–27 & 334–35 3) Barnett (1919), 86 4) Marshall **3**(1932), 96 & 98 5) Davis (1943), 289–92 6) C.J. Bain, PATR **822**(1937) 7) P. Tavernier, MP **32**, 243(1950) 8) ArmamentEngrg(1954), 38 & 43 9) W.R. Tomlinson Jr & O. E. Sheffield, PATR **1740**, Revision 1(1958), reproduced as Pamphlet in 1967 & 1971, **AMCP 706–177**, by the US Army Materiel Command, Washington, DC, 20315

EC Pulver. One of the older Ger sporting proplnts: Collodion Cotton 28.0, Guncotton 26.0, Ba & K nitrate 38.0, camphor 2.0, woodpulp 4.0, gelatinizer 0.5 & moisture 1.5%
Refs: 1) H. Brunswig, "Das rauchlose Pulver", Gruyter, Berlin (1926), p 134 2) PATR **2510**(1958), p Ger 39-R

ECRASITE (Ekrasit). An explosive of compn never revealed was used by the Austro-Hungarian Army beginning in 1892 and ending in 1918, for loading the artillery shells, petards and demolition charges. It was manufd by the Kubin and Siersch, near Pressburg. There is great confusion about its compn, as can be seen from the following.

Accdg to Daniel (Ref 1), Molina (Ref 4, p 356) and Pepin Lehalleur (Ref 7), Ecrasite consisted of Ammonium Trinitrocresylate mixed with K nitrate in such a proportion that complete combstn could be obtd. It melted below 100° and was less powerful than PA (Picric Acid). Pepin Lehalleur also stated that mixt of Ammonium Trinitrocresylate with Ammonium Nitrate was used as the French Mining Explosive known as **L'explosif de mine français, type C, n° 1b**

Accdg to Chalon (Ref 2), as quoted by Marshall (Ref 3) and Davis (Ref 8), Ecrasite was Ammonium Trinitrocresylate, prepd by treating 2,4,6-Trinitro-m-cresol with ammonia

Vennin, Burlot & Lécorché (Ref 5, p 454) were of the opinion that Ecrasite was Ammonium Trinitro-m-cresylate, which they called *Le crésylate d'ammoniaque* or simply *Crésylate*. The same authors called on p 457, the compn consisting of Crésylate 60 & Mélinite (PA) 40%, as *Crésylite No 2*. Its props are given in Ref 11, p C557-R

Under the name **Ecrasite 60/40**, Molina (Ref 4, p 391), gave the mixt of PA 60 & Trinitro-m-cresol 40% (See also Ref 12)
Note: Here is evidently an error and compn must be PA 60 & Ammonium Trinitro-m-cresylate (called in French the "Crésylite") 40%. Another error is on p 327, where Molina assigned the name Cresilite to Trinitrocresol instead of its NH_4 salt

Accdg to Stettbacher (Refs 6 & 8) Austrian expl **Ekrasit** did not contain any Ammonium Trinitro-m-cresylate but was either straight

PA or PA mixed with ca 10% of Dinitronaphthalene or Dinitrobenzene. These substances were added in order to obtain expls melting below 100°, since mp of PA is 122.5°. As far as known, the Austrian Ekrasit melted at ca 86°

A great expln of a magazine contg a large quantity of Ekrasit took place at Kiev, Russia during its Austro-Hungarian occupation in 1918

Hallett (Ref 9) proposed using Ammonium Trinitrocresylate (which he called Ecrasite) in admixture with NG and other ingredients, as a substitute for Dynamites, claiming that such mixts are less susceptible to extremes of temperature than are Dynamites

Giua (Ref 11) stated that Ecrasite is Ammonium Trinitrocresylate and that in addn to using it straight in Austria for loading shells, it was used in various mixts with AN

Refs: 1) Daniel (1902), 236–38 2) Chalon (1911), 133 (See Ref 11, p XII) 3) Marshall **2**, (1917), 557 4) Molina (1930), 356 & 391 5) Vennin, Burlot & Lécorché (1932), 454 & 457 6) Stettbacher (1933), 287 7) Pepin Lehalleur (1935), 274 8) Davis (1943), 169 9) Ch.S. Hallett, BritP 575147 (1946) & CA **41**, 4312 (1947) 10) Stettbacher (1948), 77 11) Giua, Trattato **VI** (1)(1959), 316 12) Encycl of Expls **3** (1966), p C557-R

Ectijet (Amer). An Army vehicle, 20 x 10 feet in size, designed by the Marine Co of Orlando, Florida to skim about a foot above land or water at speeds up to 50mph

Ref: Anon, Army Research and Development, Washington, DC, 10025, Vol 2, No 12 (1961) p 19

EDAP. Abbr for Ethylene Dioxyaminoperchlorate, described in Vol 6 of Encycl

EDD. Ger abbr for Ethylenediamine Dinitrate, known also as DIAMIN

EDD (or EDDN). Amer abbrns for Ethylenediamine Dinitrate, described under Ethylenediamine and Derivatives in Vol **6**

EDGEWOOD ARSENAL. A Government-owned and -operated Installation of the US Army Munitions Command (MUCOM), which is, in turn, part of the US Army Materiel Command (AMC). It was established on May 4, 1918 as Chemical Corps home at a place known as Gunpowder Neck, located in Maryland between the Gunpowder and Bush rivers. It was charged with the production of toxic chemicals for use in WWI and the development of defensive items for US troops serving in Europe. Known at the beginning as "Edgewood Arsenal", the name was changed in 1942 to the "Chemical Warfare Center", and in 1946 to the "Army Chemical Center". Its basic mission, however, remained the same – chemical research, development and procurement

Its activity was at a maximum during WWII, when about 7000 military and 8000 civilians were employed (Ref 2). In Ref 1 it was stated that in March 1943 a peak was reached with 10750 civilians employed

A general Army reorganization of the Chemical Corps in 1963 saw the return to the name of "Edgewood Arsenal"

In a reorganization approved on July 7, 1966, Edgewood Arsenal was designated as the Army's commodity center for all chemical weapons and defense equipment research and development. In this category, the Arsenal's former administrative control over the biological laboratories at Fort Detrick, Md, was relinquished and Fort Detrick was established as a separate commodity center for biological weapons and defense research and development

Three subposts fell in 1968 under the administrative control of Edgewood Arsenal. They were Pine Bluff Arsenal, Ark; Rocky Mountain Arsenal, Colo; and Weldon Spring Army Chemical Plant, Mo. The first two specialized in various aspects of chemical testing, procurement, manufacture, and research, whereas the Weldon Spring facility was readied for the production of a chemical defoliant for use in Vietnam

As of 1968, Edgewood Arsenal employed 3800 civilian employees, and over 1600 military personnel.

Among the items manufd at the Arsenal in 1968, the following, among others, are listed in Ref 2: E8 Portable Rocket Launcher (See in this Vol); E24 Cartridge, known as "Handy-Andy" (See in this Vol); Dispenser M106, known as "Mity Mite" (See in this Vol); Flame-Thrower M106, Self-Propelled (See under F's); Pop-up Adapter-Projector XM42 (See under P's) and Protective Mask M17A (not described by us)

For more detailed information on activities of the Arsenal, see Ref 2

Note 1: Accdg to Col W.D. Hodges, Ordn **54**, Sept-Oct 1971, pp 132–35, Edgewood Arsenal was combined in July 1971 with Aberdeen Proving Ground, Maryland

Ref: Expls & Pyrots **4**(10), Oct 1971

Note 2: Aberdeen Proving Ground (APG) was listed in Vol 1 of Encycl, p A2-R, but its function was not described. APG consists of ranges, test courses, laboratories, and other facilities which sustain a materiel testing mission and nine tenant activities on the installation. The major missions performed at APG are:

1) plans & directs the test and evaluation of Army weapons & equipment
2) research in propulsive force systems
3) weapons systems evaluation
4) conducts static, dynamic & environmental tests on ordnance materiel
5) performs res & develop on limited warfare items
6) conducts individual training in supply & maintenance of ordnance materiel

A principal testing laboratory located within the APG complex is the Development and Proof Services Laboratory (Ref 3)

Refs: 1) Anon, Ordnance **52**, 550 (May-June, 1968) 2) Anon, Ordnance **53**, 30–32(July-August, 1968) 3) **AMCP 70–1**, 695(1968)

Edinorog (Unicorn) Howitzer (Russian). An egg-shaped, smooth-bore, muzzle-loaded weapon invented in 1750 by an Artillery Officer, later General, Count P.I. Shuvaloff (1711–1762), who reorganized Russian Artillery during the reign of Empress Elizabeth, daughter of Peter the Great. Edinorog (pronounced Yedinorog) was used successfully by the Russian Army during Seven-Year War with Prussia (1756–1763), when Berlin was captured. The weapon was also used during the Napoleonic Wars (1799–1814) and even later until it was replaced, after the Crimean War (1854–1856) by rifled guns

Refs: 1) F. Pavlenkoff, "Encyclopedic Dictionary" (in Russ) St Petersbourgh(1918), p 726 (Edinorog) and p 2959 (Shuvaloff, P.I.) 2) M.E. Serebriakoff, "Interior Ballistics", listed in Vol 3 of Encycl, p XV. Engl translation of Dr M.E. Nekrassoff, pp 23–4

Edmunds of Calcutta, India, patented in 1893 (EnglP 23416) several expl mixtures, among them **No 1**: KNO_3 37.50, PA (Picric Acid) 9.38, $KClO_3$ 18.75, KCl 12.50, sulfur 3.12 Prussian Blue 3.12 & sawdust 15.64%, plus a caoutchouc solution as a binder; **No 3**: KNO_3 24.25, PA 15.10, $KClO_3$ 24.25, sulfur 8.10, Prussian Blue 4.02, nitrated sawdust 18.28 & silica 6.00%, plus a caoutchouc solution as a binder

Ref: Daniel (1902), 238

EDNA. Abbr for Ethylenedinitramine, known also as Haleite, in recognition of its development as a military expl by the late Dr G.C. Hale of Picatinny Arsenal. See under ETHYLENEDIAMINE AND DERIVATIVES in Vol 6

EDNAFIVES. Combinations of EDNA and Fivonite in various proportions with addn of 1% catechol. Usually EDNA 50 or 60 and Fivonite 50 or 40%. *Fivonite* is Tetramethylolcyclopentanone, also known as Nitropentanone. It is described in Vol **6** as Fivonite. EDNAFIVES are described in Vol **6**, under Ethylenedinitramine (EDNA)

EDNATOLS. Combinations of EDNA and TOL(TNT) in various proportions. Their prepn and properties are described in Vol 6, under ETHYLENEDIAMINE AND DERIVATIVES

EDTA. Abbr for (Ethylenedinitrilo) tetraacetic Acid described in Vol 6

EDX. Hercules Powder Co's experimental composite solvent-extruded proplnt, developed during WWII. It is described in Vol 3 of Encycl, pp C468-R & C469-L under "Solvent-Extruded Composite Propellants". See also "Summary of Technical Reports, Div 8, NDRC, Washington, DC (1946), p 101-L (Table)

EED. See Electro-Explosive Devices in this Vol

EEW. (Electrically Exploding Wire) or **EW** (Exploding Wire). See Exploding Bridge Wire (EBW) in Vol 6

EF (Poudre) (Fr). See Poudre EF

Effect of Compression on Sensitiveness of Initiation. See Vol 3 of Encycl, p C492-L, under Compression, Effect on Sensitiveness of Initiation

Effect of Crystal Size and Crystallographic Properties of Explosives on Explosion. See under Crystallization of Explosives in Vol 3 of Encycl, p C568

Effect of Explosions and Table of Distances for Storage of Explosives. Experiments have been carried out at various times and places to ascertain the distances at which explosions will produce a specific effect accdg to the quantity of explosive. In the French experiments conducted in 1907 by Lheure, different quantities of various expls were exploded in the open and at various distances. A number of little screens were erected, so arranged that the same degree of force would cause each of them to fall back. It was thus possible to ascertain the distances at which the same effects were produced. It was found that the distances could be approximately calcd, assuming that the distances would be proportional to the square root of the weight of the charge

This can be expressed by equation: $d=k\sqrt{c}$, where d = distance (in meters); c = weight of charge (in kg); and k = constant, depending on the nature of the expl and the sort of damage considered. For HE's causing the breaking of window panes and wooden walls, k was considered to be about 10, while for Gunpowder (BkPdr), k was ca 7. The figures in the American Table of Distances developed accdg to Marshall after the tests conducted, beginning 1910, did not agree very well with the above equation. For small quantities of expls the distances were more nearly proportional to the cube root of the wt of expl, while for very large quantities the variation of the distance was more nearly proportional to the straight increase of the weight

It is somewhat doubtful whether the equation really represented the results obtd under practical conditions. The records of explns did not indicate that any distinction should be drawn between BkPdr and HE's, when quantities of several tons were concerned (Ref 1, pp 621–22)

Accdg to DuPont Blasters' Hdb (Ref 2, p 457), the American Table of Distances for Storage of Explosives was originally formulated in 1914 by "The Institute of Makers of Explosives", after painstaking research. It has been the subject of constant study and periodic revision since that time and undoubtedly embodies the most reliable weight and distance data available. These distances vs weights, given as Table 30–1 of Ref 2 and Notes attached to Table are reprinted here (See also "Buildings and Other Structures at Ordnance Establishments" in Vol 2, pp B320 & B321 and "Damage Effects of Organic High Explosives" and "Damage Potential of Air and Ground Blast Waves" in Vol 3, pp D3 & D4)

Refs: 1) Marshall 2(1917), 621–22
2) Blasters'Hdb(1966), p 457 giving Table 30–1 and Notes

TABLE 30-1

American Table of Distances for Storage of Explosives

As Revised and Approved by The Institute of Makers of Explosives, September 30, 1955

EXPLOSIVES		DISTANCES IN FEET WHEN STORAGE IS BARRICADED				EXPLOSIVES		DISTANCES IN FEET WHEN STORAGE IS BARRICADED			
Pounds Over	Pounds Not Over	Inhabited Buildings	Passenger Railways	Public Highways	Separation of Magazines	Pounds Over	Pounds Not Over	Inhabited Buildings	Passenger Railways	Public Highways	Separation of Magazines
2	5	70	30	30	6	500	600	340	135	135	31
5	10	90	35	35	8	600	700	355	145	145	32
10	20	110	45	45	10	700	800	375	150	150	33
20	30	125	50	50	11	800	900	390	155	155	35
30	40	140	55	55	12	900	1,000	400	160	160	36
40	50	150	60	60	14	1,000	1,200	425	170	165	39
50	75	170	70	70	15	1,200	1,400	450	180	170	41
75	100	190	75	75	16	1,400	1,600	470	190	175	43
100	125	200	80	80	18	1,600	1,800	490	195	180	44
125	150	215	85	85	19	1,800	2,000	505	205	185	45
150	200	235	95	95	21	2,000	2,500	545	220	190	49
200	250	255	105	105	23	2,500	3,000	580	235	195	52
250	300	270	110	110	24	3,000	4,000	635	255	210	58
300	400	295	120	120	27	4,000	5,000	685	275	225	61
400	500	320	130	130	29	5,000	6,000	730	295	235	65
6,000	7,000	770	310	245	68	75,000	80,000	1,695	690	510	165
7,000	8,000	800	320	250	72	80,000	85,000	1,730	705	520	170
8,000	9,000	835	335	255	75	85,000	90,000	1,760	720	530	175
9,000	10,000	865	345	260	78	90,000	95,000	1,790	730	540	180
10,000	12,000	875	370	270	82	95,000	100,000	1,815	745	545	185
12,000	14,000	885	390	275	87	100,000	110,000	1,835	770	550	195
14,000	16,000	900	405	280	90	110,000	120,000	1,855	790	555	205
16,000	18,000	940	420	285	94	120,000	130,000	1,875	810	560	215
18,000	20,000	975	435	290	98	130,000	140,000	1,890	835	565	225
20,000	25,000	1,055	470	315	105	140,000	150,000	1,900	850	570	235
25,000	30,000	1,130	500	340	112	150,000	160,000	1,935	870	580	245
30,000	35,000	1,205	525	360	119	160,000	170,000	1,965	890	590	255
35,000	40,000	1,275	550	380	124	170,000	180,000	1,990	905	600	265
40,000	45,000	1,340	570	400	129	180,000	190,000	2,010	920	605	275
45,000	50,000	1,400	590	420	135	190,000	200,000	2,030	935	610	285
50,000	55,000	1,460	610	440	140	200,000	210,000	2,055	955	620	295
55,000	60,000	1,515	630	455	145	210,000	230,000	2,100	980	635	315
60,000	65,000	1,565	645	470	150	230,000	250,000	2,155	1,010	650	335
65,000	70,000	1,610	660	485	155	250,000	275,000	2,215	1,040	670	360
70,000	75,000	1,655	675	500	160	275,000	300,000	2,275	1,075	690	385

Note 1—"Explosives" means any chemical compound, mixture, or device, the primary or common purpose of which is to function by explosion, *i.e.*, with substantially instantaneous release of gas and heat, unless such compound, mixture, or device is otherwise specifically classified by the Interstate Commerce Commission.

Note 2—"Magazine" means any building or structure, other than an explosives manufacturing building, used for the permanent storage of explosives.

Note 3—"Natural Barricade" means natural features of the ground, such as hills, or timber of sufficient density that the surrounding exposures which require protection cannot be seen from the magazine when the trees are bare of leaves.

Note 4—"Artificial Barricade" means an artificial mound or revetted wall of earth of a minimum thickness of three feet.

Note 5—"Barricaded" means that a building containing explosives is effectually screened from a magazine, building, railway or highway, either by a natural barricade, or by an artificial barricade of such height that a straight line from the top of any sidewall of the building containing explosives, to the eave line of any magazine, or building, or to a point twelve feet above the center of a railway or highway, will pass through such intervening natural or artificial barricade.

Note 6—When a building containing explosives is not barricaded, the distances shown in the Table should be doubled.

Note 7 "Inhabited Building" means a building regularly occupied in whole or in part as a habitation for human beings or any church, schoolhouse, railroad station, store, or other structures where people are accustomed to assemble, except any building or structure occupied in connection with the manufacture, transportation, storage or use of explosives.

Note 8—"Railway" means any steam, electric, or other railroad or railway which carries passengers for hire.

Note 9—"Highway" means any public street or public road.

Note 10—When two or more storage magazines are located on the same property, each magazine must comply with the minimum distances specified from inhabited buildings, railways, and highways, and in addition they should be separated from each other by not less than the distances shown for "Separation of Magazines," except that the quantity of explosives contained in cap magazines shall govern in regard to the spacing of said cap magazines from magazines containing other explosives. If any two or more magazines are separated from each other by less than the specified "Separation of Magazines" distances, then such two or more magazines, as a group, must be considered as one magazine, and the total quantity of explosives stored in such group must be treated as if stored in a single magazine located on the site of any magazine of the group, and must comply with the minimum distances specified from other magazines, inhabited buildings, railways and highways.

Note 11—The Institute of Makers of Explosives does not approve the permanent storage of more than 300,000 pounds of commercial explosives in one magazine or in a group of magazines which is considered as one magazine.

Note 12—This Table applies only to the manufacture and permanent storage of commercial explosives. It is not applicable to transportation of explosives, or any handling or temporary storage necessary or incident thereto. It is not intended to apply to bombs, projectiles or other heavily encased explosives.

Note 13—All types of blasting caps in strengths through No. 8 cap should be rated at 1½ pounds of explosives per 1,000 caps. For strengths higher than No. 8 cap, consult the manufacturer.

Note 14—For quantity and distance purposes detonating fuse up to 60 grains per foot should be calculated as equivalent to nine pounds of high explosives per 1,000 feet. Heavier case loads should be rated proportionately.

Effective Calculated Calorific Values of Propellants. If it is assumed that for a certain muzzle velocity and a given projectile, the product of the charge weight and calorific value of a proplnt is constant, then by knowing the calorific value and wt of one proplnt, it is possible to calculate the calorific value of a second proplnt of a similar nature (if its chge wt had been previously detd experimentally). For example, if for one proplnt the values were 820kcal/kg and wt 4.3kg and for a 2nd proplnt Xkcal and 6.2kg, respectively, then

$$X = \frac{820 \times 4.3}{6.2} = \frac{3526}{6.2} = 570\text{kcal/kg}$$

This may be considered as the "effective calorific value" and it differs from the value detd in a calorific bomb, which is usually higher, eg 690–700kcal/kg, for the 2nd proplnt cited above

In calculating the life of a gun barrel, it was considered in Germany to relate to the "effective calorific value", more than to the values obtd in a calorific bomb (See also under "Energy Content of a Propellant Charge" and under "Erosion of the Bore")

Refs: 1) O.W. Stickland, "General Summary of Explosive Plants", PB Rept No **925**(1945), pp 16 & 82 2) B.T. Fedoroff et al, PATR **2510**(1958), pp Ger 39 & 40

Effective Fragment. In terminal ballistics, a fragment whose mass, velocity and form, upon impact with the target, are such as to enable the fragment to accomplish the desired effect

Ref: Glossary of Ord (1959), p 104-R

Effective Oxygen. See under Available Oxygen in Vol 1 of Encycl, p A515-R

Effectiveness or Efficiency of Explosives. According to Davis (Ref 4, p 23), "the *effectiveness* of an explosive depends in large part upon the rate at which its energy is liberated." For estimating the *total energy* of an expl, a test in the manometric bomb probably supplies the most satisfactory single indication. It should be remembered, however, that total energy and actual effectiveness are different matters

In the book of Vennin, Burlot & Lécorché (Ref 2, p 193), it is stated that in order to ascertain the practical efficiency of expls, it is not sufficient to test them for brisance, power and velocity of detonation, but it is necessary to conduct tests directly against the objects it is designed to destroy. For example, if the explosive is intended to be used against concrete, a known wt of expl should be exploded against a concrete wall and the damage recorded. Similarly, it might be tested against a rail, armor or inside of a projectile, such as in fragmentation test

The term *practical efficiency* is called in France *effet utile* (also known as "travail pratique", "rendement pratique en travail d'un explosif" or "travail spécifique relatif" (Compare with "Coefficient d'utilisation pratique", described in Vol 3 of Encycl, p C390)

It is stated in the book of Pepin Lehalleur (Ref 3, pp 43, 64 & 77) that "effet utile" is the same as "potentiel" and can be detd by one of the following methods: French modification of Trauzl test, mortar test, ballistic pendulum test, or cratering test

Vennin et al (Ref 2, pp 166–69) in the chapter entitled "Étude de l'Effet Utile" lists the following equation developed by Mallard and Le Chatelier (Ref 1, pp 461–65)

$$Q_u = Q - \Sigma(at + \frac{b}{2}t^2), \text{ where}$$

Q_u = Quantity of heat transformed into work by detonation

Q = Potential of an explosive

t = Final temp of the gases of expln after expansion (at atmospheric pressure)

a and b = constants calcd for some expls in Ref 1, p 464

In Table on p 169 of Ref 2 are given values of Q_u and Q in kcal/kg as follows: Q_u for PA 724 and for NG 11.28; Q for PA 737 and for NG 1478. (See also "Economy in Commercial Explosives")

Refs: 1) ? Mallard & ?. Le Chatelier, MP **2**, 461–65 (1884–1889) 2) Vennin, Burlot & Lécorché (1932), 166–69 & 193 3) Pepin Lehalleur (1935), 43, 64 & 77 4) Davis (1943), 23

Effect of Replacement of Air in Explosive Charges by Nonexplosive Liquids on Detonation Velocity. See Vol 4 of Encycl, p D256-L

Effects of Blast and Shock Waves of Detonation (or Explosion) Produced on Structures. See Vol 4, pp D256 & D257

Effects of Boundary Rarefaction on Impulse Delivered by Explosive Charges During Detonation (or Explosion). Described in the paper of Taylor listed in Vol 4, p D257-R

Effects of Explosive Properties on Cratering is discussed in Refs 1 & 2

Refs: 1) R.L. Fischer, D.V. D'Andrea & D.E. Fogelson in "Effects of Explosive Properties on Free-Surface Displacement Pulses and Crater Dimensions", Report of Investigation **RI 7407**, July 1970; USBurMines, 4800 Forbes Ave, Pittsburgh, Pa 15213
2) Expls & Pyrots 3(10)(1970)

Effects of Inert Components on Detonation (or Explosion). Described in the paper of Copp & Ubbelohde, listed in Vol 4, p D257-R

Effects of Ionizing Radiations on Propellants and of the X-Irradiation of Ammonia and Hydrazine. Preliminary study by Lucien of NASA (Ref) was conducted in the following manner: NH_3 and N_2H_4 were X-irradiated at 100kr to det the effects of space environment on the storability of proplnts. At pressures $P > 100$mm and dosages >45kr, 0.1% of NH_3 decompd to give N, H and an IR spectrum different than for NH_3. At 1295mm and 104kr, 0.5% of NH_3 decompd to a gas contg N and H. IR spectra on 3 fractions of the product distd *in vacuo* showed NH_3 in the 1st two fractions and N_2H_4 in the 3rd. Liq NH_3 at 115kr gave 0.5% of condensable gas of N_2H_4 and 0.4ml of a permanent gas contg 90% N and 10% H. The P decreased during irradiation and then increased at const slope; the P diminished to a value > initial P with the source off, and the cycle re-occurred when the source was reactivated. At 15mm and 590kr, 3% N_2H_4 was converted to 1% NH_3, a noncondensable gas, N, H and an unidentified product. During irradiation, the P diminished to 0.5 the initial P. The irradiated product, fractionated into 0°, −35°, −90° and −196° traps, showed 20% by vol of N_2H_4 at −35°, 75% of the product at −90°, and NH_3 at −196°. P-time curves indicated formation of mols larger than N_2H_4. The possibility of larger N chains is remote, but HN_3 and azides are possible

Refs: 1) H.W. Lucien (Natl Aeron & Space Adm, LewisResCenter, Cleveland, Ohio) NASA **Tech Note D-1193**, 9 pp(1962); CA **56**, 11871(1962) (Abstract by J.H. Modeen)
2) H.W. Lucien & M.L. Pinns, NASA **Tech Note D-2452**, 13 pp(1964) & CA **62**, 4812 (1965)(X-irradiation of hydrazine & 1,1-dimethylhydrazine)

Effects of Materials on the Properties of Explosives. See Compatibility of Explosives with other substances in Vol 3 of Encycl, pp C461-L to C462-L

Effects of Polymers on Explosives. See Compatibility of Explosives with other Substances in Vol 3 of Encycl, pp C461-L to C462-L and PA Plastec Rept **33**, "Compatibility of Explosives with Polymers" (April 1968) by N.E. Beach & V.K. Canfield

"Effects of Metallic Additives on the Deflagration of Condensed Systems". Title of a paper by A.A. Zennin et al, in the 12th SympCombstn(1968), pp 27–35

Effects of the Physical Structure and the State of Aggregation on the Detonating Capacity of Explosives. Described in the paper of Apin & Bobolev, listed in Vol 4, p D257

Effects Produced in Air, Earth and Water During Detonation (or Explosion). See Vol 4 of Encycl, pp D257-R & D258-L

Effects of Sheaths on Gases from Explosions in Coal Mines. See under Coal Mining Explosives, Permissible, p C454 of Vol 3 of Encycl, and the following addnl Ref: B. Lewis, USBurMines **RI 4502**(1949), 92pp & CA **43**, 6826–27(1949) (Effect of sheaths upon gaseous products from permissible expls)

Effects of Temperature on Explosives. Accdg to Marshall (Ref 2), expls at high temps are considerably more sensitive; conversely their sensitiveness is reduced on cooling them. In experiments conducted in France by Kling & Florentin (Ref 1), expls, as well as their detonators were cooled to temp −80°C by means of solid carbon dioxide and acetone, or to about −190° with liquid nitrogen. Table E2 lists the results of the tests

During these experiments it was found that cooling of detonators reduced their efficiencies. For example, when a detonator was cooled to −190°, it was necessary for it to contain 1 g MF in order to detonate PA of d 1.0, whereas with the detonator at ordinary temp, 0.25g sufficed

Refs: 1) A. Kling & D. Florentin, MP XVII, 145(1913) 2) Marshall **2**(1917), 430 3) M.M. Jones & H.J. Jackson, "Heat Sensitization of Explosives", Explosivstoffe 9, 2–4(1969) (A study of the Memory Effect in Some Common Explosives)

Effet brisant (Fr). Shattering Effect or Brisance. See Vol 2, pp B265 ff

Effet d'éclatement (Fr.) Bursting (or Splintering) Effect. See Fragmentation Tests in Vol 3, pp C345 ff

Effet Neumann (Fr). See Munroe-Neumann Effect (known in the US as Shaped Charge Effect) in Vol 4, p D442-R to D454-L

Effet perforant (Fr). Armour-Piercing Effect

Effet utile (Fr). Practical Efficiency. See under Effectiveness or Efficiency of Explosives

Efficiency of Explosives. See Effectiveness or Efficiency of Explosives

Efficiency of Propellants. Accdg to investigations conducted in Europe before and after WWI, of the total energy of a proplnt, only from 15 to 40% is actually utilized as kinetic energy to the projectile. Of the remainder the greater part remains in the

Table E2
Sensitiveness of Explosives at Various Temperatures

Explosive	Density	Grams of MF Required		
		Room Temp	−80°C	−190°C
MF (Mercuric Fulminate)	1.20	<0.25	–	1.0
Blasting Gelatin	–	<0.25	0.25	1.0
Cheddite No 2	1.0	0.25	0.75	>2.0
PA (Picric Acid)	0.90	0.25	1.0	>2.0
Guncotton	0.40	0.25	0.25	>2.0

proplnt gases as heat and kinetic energy, but the barrel also absorbs a considerable proportion as heat. An investigation conducted in 1908 with the German rifle M98-S gave the following distribution: Bullet velocity energy 32.7, Bullet rotation energy 0.2, Gases in cartridge case 43.7, Heating of barrel 22.5 & recoil 0.9% (Refs 1 & 2)

The distribution of the energy liberated by the combustion of Cordite Mk VII rifle bullet cartridge had been calcd by Hardcastle (Ref 3) and by others (Refs 4 & 5). It is presented here in Table E3

Table E3

	Ft-lbs	Percent
Carried away by bullet as muzzle energy	2300	31.4
Carried away as gas energy to muzzle	254	3.5
Carried away as heat	260	3.5
Carried away as energy of rotation	35	0.5
Imparted to barrel by friction	260	29.2
Imparted to barrel by hot gases	1880	
Recoil	11	0.1
Heating cartridge case	5	0.1
Ejected at muzzle	2315	31.7
Total	7320	100.0

Similar calcns regarding the Mark VI cartridge were done by Kelly (Ref 4)

Refs: 1) C. Cranz & R. Rothe, SS **3**, 303 (1908) 2) Marshall **1**(1917), 320–21 3) J.H. Hardcastle, JRoyArtillery **45**, 157 (1918) 4) F.H. Kelly, Arms and Explosives **1919**, p 58 5) Marshall **3**(1932), 94–5 6) A.M. Ball, "Solid Propellants", AMC Pamphlet **706–175**(Sept 1964)

Efficiency, Thermodynamic. A laboratory method for determining the thermodynamic efficiency of HE's is described by Felts et al (Ref). Eighteen neat cement cubes were blasted in a steel plate box and the breakage energy was evaluated by comparison with drop crusher results using Rittinger's (CA **20**, 1477), Kick's, and Bond's (CA **46**, 5898) theories. It was necessary to extrapolate the particle-size-distribution curves well below the usual cut off size. Efficiencies of the blasting process indicated that the values may be as high as 72% when the 3 different theories are used. Results obtd by use of Rittinger's & Bond's theories approx more closely the true efficiencies since Kick's theory gives so little wt to particles in the smaller range size

Ref: L.L. Felts et al, TransAmInstMiningMetEngrgs **205**, TechPubl No **4213A** (in MiningEngr **8**, No 3, 318–22)(1956) & CA **50**, 9019(1956)

Efflorescence. The property of some hydrated crystals to change upon exposure to air to a mealy or powdery substance either on the surface or throughout the material. This is due to partial or complete loss of water of crystallization

Ref: "The American College Dictionary", Random House, NY (1952), 384-L

EFR. Hercules castable rocket proplnt described in conf Propellant Manuals SPIA/M2 (1962) as Unit No 635 and in SPIA/M2 (1969) as Unit No 1070

Egelit. One of the names for *Miedziankit,* which consisted of K chlorate 90 & petroleum 10%

Refs: 1) Marshall, Dictionary (1920), 61 2) Stettbacher (1948), 91 3) PATR **2510** (1958), p Ger 113-L

Egg for Manipulation of Acids. It was mentioned on p A88-L of Encycl, Vol 1, where two references are given. A more comprehensive description is given in Marshall **1**(1917), pp 129–30

Egoutage des poudres [French for Drainage (or Dripping) of Propellants]. When the *trempage* ("water-drying procedure") for removing volatile solvent from proplnt grains is used, the water adhering to the grains can

be partially removed by the égoutage. This procedure was followed by *séchage*, which means "drying" which was done by heat
Ref: L. Butin, MP **19**, 342–47 (1922)

Ehrhardt patented in 1864 & 1865 (Engl P's 1694 & 402) expl mixts consisting of KClO$_3$, KNO$_3$, charcoal, tannic acid and rosin
Ref: Daniel (1902), 238

EI. Abbr for "End of Injection" (of liquid rocket propellant)
Ref: F. Bellinger et al, IEC **38**, 167 (1946)

Eimite (Explosive). A Black Powder Modification described by H. Hassmann in PATR **2515**(1957), entitled "Evaluation of Eimite as a Substitute for Black Powder in Artillery Primers". The title was (U), while the rept was (C), now declassified. Eimite is a stoichiometric mixt of atomized Mg, recrystallized KNO$_3$ & 20–40% NC (13.15% N). It is manufd by using conventional solvent propellant processing equipment. The extruded strands are cut to fit lengthwise in the primer body. Eimite is a satisfactory igniter material for artillery-type primers and can be a suitable substitute for BlkPdr. See also US Spec **AEZ–54**, Rev 1 (16 Aug 1960)

Eindrahtzünder. Ger for One Wire Igniter or Primer
Ref: Beyling & Drekopf (1936), 220

Einfachezünder. Ger for Simple Igniter or Primer
Ref: Beyling & Drekopf (1936), 172, 174 & 177

Einheitspulver (EP). Ger for Standard Propellant, corresponding to Brit Service Propellant (Gun or BkPdr)

Einheitspulver (EP). A German *standard* propellant of later period of WWII, called "Unit Powder" by Pike (Ref 1). It was one of the "G" Pulvern (DEGDN proplnts), which contd NC & DEGDN, 1.5% K nitrate and 3% hydrocellulose. Its calorific value was 710–730 kcal/kg. It was introduced in 1944 as the "Service" proplnt for all ammunition in order to minimize the differences in ballistics previously obtd when proplnts of the same formulations were manufd at different plants. The incorporation of K nitrate and of hydrocellulose was claimed to give much more uniform interplant ballistics of Einheitspulver
Refs: 1) H.H.M. Pike, CIOS Rept **31–68** (1945), p 6 2) B.T. Fedoroff et al, PATR **2510**(1958), p Ger 190

Einheitspulver R61. One of the German WWII rocket proplnts: NC (12.5% N) 59.8, EGDN (Ethyleneglycol Dinitrate) 35.3, hydrocellulose 1.5, EtPhUrethane 1.1, DPhUrethane 0.8, IG Wax E 0.4, Mg oxide 0.2, K nitrate 0.6 & Acardite 0.3%
Ref: Anon, "Summary Technical Reports of Div 8, NDRC, Vol 1(1946), p 121

Einheitsverbindungen (Ger). See Vol 3 of Encycl, p C327, under "Clathrates and Other Inclusion Compounds"

Einmann U-Boot (One-Man U-Boat). A German weapon of WWII which was also known as **Einmann Torpedo** (One-Man Torpedo). It consisted of a small submarine to the bottom

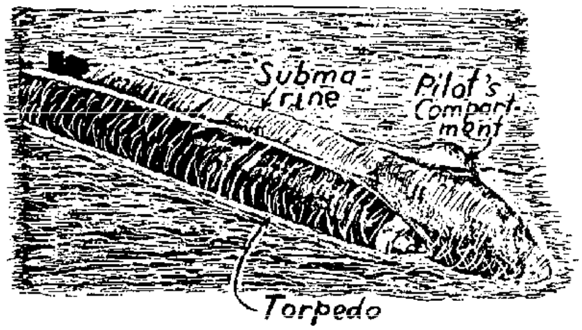


FIG E2 EINMANN U–BOOT

of which a torpedo was attached by shackles. (Fig E2). The ensemble, released from a "mother" ship, was propelled by an electric motor operated by a storage battery, or in later models by a small internal combustion engine. These were located in the rear section of submarine, while in the front section was a compartment for one pilot. When the pilot brought the submarine to within a fairly short distance of the target (such as an enemy ship, dock, warehouse, etc), the torpedo was aimed by lining up the submarine with the target and then firing. Immediately after firing the torpedo, the pilot turned about and headed in the direction of his "mother" ship
Refs: 1) Anon, Field Artillery Journal **34**, 505(1944) 2) B.T. Fedoroff et al, PATR **2510**(1958), p Ger 211-L

Eisler (Poudre). A mining explosive consisting of Na nitrate, sulfur & sugar
Refs: 1) Daniel (1902), 238 2) Molina (1930), 177

Eismine 42 oder Flascheneismine (Icemine or Flask Icemine). One of the German landmines used during WWII. It was in the shape of a wide-mouth bottle. It contd 5lb, 5oz of *Gelatine-Donarit* and was provided with a pull or pressure igniter. Originally intended for use under ice, it was also adopted for use as an A/P (Antipersonnel) Land Mine, by encasing it in a concrete sheath contg shrapnel slugs
Refs: 1) Anon, "German Explosive Ordnance", **TM 9–1985–2** (1953), pp 281–82 2) B.T. Fedoroff et al, PATR **2510**(1958), p Ger 107-R

EJ (Propellants). American solvent-extruded propellants developed during WWII at the Allegheny Ballistics Laboratory and at the Radford Development Department of the Hercules Powder Co. They contd K perchlorate-carbon black filler in a double-base proplnt matrix

The following varieties were developed:
EJA, known, accdg to Mr. G.D. Clift as **Pigseye Powder**, is described in Vol 3 of Encycl, pp C468-R and C469-L, under Solvent-Extruded Composite Propellants. Its ballistic props were remarkably insensitive to changes of temperature and pressure, but it produced some smoke. Analytical procedures are given in Ref 2

EJB was a modification of EJA in which filler ($KClO_4$ + C black) was reduced from 65% to 30%, making its compn! $KClO_4$ 26 & C black 4% in a double-base proplnt matrix. In this proplnt the smoke was reduced to a minimum, while changing the ballistic properties only to a small extent

EJC. It was Hercules castable rocket proplnt of classified comp, listed in conf Ref 4 as Unit No 636 and in Ref 5 as Unit No 1071. It does not actually belong to the group of EJ proplnts developed during WWII, because they are solvent-extruded composite proplnts, while EJC is castable

EJE. A propellant consisting of NC (13.1% N) 26.0%, NG 22.0, EC 2.0, carbon black 7.0, $KClO_4$ 43.0, containing MgO, 0.5 & Mg stearate 0.5

EJX. Designation of any experimental Hercules Co solvent-extruded proplnt contg K perchlorate. As soon as the compn becomes standardized, another letter is substituted for X

To the same group of solvent-extruded proplnts belonged the following three formulations, which were not designated as EJ:
BBP (Blastless Bazooka Powder) or **T–4** is described in Vol 2 of Encycl, p B26-R and in Vol 3, pp C468-R and C469-L
EDX is described in Vol 3 of Encycl, pp C468-R & C469-L
MJA is described in Vol 3 of Encycl, pp C468-R & C469-L
EFR is described in Vol 3 of Encycl, pp C468-R & C469-L
Refs: 1) ERL (Explosive Research Labora-

tory), Bruceton, Pennsylvania, "The Properties and Applications of EJ Powders", OSRD Rept No **4053**(1944) 2) L. Pauling, "Procedures for the Analysis of EJA Powder", OSRD **4581**(1945) 3) R.A. Connor, Chief of Div 8, "Summary Technical Report of Div 8, NDRC", Vol 1, Washington, DC (1946) 4) Anon, "Propellant Manual" SPIA/M2(1962), Unit Nos 635 & 636(Conf) 5) Ibid (1969), Unit Nos 1060, 1070 & 1071

Ejecta. Jet fragment ejected essentially beyond primary target plate in the testing of shaped charges
Ref: Glossary of Ord (1959), p 105-L

Ejecting Projectiles (Ger). Special projectiles which ejected incendiary missiles on approaching a target (such as an airplane) were developed during WWII at Krümmel Fabrik of Dynamit AG but did not come to the manufacturing stage. These projs contd several hollow steel cylinders, each of which was filled with an incendiary mixture consisting of Ba peroxide, powdered Al and Fe. A charge of ca 15g of HE was required for ejecting each cylinder from the proj and to impart to it a velocity of 1000m/sec. Each of these cylinders burned in flight and if one of them hit a combustible object (such as a gasoline tank of an airplane) a fire, or even expln could take place
Refs: 1) O.W. Stickland et al, "General Survey of Explosives Plants", PB Rept No **925**(1945), Appendices 2) B.T. Fedoroff et al, PATR **2510**(1958), p Ger 103-L

Ejector. Mechanism in small arms and rapid fire cannons which automatically throws out an empty cartridge case from the breach or receiver. Also, the open aft portion of a jet-engine shroud, which permits the escape of cooling air
Ref: Glossary of Ord (1959), p 105-L

Ekrasit. Same as Ecrasite

EL-389A, EL-389B, EL-389C. Compositions consisting of NG, DNT, TNT and stabilizer. **A** – 55/31.12/13.33/0.55; **B** – 60/27.6/11.8/0.6, **C** – 50/35/15/0.5. The NG used was a commercial grade containing 25% Ethylene Glycol Dinitrate or 30% Diglycerin Tetranitrate to depress its freezing point. These explosives were used as hose fillers for minefield clearance
Ref: P.C. Keenan & D. Pipers, NAVORD Rept **87–46** (1946) "Table of Military High Explosives", Navy Dept BuOrd, Washington, DC

Elastic Detonation Wave. See Vol 4 of Encycl, p D684

Elasticity and Young's Modulus of Elasticity. Elasticity is the inherent property in bodies by which they recover their former figure or state after the *force* (stress) of external pressure, tension, or distortion have been removed (as for instance elasticity of gases, rubber, etc). Any force or distribution of forces which acts upon a body and is balanced by equal and opposite forces in the body is, in general, termed as a *stress*, although the term is more particularly applied to the *force per unit area* acting upon the body. The change in size per unit size, or the change in some dimension per its unit, produced by the stress is called a *strain*. For each substance and for each kind of strain there is some limit beyond which *Hooke's Law* does not apply. This is known as *elastic limit*
Note: Hooke's Law was defined by English scientist Robert Hooke (1635–1703) as: the stress applied to stretch or compress a body is proportional to the strain, or alteration in length, so produced

Within the elastic limit the substance returns to its original state when the stress is removed. In this case the *elastic coefficient*, which is stress divided by strain is constant and is known as the *modulus of elasticity*

If, in case of solids, the elastic limit is exceeded, then the body does not return to its original state when the stress is removed. The **Young's Modulus of Elasticity** (known also as *Longitudinal Elasticity*) is, as was defined by English physicist Thomas Young (1773–1829), the force (dynes per sq cm or pounds per sq in) that will permanently deform a material by stretching, bending, or twisting. The stretching modulus may be expressed as:

$M = \text{Stress}/\text{Strain} = mg\lambda/\pi r^2 s$, where

M = Young's Modulus; s = elongation produced by weight, m, in a wire (or bar) of length, λ, and cross section of radius, r

It must be noted that a pull which produces an elongation will at the same time produce a lateral contraction, so that the wire (or bar) will become thinner. The ratio of lateral contraction to the longitudinal extension is called *Poisson's Ratio*

For most materials, the value of Young's Modulus is very great, which means that even for the large stretching force only a slight elongation is obtd

By using two very long wires suspended from the same support and attaching a constant wt to one wire and varying loads to the other, the elongation may be measured directly by means of a scale attached to one wire and vernier to the other

A more delicate and precise instrument is the *Searle's Extensometer*, described in Ref 2, p 68 (Compare with "Compressibility of Explosives and of Propellants" in Vol 3 of Encycl, p C491)

Refs: 1) Hackh's Chemical Dictionary (1944), p 413-R (Hooke's Law); p 669-L (Poisson's Ratio); p 919-L (Young's Modulus) 2) S.G. Starling & A.G. Woodall, "Physics", Longman's, Green & Co, London (1950), pp 67–84

Elayl. One of the names for Ethylene (Ethene)

"Elcord" Seismic Delay Units of DuPont Co are precision ultra-short delay devices for seismic prospecting to match the deton velocities of the expl columns to the velocities of the surrounding formation when using sequential shooting technique. Its description is given in Blasters' Hdb (1966), p 109–10

Electra. See Elektra

Electret. The electrical equivalent of a permanent magnet. When a block of dielectric material, such as carnauba wax, is allowed to be solidified in a strong electric field it acquires a permanent state of electrostatic polarization (orientation of molecules) in the direction of the field. Swann (Ref 3) traced the mathematical consequences on the assumption that an electret consists of: a) a distribution of polarization which decays with time and b) a distribution of surface & volume chge which disappears accdg to ohmic conductivity having no relation to the decay of polarization

Refs: 1) Hackh's Dict (1944), 296-L 2) F. Gutmann, RevModPhys **30**, 457–72 (1948) 3) F.W.G. Swann, JFranklinInst **250**, 219–48 (1950) (On certain matters pertaining to electrets) 4) Kansas University, Third Quarterly Progress Report Research Studies on Electrets Project **152B-O-M19** (15 Aug to 15 Nov 1951)

Electric. Charged with or capable of developing *electricity*. This is a form of energy that produces magnetic, chemical, thermal and radiant effects, generated by friction, induction, or chemical means

Ref: Hackh's Dict (1944), 296–97

Electric Actuator. Same as Electric Squib

Electric Aerial Burst Fuzes, used by the Germans during WWII to initiate bursting charges in bombs, while they were still in flight, were described in Refs 1 & 2. The most important of them were:

a) ElZtZ 59A consisted of two igniter bridges connected directly to two plungers w/o any intervening condensers or resistances. The bridges were fired as soon as the bomb or flare left the aircraft, thus initiating pyrotechnic delay trains which provided the aerial burst functioning. The shorter delay was fired from the A plunger (See Fig E3), the longer delay from the B plunger. If both plungers were charged the short delay functioned, and if only B was charged, the longer delay functioned. Fuze ElZtZ(59A) was of identical construction but twice as large (Ref 1, p 172 & Ref 2, p Ger 2)

b) ElZtZ(59B) used in General Purpose 250kg Bomb and in some parachute flares differed from 59A & (59A) fuzes by having three igniters instead of two. The igniter under the A plunger (See Fig E4) was in such a position as to give a 12-sec delay, while the other two igniters were under the B plunger and gave 41 & 58-sec delay (Ref 1, 172–3 & Ref 2, p Ger 2)

C) ElZtZ 69CII consisted of Al cylinder contg igniter bridge (See Fig E5) which on release of the bomb from the plane fired the loose BkPdr chge which in turn ignited the

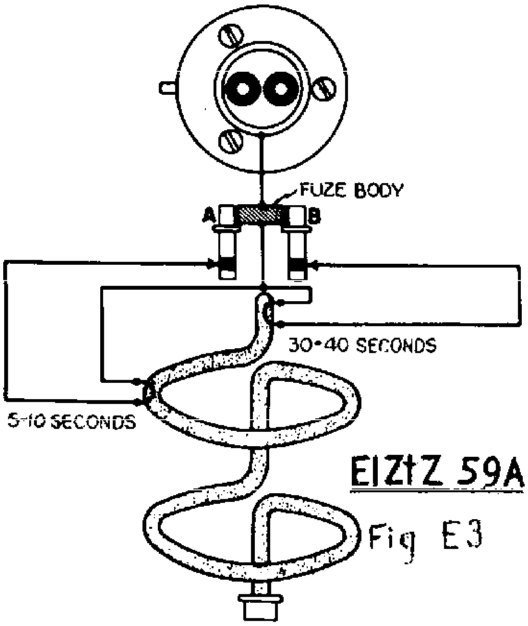


Fig E3

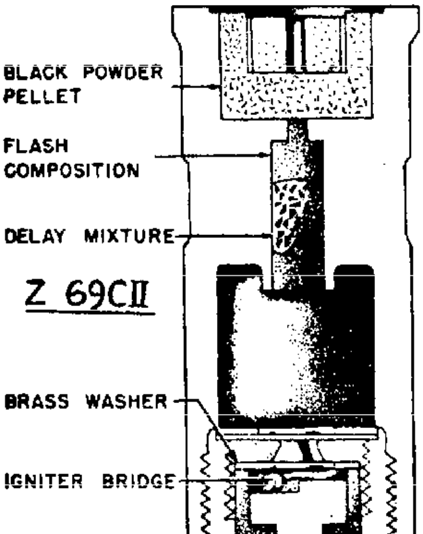


Fig E5

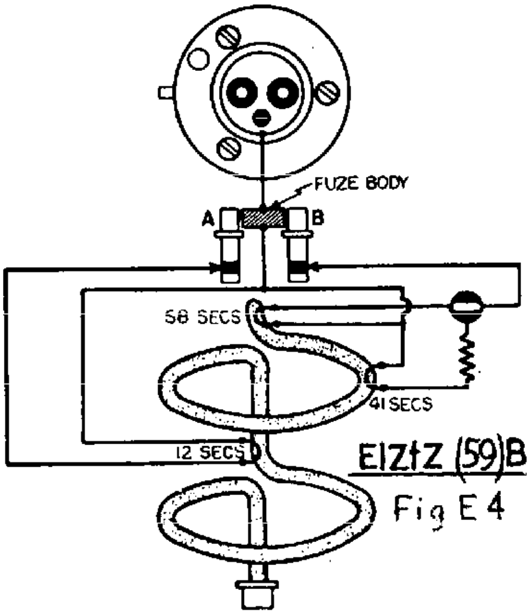


Fig E4

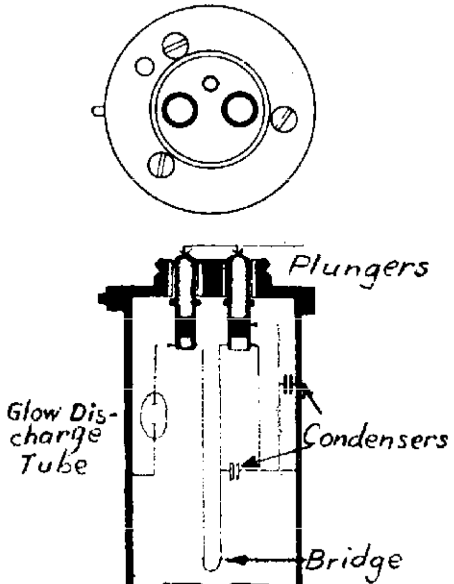


Fig E6

ElZtZ (9)

pyrotechnic delay mixture. On expiration of burning of the delay, the flash compn and the BkPdr pellet were ignited, etc (Ref 1, pp 174–75 & Ref 2, pp Ger 2 & 3)

d) ElZtZ 9 was a short time fuze intended for use in some parachute flares and photoflash bombs. It was cylindrical in shape (See Fig E6) and contained a glow discharge tube, two condensers, a resistance, a bridge and two charging plungers. The 3rd smaller plunger was believed to be used for testing the glow discharge tube. Before dropping the flare, the elec chge from the plane passed thru the plungers into the charging condenser and from there slowly leaked thru a resistance to the firing condenser. At the same time a similar chge was built up on one plate of the neon-filled glow-discharge tube. When the chge which had slowly leaked by and thru the igniter bridge, had built-up on the other plate of the discharge tube to the striking voltage of the gas, the current surged thru the tube. The bridge then ignited the quick-match train, which in turn fired the bursting chge of the flare or of the photostat bomb. The function of the neon tube was similar to that of a condenser (Ref 1, pp 177–78 & Ref 2, pp Ger 2 & 3)

Refs: 1) TM 9–1985–2(1953), pp 172–78
2) PATR **2510**(1958), pp Ger 2–3

Electrical. Pertaining to electricity

Electrical Charge Formation in Handling Anhydric Flammable Liquids. Origin of electrical charges in handling anhydric flammable liquids as well as dangers presented by such charges and means to avoid them are discussed in Ref

Ref: M. Lecaustey, BullMensInform ITERG (Centre Technique Industriel) **7**, 5–10(1953) & CA **47**, 4011(1953)

Electrical Chronographs Used for Velocity Measurements of Projectiles, such as of Le Boulangé, Aberdeen, Camera Counter Chronograph, Machine Gun Chronograph and Rotating Disc Chronograph are described in Vol 3 of Encycl, pp C306-L to C310-R. See also K.J. Cranz, "Lehrbuch der Ballistik", Springer, Berlin, Vol **3**(1927), 52–3 & 56–66

Electrical Chronographs Used for Velocity of Detonation Measurements, such as of Mettegang, Pin Chronograph and Micro-Wave Technique Chronograph are described in Vol 3 of Encycl, pp C313-L to C316

Electrical Clock of Wheatstone, Improved by Hipp (Elektrische Uhr von Whetstone, verbessert von Hipp) for measuring time intervals in solving ballistic problems is described in Cranz, Vol **3**(1927), 129–32

Electrical Conductivity. See Conductivity, Electrical; Conductometry and Conductometric Titrations in Vol 3 of Encycl, pp C497-R & C498-L

Electrical Conductivity in Detonation Products. See Vol 4, p D339-L and Ref 72

Electrical Effects of Shock Waves. Conductivity of CsI, KI and NaCl

Ref: W. Murri et al, Stanford Research Institute (31 Jan 1965). Contract DA-04-200-ORD-1279

Electrical Engineering. The art or science of making practical application of the knowledge of *electricity*, which is defined here under *electric*. It includes: electrical circuits, magnetic circuits, electric and magnetic relations, electrical measurements, photo-piezo-electricity, thermoelectricity, thermionic tubes, conduction of electricity thru gases, gaseous discharge tubes, rectifiers, rheostats, magnets, electrical wiring, illumination, transformers, generators, motors, dynamos, application of electricity for initiation of explosives, propellants and pyrotechnic compositions

Refs: 1) J.H. Perry, Edit, "Chemical Engineers' Handbook", McGraw-Hill, NY(1950), pp 1731–1770: R.G. Warner & F.T. McNamara, "Electricity and Electrical Engineering" 2) O.W. Eshbach, "Handbook of Engineering Fundamentals, J. Wiley, NY (1952), pp 9-01 to 9-77: A.M. Lyon, "Electricity and Magnetism" 3) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **7**(1965) (Electrical Testing)

Electrical Explosions of several materials in a variety of sizes and environments caused by an electrical wire exploder capable of vaporizing small wires in times shorter than 10^{-7} second was studied by the Electro-Optical Systems, Inc, and described in Ref. Materials tested included silver, copper and tungsten wires, as well as single crystals of semiconductors. Environments included air, vacuum and oil. Simultaneous voltage and current oscillograms with nanosecond resolution were obtd, and time-resolved Kerr cell photographs of wires were taken at different times during the expln. Also covered by this Ref are preliminary studies of **exploding films**, attempts to explode needles of conducting materials, and some work on a very fast dielectric switch

Ref: Electro-Optical Systems, Inc (Subsidiary of Xerox Corp, Rochester, NY), Final Report **DA-04-495-ORD-1298**, 16 February 1961 (250pp) (PA Library U78191)

Electrical Firing Properties of Some Lead Compounds of Hydrobenzene Derivatives. The compd examined was Lead Dinitro-o-cresol (LDNOC) (See Vol 3 of Encycl, p C556-R). It was found that accdg to the conditions of pptn, one can obtn two types of compd – one contg 57% Pb was fast burning, the other contg 34% Pb was slow burning. Electric squibs were made with both types of LDNOC, the nichrome wire and geometry of the squibs remaining the same. The 34% Pb squibs gave a transfer time of about 5msecs; even as low a current as 0.8 amps for 4msecs was enough to set off a series of 20 detonators crimped with the squibs. Detonators with 57% Pb LDNOC gave a transfer time of ca 2msecs and the scatter was low. However, a series of 20 such detonators could only be fired when a current of 1.2 amps was passed for 4msecs

Refs: 1) R. Rhujanga Rao et al, R&D of Indian Detonators Ltd, Heyderabad, India (No date is given in Ref 2) 2) Gunther Cohn, Edit, Expls & Pyrots **4**(5), May 1971 (Abstract of Ref 1)

Electrical Firing Properties of Some Lead Compounds of Nitrated Resorcinol. Lead Mononitroresorcinol (LMNR), $PbO_2C_6H_3(NO_2)$ (See p D1271-L in this Vol), used in electric squibs has a lead content betw 68 & 77%. The transfer time of such squibs was found by Rao et al (Ref 1) to be ca 1.5msecs. In order to vary its Pb content, pptn was conducted under different conditions. The resulting compds showed no variations of burning characteristics and hence in transfer time. In this respect LMNR seemed to behave similarly to Lead 4,6-Dinitroresorcinol (4,6-LDNR), $PbO_2C_6H_2(NO_2)_2$ (See p D1275-R in this Vol)

Refs: 1) K. Rhujanga Rao et al, R&D Department of Indian Detonators Ltd, Heyderabad, India (No date is given in Ref 2) 2) Gunther Cohn, Edit, Expls & Pyrots **4**(5), May 1971 (Abstract of Ref 1)

Electrically Conductive Hose "Custom Flex" and Conductive Film "Custom Velostat". Because standard, non-conductive hose is a hazard in many applications with expls, since a static charge can build up to cause a spark discharge, the Customs Materials, Inc, 279 Billerica Rd, Chelmsford, Mass, 01824, developed a special hose which is electrically conductive and, when grounded, will dissipate any static electric charges generated. The hose is designed for use under internal or external pressures. The same Company also makes *Custom Velostat,* a conductive plastic film for draining off static charge by providing a path to ground

Refs: 1) Custom Materials Inc, 279 Billerica Rd, Chelmsford, Mass, 01824 (No date)
2) Gunther Cohn, Edit, Expls & Pyrots **1**(9), Sept 1968 (Abstract)

Electrically Exploding Wire (EEW). See Exploding Bridge Wire (EBW) in Vol 6

Electrically Exploding Wire Detonators. See Exploding Bridge Wire (EBW) Detonators in Vol 4, pp D807 to D810-L

Electrically Produced Nitroexplosives. An apparatus and method were patented by Bloom (Ref) for using AC current to prep various expl compns. For example, in prepn of Cordite, contg NG 65, NC 30 & vaseline 5%, NG & NC were introduced in acetone and subjected to blending in AC current field. Then most of acet was removed and vaseline was incorporated with NC, gelatinized by NG. It was claimed in the patent that elec treatment lessened the danger of exudation of NG from finished product and improved its stability

In prepg expls contg chlorates mixed with aromatic nitrocompds and animal or vegetable oil, the nitro compds were first dissolved in the oil by warming it and then finely pulverized chlorate was mixed in. During mixing an elec current was employed to promote adsorption and increase stability of the final product. During this treatment chlorate particles became coated with a plastic oil mixt, which promoted its stability and decreased its sensitiveness to shock or heat. It was also claimed in the patent that an AC treatment can be used during nitration of cellulose and washing of NC

Ref: J.E. Bloom, USP 1333701(1920) & CA **14**, 1441(1920)

Electrical Measurements in Reaction Zones. Title of the paper by B. Hayes, presented at the 10th Symposium of Combustion (1965)

Electrical Probe Technique for Measurement of Detonation and Deflagration. See Vol 4, p D341-L and Ref 75

Electrical Transducer Studies of Initiation of Liquid Explosives. See Vol 4, p D339-R, Ref 74

Electrical Units. They may be subdivided into EMU (emu)-electromagnetic and ESU (esu) electrostatic units. **EMU** are based on the strength of magnetic poles (m and m′): $M = r\sqrt{F\mu}$, where μ is the magnetic permeability of the medium (assumed to be equal to unity for air); F, the force at a point in magnetic field; and r the distance between the poles. It follows that $F = mm'/\mu r^2$
ESU are based on the strength of electrical charges (q and q′): $q = r\sqrt{Fk}$, where k is the specific inductive capacity or dielectric constant (assumed as unity for air); F, the force between two charges; and r the distance. It follows that $F = qq'/kr^2$ (See also "Electrostatic Law of Coulomb")

Table E4
Relationships of Electrical Units

Symbol	*Unit of:*	*Practical CGS*		*CGS*
		emu	*emu*	*esu*
O	Resistance	1 ohm	10^9	1.1124×10^{-12}
A	Current (Strength)	1 ampere	10^{-1}	2.998×10^9
V	Electromotive Force(Potential)	1 volt	10^8	0.0033349
F	Capacity	1 farad	10^{-9}	8.9892×10^{11}
C	Quantity	1 coulomb	10^{-1}	2.998×10^9
H	Inductance	1 henry	10^9	1.1124×10^{-12}
J	Work	1 joule	10^7	10^7

CGS is an abbreviation for centimeter-gram-second units adopted since 1874
Ref: Hackh's Dict (1944), 297

Electric Blasting Caps; also known as Electric Caps or Electric Commercial Detonators. See Vol 2 of Encycl, pp B186-L to B188 and Vol 4, pp D737 to D742. The caps of DuPont Co are described in Blasters' Hdb (1966), pp 87 to 91 and 136. See also Electric Detonators (Industrial)

Electric Blasting Cap, US Military, known as Electric Cap, Special, Military M6. See Vol 4, pp D806 to D807 with Fig 71

Electric Blasting Caps and RF (Radio Frequency) Energy. A potential hazard in the use of electric blasting caps when in close proximity to RF sources is discussed in the booklet (Ref 1) and reviewed in Ref 2. The booklet is intended to give the commercial blaster information about operating with electric caps under what could be considered unsafe conditions. It gives basic info on the mechanism of RF initiation and its avoidance. The booklet warns that it is applied only to commercial electric blasting caps manufd in the US, andnot to military firing devices

Accdg to reviewer (Ref 2), probably the most important section of the booklet is its "Table of Distances", giving the minimum dist that should be kept betw the sourceof RF energy and blasting cap circuits

Refs: 1) Publication No 20, Safety Library, "Radio Frequency Energy, A Potential Hazard in the Use of Electric Blasting Caps", Institute of Makers of Explosives, 420 Lexington Ave, New York, NY, 10017 (Publd in March 1968) 2) Gunther Cohn, Edit, Expls & Pyrots 1(7), July 1968(Review of Ref 1)

Electric Bomb Fuzes of WWII (German). The development of electric time and impact fuzes had been carried on in Germany since 1926 and the greater part of the work was done by the Rheinmetall-Borsig Co under the direction of H. Rhulemann. The original object was to produce a fuze which could be set at the instant of firing. However, before this work was completed a successful fuze was developed in 1937 and adopted by Luftwaffe. This was followed by several other impact or time types of elec bomb fuzes. All of these fuzes were cylindrical in shape and, with the exception of Type 5, used Al for the case

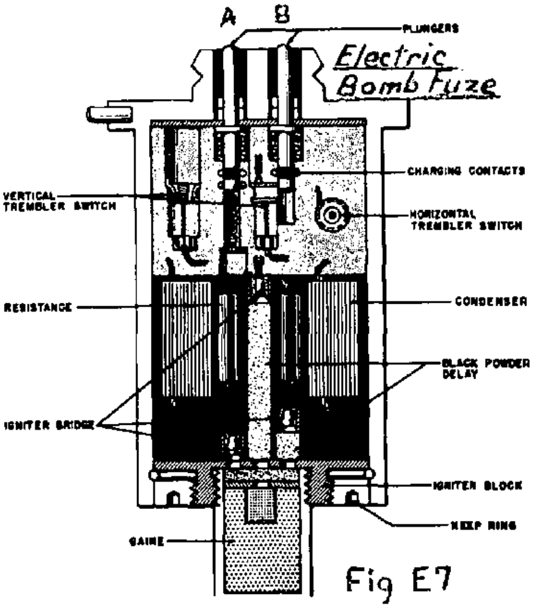


Fig E7

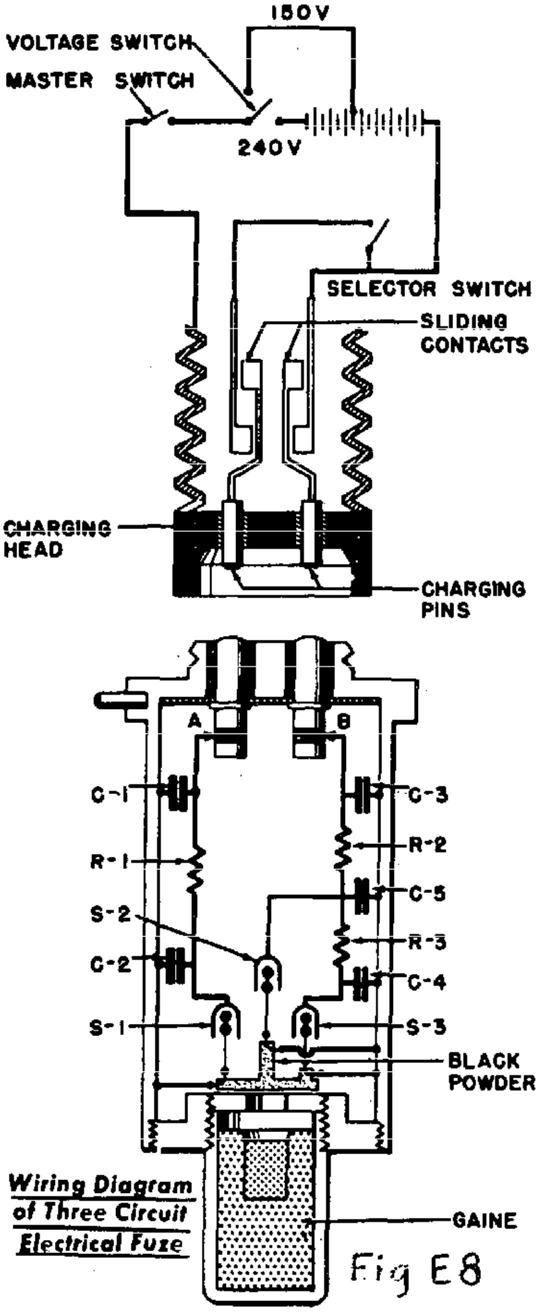
The inner part of typical fuze consisted of two sections, as shown in Fig E7

a) The upper section, called the switch block, was molded polystyrene which has been machined to take various plunger contacts, the trembler switches, and in some cases long delay igniter bridge

b) The lower section contained the storage and firing condensers, the resistances and instantaneous and short delay igniters. All these items were held in place by a black bitumen caulking substance. Condensers were constructed of metal foil strips separated by wax paper, all wound on one cardboard cylinder. The carbon resistances were located inside this cylinder. The igniter block fitted into the bottom of the fuze and contd the BkPdr flash pellet, the cover with three perforations leading from the pellet to the igniter bridge, and the short delay train

Following is a brief description of operation of a three circuit elec impact fuze illustrated in Fig E8

As the bomb was placed in the plane, a



Wiring Diagram of Three Circuit Electrical Fuze

Fig E8

charging head was clamped on the fuze head. The charging pins contacted the plungers and depressed them so that they could make electrical contact with the storage condensers. The two charging pins were connected to the sliding contacts located in the charging arm. These contacts closed when the bomb had fallen from 1 to 3 inches from the rack. This prevented charging of the fuze while the bomb was still in the aircraft. The two sliding contacts were connected to the positive terminal of the 240 volt battery. The B plunger circuit was connected directly while the A plunger circuit was connected thru a selector switch which had two positions: open (MV) with delay, and closed (OV) without delay. The battery was tapped at 240 and 150 volts and the two leads were run to the voltage switch. This switch was set at 150V for level bombing and at 240V for dive bombing, but it could not be used to open the circuit. The voltage switch was connected to the master switch which was used to jettison the bombs. The master switch was connected to the charging head which contacted the fuze head and completed the electrical circuit thru the fuze body to the storage condensers

Prior to the release of the bomb, the master switch was closed completing the circuit from the batteries thru to the fuze except for the sliding contacts in the charging head. When the bomb was dropped, the charging arm was extended, causing the sliding contacts to meet for about 1/3000 of a second, the ground return circuit being thru the fuze body. If the selector switch was closed, both plungers received the current and the storage condensers, C-1 and C-3, were charged. The charge of C-1 leaked slowly thru the resistance R-1 into the firing condenser C-2 (The time required for the current to pass from C-1 to C-2 and build up sufficiently to fire the igniter is called the arming time). At the same time the charge of C-3 leaked thru R-2 into the firing condenser C-5 and also part of the current leaked thru R-3 into the firing condenser C-4. On impact, the tremblers of switches S-1, S-2 and S-3, made contacts with their cups, causing the current to flow thru the igniter bridges. These were thereby heated and fired the match compositions surrounding them. When all three igniter bridges fired simultaneously the instantaneous bridge fired the flash pellet and detonated the bomb thru the normal explosive train. The short and long delay trains started to burn just at the instant of detonation

If the selector switch was held open, then the charge went thru plunger B to the storage condenser C-3 and nothing passed to the instantaneous circuit. The circuit thru the resistance R-2 to the condenser C-5 became armed before the circuit thru both resistances R-2 and R-3 to firing condenser C-4. If the bomb had been dropped from an altitude of less than 1170 ft, the latter circuit would not be armed before impact and the igniter bridge associated with the trembler switch S-2 would fire the long delay pellet which acting thru the explosive train of the fuze would detonate the bomb. If the bomb was dropped from an altitude greater than 1170 ft, both circuits would be armed before impact, but because of the shorter delay train used in conjunction with the trembler switch S-3, the short delay would initiate the final explosive train

Electrical time fuzes (ElZtZ) contained essentially the same basic parts as the electrical impact fuzes (ElAZ), except that the trembler switches were replaced by a vacuum tube which became conducting at a critical predetermined voltage. At the instant the bomb was started on its trajectory, an electric charge was put on the storage condenser, and another smaller charge was put on the firing condenser. The time setting of the fuze was adjusted by varying the amount of charge placed on the firing condenser. During flight, part of the charge on the storage condenser leaked thru the resistor to the firing condenser. As the charge on the firing condenser increased, the voltage across the vacuum tube also increased. When the firing voltage of the tube had been reached, the firing condenser discharged thru the tube and the igniter bridge thus firing the fuze

Electrical bomb fuzes are described in Refs 1 and 3 and are listed in this work under Fuze. Some of these fuzes are described in this work under Aerial Burst Fuzes

An electrical time fuze (EIZtZ S/30) for use in projectiles is briefly described in Ref 4, pp 605–8. Prior to firing the projectile the fuze is charged either by hand or by a machine by putting 300 to 500 volts across the shell and an insulated contact which put voltage on the annular storage condenser. The charging could also be done by allowing the "feeler wire" (connected to the electrical circuit of the fuze) to contact the "muzzle charging ring" as the projectile was leaving the gun. A brief description of a muzzle charging ring is given in Ref 4, pp 606

A device, described in Refs 2, p 422 and 4 p 623 as the electric fuze, ERZ 39, was used for igniting the black powder charge which set off the propellant of 15 cm and 21 cm rockets. This device is briefly described in this work under Rocket Propellant Igniter

Refs: 1) Anon, War Dept Tech Manual **TM E9–1983** (1942), Enemy Bombs and Fuzes, File Numbers 2321.5, 2321.8, 2324.92 & 2324.93 (2) Anon, Ordnance Bomb Disposal Center, Aberdeen Proving Ground, Md (No date): German Artillery Projectiles and Fuzes p 422 (3) Anon, Dept of the Army Tech Manual **TM 9–1985–2** (1953), German Bombs, Fuzes&Rockets, etc, pp 125–132 and others 4) Anon, Dept of the Army Tech Manual **TM9–1985–3** (1953), German Projectiles and Fuzes, pp 605–7 and 623 5) Fedoroff et al PATR **2510,** pp Ger 40 & Ger 41

Electrically Initiated Explosive Devices, Bridgewire Welding for. See Hugh H. Snider et al, NOLTR **65–37** (26 Feb 1965), Naval Ordnance Laboratory

Electric Bomb and Pyrotechnic Fuzes (Ger). In addn to electric impact and time fuzes, there were also developed "Electric Clockwork Time Fuze (17A)" and "Electric Impact Tail Fuze". They and some other elec fuzes are described in Ref 1 and listed in Ref 2, but not described. The cuts of the above two fuzes are, however, reproduced on p Ger 55

Refs: 1) **TM 9–1985–2** (1953), pp 139–49, 152–57, 172–73 & 181–84 2) PATR **2510** (1958), pp Ger 54 to Ger 56

Electric Delay Detonators (Military) (US). Two detonators for use in fuzes are described in Vol 4 of Encycl:

a) Electric Delay Detonator T65 (p D849, Fig 1–32) and

b) Electric Delay Detonator Mk35Mod 1(Navy) (p D865, with Fig 1–59 on p D866)

In the pamphlet of S. Odierno, "Information Pertaining to Fuzes", Vol IV, published in 1964 by Picatinny Arsenal are listed in Table of p V A (Our Table E5) three Electric Delay Detonators. The T65 is also described in Vol 4 of Encycl

Table E5

	Physical Characteristics				Electrical Characteristics				Explosive Train Milligrams(Mean)			
Nomenclature	Lead	Dia. Max in	Body Length Max in	Bridge	Capacitor Discharge Microfarad	Volts	Ergs	Firing Time Max Second	Spot	Priming DLA	Base	Other
XM60	B2	.194	.535	C	.02	100	1000	.0005	5 LMNR	70	75 PETN	21 LMNR upper charge
T65	B1	.194	.685	C	.004	100	200	.05	MLSt	227	55R	I & D
T68	B1	.194	.600	C	004	100	200	.05	MLSt	235	53R	I & D

Symbols: Lead – B – Button with length B1 0.065 and B2 0.167 inch
Bridge – C – Carbon (1K to 10K ohms)
Expl Train – LMNR – Leadmononitroresorcinate
MLSt – Milled Normal Lead Styphnate mixed with NC lacquer
DLA – Dextrinated Lead Azide
I – Igniter 30/70-Boron/Lead Peroxide
D – Delay 10/90-Boron/Barium Chromate
R – RDX

Electric Delay Primers (Military) (US). Two of such electric delay primers: Gasless Electric Delay Fuze Primer, Mk115 and Experimental Delay Fuze Primer are described in Vol 4, pp D868 & D869, Figs 1–62 & 1–63

Electric Detonation Wave. See Vol 4, p D684-L

Electric Detonators (Industrial). Electric Detonator is the British name for them, but in the USA they are known as "Electric Blasting Caps" or "Electric Blasting Initiators" described in Vol 2, pp B186-R to B188. Figs of three types are given on p B188. DuPont's Caps are described in Blasters' Hdb (1966), pp 87 to 91 and 136

A "Special Military Electric Cap, M6" is described in Vol 4, pp D806-R & D807 with Fig 71

British Electric Detonators, which are actually Electric Blasting Caps are described in Vol 4, pp D738 to D741 with Figs 7, 8, 9, 10, 11 & 12

Electric Detonators, Low-Firing Energy (Industrial). Five low-firing energy electric detonators were developed before 1970 by the DuPont Co. They are minute in size (from 0.134 in diam by 0.350 in length), low in firing energy and fast in response. These features permit conservation of space and

electrical energy
Refs: 1) E.I. DuPont de Nemours & Co, Explosives Dept, Wilmington, Del, 19898 2) Gunther Cohn, Edit, Expls & Pyrots 3 (2), Feb 1970

Electric Detonators (Military) (US). The following electric detonators employed in US fuzes are described in Vol 4 of Encycl, Section 4, Part D:
a) Instantaneous Navy Electric Detonator Mk46Mod0 (p D846, Fig 1–27)
b) Instantaneous Navy Electric Detonator Mk51Mod0 (p D846, Fig 1–28 on p D847)
c) Instantaneous Army Electric Detonator M36 (p D847, Fig 1–29)
d) Instantaneous Army Electric Detonator M36A1 (pD848-L, no Fig)
e) Instantaneous Army Electric Detonator M48 (p D848, Fig 1–30)
e₁) Instantaneous Army Electric Detonator M51 (p D848, Fig 1–31)
f) Instantaneous Army Electric Detonator T20E1 (p D848, no Fig)
g) Instantaneous Button-Type Electric Detonator T44 (p D849-L, no Fig)
h) Instantaneous Stab-Electric Detonator T29 (p D849-R, no Fig)
i) Instantaneous Electric Detonator T39 (p D849-R, no Fig)
j) Instantaneous Button-Type Electric Detonator T62 (p D849-R & Fig 1–33 on p D850)
Note: Accdg to Mr Paul Tweed, formerly of PicArsn, Ordnance 44, 653(1959–60),M36 was the first Army Ordnance Corps Electric Detonator used in VT Fuzes (See also Electric Delay Detonators Used in US Fuzes)

In the pamphlet of S. Odierno, "Information Pertaining to Fuzes", Vol IV, published by Picatinny Arsenal in 1964 are listed in Tables on pp IV A to IV E forty-four US Electric Fuze Detonators, whereas on p IV F are listed materials used in their manufacture

The electric detonators consist of the following parts:
Bodies – Al or Al base alloys if LA is used as an ingredient of a charge and Cu or Cu alloys (such as gilding metal) for chges contg MF
Leads – either wires (W) or buttons (B) of various lengths; in one of the listed detonators (XM72), two pins are used for a lead
Bridges – either carbon (C) or wire (W); in one of the listed detonators the bridge consists of EBW, while in two detonators SG (spark gaps) are used
Note: Description of EBW Detonators is given in Vol 4, pp D807 to D810
Explosive Trains consist of one or several of the following materials: CLA (Colloidal Lead Azide), DLA (Dextrinated Lead Azide), LMNR (Leadmononitroresorcinate), LSt (Lead Styphnate with gum binder), MLA (Milled Lead Azide), MLS (Milled Normal Lead Styphnate mixed with NC lacquer), PETN and RDX
Addnl Refs: A) O.A.J. Gurton & I.O. Lewis, BritP 787346(1957) & CA **52**, 6796(1958) (Ventless delay electric detonators)
B) NipponChemDrugCo, JapanP 12093(1960) & CA **54**, 25829(1960) (A semiconducting SiC or powdered mixture of Si & Zr is placed betw two electrodes as a thermistor, above which is placed a priming powder, for ignition at low voltage) C) S.G. Nesbitt, NOLTR **71–70**, 21 July 1971 (Functioning characteristics of Electric Detonator, Mk101 Type designed to meet the requirements of Specification MIL-I-23659 were investigated at NOL. The detonator is a 1-Amp/1-Watt Type)
D) G. Cohn, "Explosives and Pyrotechnics", Philadelphia, Vol 5, No 2, p 3 (Feb 1971) (Résumé of the above NOLTR)

Electric Detonators, Military (Except for Fuzes). See Vol 4, pp D803 to D807

Electric Detonators, Military (For Fuzes). See Vol 4, pp D846 to D850 and also: H. Freiwald, "Moderne elektrische Detonatoren", Wohrtechnische Monatshefter", **1966**(11), 488–97, with 20 refs

Electric Detonators, Primers and Blasting Caps. Analytical Procedures. See StdMethodsChemAnalysis **2B**(1963), pp 1365–70 which contain:
p 1365 – Preparation of Sample

p 1366 – Analysis of Compositions Containing Mercury Fulminate and Potassium Chlorate
p 1366 – Analysis of Compositions Containing Nitrocompounds
p 1367 – **Primers**, Variations of Compositions, which on
p 1368 – includes Preparation of Sample, Qualitative Examination and Quantitative Analysis
p 1369 – Example 1: Detonator Primer Composition Containing Potassium Chlorate, Antimony Sulfide, Lead Azide and Carborundum
p 1369 – Example 2: Primer Composition Containing Barium Nitrate, Normal or Basic Lead Styphnate, Tetracene, Antimony Sulfide, and Lead Azide

Electric Detonators Used in Italian Mining Industry. Protection Against Static Electricity. See ItalP 538568(1956) by Montecatini Co, listed in CA **52**, 4988(1958)

Electric Dust Fuze (Ger). See Dust Fuze in Vol 5

Electric Equipment in Explosion-Exposed Installations. This equipment includes electric motors, switches, wiring, etc. They must be of fireproof type. The discussion on this subject is in the following refs:
Refs: 1) W. Veitl, Explosivstoffe **1956**, 102–04 (Protection of electrical wiring and switches in explosion-exposed buildings) 2) E. Diancourt, Ibid **1957**, 209–13 (Electric motors in explosion-exposed rooms) 3) H. Busch, Ibid **1958**, 54–9(6 refs) (Types of protection in explosion-exposed installations) 4) E.F. Richter & W. Knittel, Kunststoffe **50**, 267–76 (1960) (Creeping current as possible cause of explns)

Electric Exploders or Blasting Machines are discussed in Vol 2 of Encycl, p B212 and in DuPont's Blasters' Hdb (1966), under "Blasting Machines", pp 113–20. A "push-down blasting machine" is described on p 154

Electric Firing Techniques are discussed in DuPont's Blasters' Hdb (1966), pp 135 to 168

Electric Fuses are devices designed to ignite suitably constructed fuses by electric means, and thus to transmit the flame produced by the fuse to some ignitable substances. These substances may be either a blasting charge of Black Powder, an ignition (flash) mixture similar to those used in regular blasting caps or a pyrotechnic composition, etc

The original electric fuse was invented in 1745 by Dr Watson of England with the intention of firing gunpowder charges directly. This electric fuse did not work very satisfactorily and in 1749 Benjamin Franklin improved it by introducing a pellet of BlkPdr, which had to be ignited by an electric spark They did not find commercial application until B. Shaw introduced similar devices in 1830. Originally, the ignition of these fuses was accomplished either by an "influence machine" or by a Leyden jar, but they were replaced in 1831 by a galvanic battery by R. Hare of Pennsylvania, which was a considerable improvement

The original fuses were of high tension type, producing a spark at the gap between two wires, but now they have been replaced by a low tension type (See also Electric Blasting Caps under Blasting Caps; compare Electric Igniters)
Refs: 1) Marshall, **2**(1917), 543 and **3**(1932), 6 2) C.E. Monroe, Explosives Engineer **1925** (April & June) 3) Van Gelder & Schlatter (1927), 736 4) Z. Akashi, JapP 3045(1951) & CA **47**, 866(1953) (Delayed action elec fuse) 5) W. Rauch & O. Ernst, GerP 1121710 (1962) & CA **57**, 9352(1962) (Sealed elec fuse fabricated from Cu 80, Ag 15 & P 5%)

Electric (or Electronic) Fuze is one which depends for its arming and functioning upon events of an electronic nature. Such a fuze may be provided by any of the electric detonators described here under Electric Detonators Used in US Fuzes. For example, Point Initiating Base Detonating Fuze, PIBDF M530A, described in Vol 4, p D916 with Fig 1–96 is provided with Electric Detonator M69. Parts for this detonator are described in Specification MIL-D-60027 (15 April 1964) and its loading assembly & packing requirements are found in Specification MIL-D-60031 (15 April 1964)

An electric fuze contg mechanical components is known as "Electromechanical Fuze" (qv)

Electric Fuze Detonators. See Vol 4, pp D846 to D850 and Figs 1–27, 1–28, 1–29, 1–30, 1–31, 1–32 and 1–33

Electric Fuze for German Projectile. In the article published under the title "Probleme um die elektrische Zündung" in SS **36**, 135–37 (1941) by G. Grötsch is described a type of electric fuze developed in Germany before WWII. The novel feature of the fuze was that it did not contain inside any electrically charged item (like a battery), but only a condenser (capacitor) which did not become charged (by induction) until the projectile started to leave the muzzle of the gun. This feature of the fuze made it safe to handle and to store

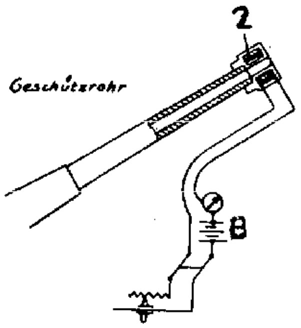


Fig E9

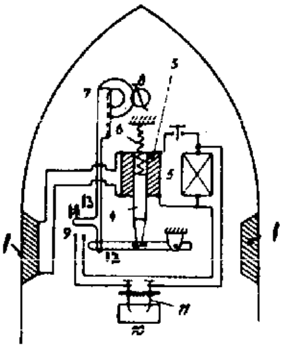


Fig E10

The gun intended for propelling projectiles equipped with above fuze is presented in Fig E9. Around the muzzle of the barrel is placed the "excitation coil" (Erregerspule)(2) connected with an outside source of current such as battery (B). The fuze, which is located in the nose of a projectile is presented here in Fig E10. It is provided with an induction coil (1), located behind bourrelet (Führungswulst), the purpose of which is to allow the current from battery (B) to pass thru excitation coil (2) of Fig E9 to the fuze at the time of passage thru the muzzle of the gun (Rohrmündung). From coil (1) the current goes to magnetic coil (Magnetspule) (3) and then to condenser (Kondensator) (5) where the charge is stored. In the center of coil (3) is located movable iron core (Eisenkern) (4), suspended against the compression spring (Federkraft) (6). At the time of current passing thru coil (3), the core (4) moves upwards against the compression of spring (6). At the same time the lever (12), connected to lower part of core (4) pushes the rod (13) upwards, thus setting in motion the geared wheel mechanism (Hebelrädermechanismus) (7) and the rotating air-resisting vane (Luftwiderstandsflügel) (8). As soon as the projectile leaves the muzzle of the gun, the supply of current thru excitation coil (2) of Fig E9 stops entering the fuze thru induction coil (1) of Fig E10 and this will cause the disconnection of condenser (5) from magnetic coil (3). As there will be no current in coil (3), the core (4), not being able to resist the pressure of spring (6), will move downwards pushing the lever (12) and rod (13) in the same direction. This will cause the mechanism (7) to stop the rotation of vane (8). At the same time the downward movement of lever (12), will cause the cross-rod (14) to close the contact switch (Kontaktschluss) (9), thus permitting the electrostatic charge stored in condenser (5) to pass to electric detonator (10) thru the poles (11). Initiation of charge is produced by spark jumping across the poles (spark-gap method)

A mathematical treatment for the above fuze given by Grötsch on pp 136 & 137 is not included by us

Two similar fuzes shown in Abb 3 and Abb 4 of p 137 are not included here because they do not seem to be any better than the above described fuze and, besides, their description is too sketchy

We have no information if the fuzes charged by induction on passing thru the muzzle were ever used by the German Armed Forces (Wehrmacht)

Note: Two Ger electric fuzes of WWII were described in **TM 9–1985–3** (1953), pp 605 & 623. They were ElZtZ S30 (Electric Time Fuze S30) and ERZ 39 (Electric Rimvent Fuze 39). They are listed in PATR **2510** (1958), pp Ger 59-R and Ger 63-L

Electric Fuze Detonators, Delay. See under Electric Delay Detonators (Military) (US)

Electric Fuze Detonators, Instantaneous. See under Electric Detonators (Military) (US)

Electric Fuze Primers. See Vol 4, pp D854 to D856 and Figs 1–43 and 1–45

Electric Igniters and Primers (Industrial). See Electric Squibs in this Vol and also under "Nonmilitary Igniters" in Vol 4, pp D733 & D734-L and "Nonmilitary Primers", pp D734-L to D737-L

Electric Igniters, Military (Ger), such as Elektrische Zünder **ESMiZ 40**, is described in Ref 1, pp 281–82 and in Ref 2, p Ger 96-R, with Fig on p Ger 97. It was used in the Landmine known as Eismine 42, or Flaschenmine, described in Ref 2, p Ger 107-R, with Fig on p D106

Refs: 1) **TM 9–1985–2** (1953), pp 281–82
2) PATR **2510** (1958), pp Ger 96–97 & Ger 106–107

Electric Igniter M59 for Use in Pyrotechnics. See Vol 4, p D761-R

Electric Igniter-Primer M74. US Military Specification Requirements and analytical procedures are given in Spec MIL-I-46521B (MU)(1966) and also in Vol 4, pp D1066 & D1067-L

Electric Indicators are simulated electric detonators. Two wire bridge types, T-20E1 and T-24E1, are constructed as follows: A .040" thick cellulose acetate window is pressed in the bottom of a stainless steel cup, followed by a wafer of 0.002" tissue paper impregnated with a flash mixture consisting of $KClO_3$ 46.0, Paris Green 20.3, powd shellac 5.8 & dextrin 27.9%. This presented a green spot for viewing. The wafer is held in place by a polyethylene sleeve 0.130" long. A 0.003" thick acetate diaphragm is then pressed into the cup to act as one end of a confining chamber for 4mg of the above flash mixture surrounding the wire bridge. A hollow polyethylene column .062" long forms the outer container of the flash mix. The firing plug is crimped in place in the usual manner. The firing of the simulated detonator is shown by change in green color of the viewing window

Ref: Barkley & Dexter Laboratories, Boston, Mass, Repts 1–21 on Cont DA1–19–020–501 Ord(P)37, Project TA1–3707(1953–54)

Electric Initiating Devices of DuPont Company. Under this title are listed in DuPont's Blaters' Hdb (1966), pp 87–95, the following items:

a) "Acudet" Mark V Delay, p 93. They are used principally in underground wiring and are of long interval delay type (Compare with items d, e and i)

b) Aluminum Wire Instantaneous for use in aluminum EB Caps, p 90. They are intended for mining the products which must be kept free of both Cu & Fe, such as talc and ceramic clay

c) Coal Mine Delays, p 94, are a special series of MS (millisecond) Delay EB Caps designed for use in coal mines. They are available only with bronze shells and iron leg wires

d) Copper Wire Instantaneous EB Caps of No 6 strength, p 89. They have 1-1/8 inch long Al shells and Cu leg wires. Caps with Cu alloy (bronze) shells are also available, but the Al shells are usually preferred because of their superior corrosive resistance

Note: The US Bureau of Mines prohibits the use of Al shell BC's for underground coal mines in which permissible expls are required

e) Delay Electric Blasting Caps, pp 91–2. They are intended to detonate at a predetermined period of time after energy is applied to the ignition system and to fire a number of expl charges in accurate rotation. The DuPont Co manufs three different series of Delay EB caps: DuPont "Acudet" Mark V Delays, DuPont MS Delays and DuPont Coal Mine Delays

f) Electric Squibs. See below

g) Iron Wire Instantaneous EB Caps of No 6 strength, p 90. They have bronze shells and iron leg wires and are permitted to be used in underground coal mines

h) Jet Tapper Caps, p 91, are DuPont special high temperature instantaneous EB Caps for use with DuPont Jet Tappers, which are subject to very high temperatures in tapping open hearth furnaces. These caps have Cu-alloy shells and 50-foot iron leg wire with white duplex insulation. When assembled with Jet Tapper, the cap will withstand temps of 1500–2000°F for ca 3 minutes before detonating voluntarily

i) MS Delays, p 93. These DuPont Millisecond (MS) Delay EB Caps have much shorter delay intervals than the "Acudet" Mark V Delays. The MS Delay EB Caps are normally supplied with Al shells having lengths varying from 1.5 to 3.0 inches, depending upon the delay period. Leg wires may be either Cu, Fe or Al as in "Acudet" Mark V Delays. The MS Delay Caps are used principally to detonate charges in small diameter drill holes. The advantages of MS delay firing over long interval delay firing are listed on p 94 of Blasters' Hdb (1966)

j) "SSS" Seismograph Electric Blasting Cap was developed by the DuPont Co to meet the critical needs of seismic prospecting (pp 90–91). It is a precision instantaneous EB Cap which is supplied only in the No 8 strength. When intended for use where alkaline drilling muds are present, the cap is supplied with a bronze shell to prevent corrosion which would occur if Al or other metal shells were employed. For other seismograph uses, i.e. water work, other metals or alloys may be used. Standard DuPont "SSS" EB Caps have plastic insulated 20-gage Cu leg wires up to 400 feet long. Because seismograph caps are very fast in firing, a special current source is required for series firing (pattern work) to prevent failures caused by the current being cut off too soon. A Condenser Discharge Blasting Machine [Such as described in Blasters' Hdb (1966), p 114] should be used for this purpose

Electric Initiating Device of Zebree has a delay fuse element and a loose ignition mixture (Pb 72.4 & Se 27.6%) in contact. Incorporation of 1–5% Si which increases the heat of combstn of this mixture, compensates for the deteriorations which occur in ignition mixtures

Ref: D.T. Zebree, USP 2908559(1959) & CA **54**, 2745(1960)

Electric Initiators, Activated, can be divided in 5 main classes basedon the type of transducer used for conversion of the electrical stimulus: (a) resistive bridgewire, exemplified by electric blasting caps, squibs, M-36A1 detonator (See also under detonators) (b) carbon bridge (See under detonators) (c) conductive mix initiators used in gun primers such as the M52A3, A4 and A5 series (d) spark gap detonators characterized by extreme high bridge impedance such as the T-39. See also Electro-Explosive Transducer

Ref: M.T. Hedges, PATM Rept 4 ORDBB–DR4–36(1960) (Exploding Wire Initiators)

Electric Initiators of Atlas Chemical Inds,

Inc, Aerospace Comp Div, Valley Forge, Pa, 19481, included in 1971-227 series of *dimple actuators* and M-100 & M-200 series of *electric matches*
Ref: Expls & Pyrots 4(3)(1971)

Electric Instruments. Many and various kinds of electric instruments are used in industrial processes and in analytical and research laboratories, including those dealing with expls and ammunition. Among such instruments may be mentioned: ammeters, voltmeters, galvanometers, thermocouples, ohmmeters, wattmeters, frequency meters, oscillographs, computers, etc
Refs: 1) C.A. Culver, "Theory and Application of Electricity and Magnetism", McGraw-Hill, NY (1947) 2) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol 5(1950), pp 478–87: K.G. Courlee, "Electric Instruments" (18 references) 3) Ditto, 2nd Edition, Vol 7 (1965), pp 726–84, D.J. Gross & R.W. Murray, "Electroanalytical Methods"

Electricity in Blasting; History. Although the first electrical machine was invented as early as the 17th century (by Otto von Guericke), nobody had an idea at that time to utilize the electric *spark* produced by that machine for igniting combustible or explosive substances. It was at the inauguration of the Royal Academy of Science in Berlin by Friederich the Great in 1744 that Dr Ludolf ignited the ether by means of an electric spark produced by his electrostatic machine
Note: This experiment was described in 1767 by Priestley in his "History of Electricity", p 73, but it does not mean that Priestley was the originator of ignition by means of an electric spark

In the year 1745, Dr Watson of London tried to ignite Black Powder (BkPdr) by means of an electric spark but did not succeed because the explosive was in loose powder form and most of it scattered before ignition took place. Benjamin Franklin found in 1751 a method to overcome the difficulties encountered by Watson and he succeeded in priming the BkPdr by an electric spark. For this Franklin loaded the grains of BkPdr into cartridges by compression and thus partly crushed the grains

Due to the fact that electrostatic machines existing in the 18th century were not convenient to be used in field work, the electrical method of blasting did not find any practical application at that time. The introduction of the *battery* by Wollaston (which was the first practical improvement of Volta's pile invented in 1800 and modified in 1801 by Dr Cruickshank) may be considered as the real beginning of application of electricity in blasting. Another improvement in battery blasting was introduced in 1812 by Baron Schelling. His method called "galvanism" used to ignite by the current of the battery a charge of charcoal, which, in turn, ignited the BkPdr

Still another important improvement in batteries was developed in 1822 by Prof R. Hare of Pennsylvania. His battery was introduced in mining in the USA where it was used extensively for many years. By 1835 to 1840, the electrical method of priming BkPdr became well established in the USA and also in England. The first large scale blasting by electrical battery method was conducted in 1843 in Dover, England. In this work, 9¼ tons of BkPdr, loaded in three separate charges, were fired simultaneously by means of a very large Wollaston battery. As a result of this expln 30580 cubic meters of rock were crushed

In France the battery method of blasting was first used in 1838 by military engineers, while the first industrial application was in 1851. It was done for increasing the depth of a well in Veyras (Ardèche). For this, a chge of BkPdr had inserted in it a piece of very fine iron wire (serving as resistance), two ends of which were attached to long isolated copper wires serving as "leads" The ensemble was lowered to nearly the bottom of the well and the upper parts of wires connected to the battery located outside the well. When the current was passed, the resistance wire inside the BkPdr chge became redhot and this caused the expln of BkPdr

Besides batteries, other sources of electricity started to be introduced beginning in

1850's. Among these may be cited *friction machines* (exploders) of Ramsden, Baron von Ebner, Bornhardt, Abbey, Mahler, Nobel, Elsner, Ladd and Mowbray. Although these machines were heavy and inconvenient in handling and transportation, they were used to a great extent

A different type of apparatus was the *induction machine* of Count Ruhmkorff, introduced in 1851. The tests conducted in 1853 at la Villette, near Paris proved to be so successful that the machine was adopted in France for mining. It was successfully used in 1854 by Count de Moncel for blasting in Port of Cherbourg. The machine was also patented in England, in 1855, 1862 & 1864. The induction exploders are not used any more being replaced by other systems, such as magneto type, etc

Although the magneto was invented by Faraday as early as 1831, its principle was not applied to blasting until 1856, when Sir Ch. Whetstone introduced his magneto exploder. Several other machines, such as of Ducretet of Paris, Hunter & Warrent of Glasgow and Ingersoll Rock Drill Co of NY were invented about the 1950's. All the above machines produced AC (alternating current), but there were also machines producing DC (direct current). The earliest DC machine was that of Bréguet (improved by Ducretet) and then of Hickley and Scola

An interesting exploder was invented nearly at the end of the 19th century by Schmitt & Co of Germany. This device, known as *Gnom*, was so small that it could be carried in a pocket. It was described in detail by Daniel (Ref 1, p 243, Figs 25 & 26)

The description given up to this place is a slightly abbreviated translation of Daniel's book (Ref 1, p 239–43)

The following description covers briefly pp 244–52 of the same book

It might be noted that ordinary batteries (such as of Leclanché) as well as dry cells and storage batteries continued to be used in exploders (blasting machines), especially for military purposes. They were, however, partly replaced by magnetos or dynamos, such as in Push-Down and Twist Exploders

Some electric blasting caps, called by Daniel "amorces électriques", used in the 19th century, were described on pp 244–50 of Ref 1. Among them the US caps of Laflin and Rand Powder Co are represented in Fig 27, p 245, together with their detailed description. Some other systems are mentioned on pp 246–48, while on p 249 was described with Fig 28 the Spaltglühzünder manufd by the Fabrik elektrischer Zünder at Köln, Germany. This cap was patented in Belgium as No 149662 (1900)

Ducretet of Paris patented in 1900 an improved exploder which was briefly discussed on p 250 of Ref 1

A brief description of method of conducting electrical blasting operations is given on pp 250–52 of Ref 1

The description of current methods of application of electricity for blasting operation is given in DuPont's Blasters' Hdb (Ref 2)

Refs: 1) J. Daniel, "Dictionnaire des Matières Explosives", Dunod, Paris (1902), pp 239–52: "Electricité (Application de l') au tirage de mines" 2) "Blasters' Handbook", E.I. DuPont de Nemours & Co (Inc), Wilmington, Delaware, 19898, Fifteenth Edition (1966), pp 113–26 "Blasting Machines" and pp 135–68 "Electric Firing Techniques"

ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT.

Introduction. The term *extraneous electricity* means the unwanted electrical energy that may enter electric blasting circuits from any outside source. The sources of this electricity can be either those generated by nature, or those generated by man. Those generated by nature include: lightning, static and galvanic action. Those generated by man include: radio frequency currents, static generators, stray ground currents from improperly installed or malfunctioning electrical equipment, induced magnetic and electrostatic currents, corona discharges from high-voltage transmission lines, and strong ground currents from power lines or rails in the vicinity of the blasting site or of the plants manufacturing, storing, or handling explosives and propellants. Static electricity can also be accumulated on individuals, such as by friction or successive contact and separation of unlike surfaces, at least one of which is usually an insulator. A charged conductor such as an individual or a piece of equipment acts as an electrical condenser, and when any part of it comes in sufficiently close proximity to another conductor a spark discharge can occur. Then materials in the path of the discharge may be ignited (or even exploded) if they are sufficiently sensitive and the energy in the discharge is sufficiently high (Ref 27, p 169 & Ref 4, pp 1–2) (See also item VIII. Electrostatic Discharges and Sensitivities of Explosives to Initiation by)

Types of Extraneous Electricity:

I. Lightning. If it strikes an explosive or a blasting circuit, detonation is very probable, regardless of any precautions known at present time. Even a near miss will probably cause a deton. Electric charges, large enough to detonate electric caps, have been induced in blasting circuits by strokes several miles away. The danger from lightning is considerably increased if there is a transmission line, fence or stream to conduct electricity betw the storm and the blasting point. Since there is positively no way to render an electric blasting cap or electric circuit insensitive to the hazardous influence of lightning, all blasting operations on land, underground or on water should be suspended and all personnel removed from the blasting area when an electric storm approaches. A standard warning system should be devised and used to alert the personnel (Ref 27, p 169 & Refs 30, 30a & 30b)

II. Static Electricity Charge. It can be built in atmosphere (even when considerably removed from lightning) and then stored on any conductive body, such as man or equipment, insulated from ground. When potential of the chge becomes high, a mere contact of a charged body with insulated cap wires is sufficient to cause the detonation of a cap. The shunt shield insulation of the wires offers no protection against such high potentials

Static electricity can also be generated by the movement of particles, such as dusts or snow driven by high winds, or by steam driven by high pressure

Atmospheric static is a particular hazard in seismograph work, because the work is frequently carried out at high altitudes and in areas subject to dust storms, snow storms and low humidity. Another danger exists when there are long electric cap wires, generally required in seismographic work

Recommended precautions to be taken where static electricity can be generated mechanically include: 1) All parts of moving equipment should be electrically connected to a common point, which is attached to a good earth ground rod; 2) All conductors and metal parts of such a system should be kept away from blasting caps and circuit wires or otherwise electrically insulated; 3) The ground wires and earth ground rod for such a system should be kept away from rails, or from wiring and piping which might conduct stray electric currents from these sources to the blasting site; and 4) All such moving equipment should be shut down while blasting circuits are being connected and until the blast has been fired (Ref 27, pp 170–71 and Ref 30)

III Static Hazards in Pneumatic Loading Systems. When powdery materials such as AN–Fuel Oil mixtures are conveyed pneumatically for loading into blastholes, some static electricity is developed. Since loading is conducted over electric blasting cap leg wires, there is a danger of premature initiation, although the charges are generally small (Ref 27, p 171)

Note: A discussion of the pneumatic loading system and the hazards involved is presented in the DuPont Technical Bulletin, entitled "Static Electricity Hazards and Their Control in Pneumatic Loading Systems"

IV. Stray Electric Current. This includes current flowing from a source such as a battery, a generator or a transformer, thru power lines to electrical equipment, which returns to that source over whatever paths are available to it. These paths include additional conductors insulated from ground (such as electric cables), conductors not insulated from ground for electric haulage (such as rails) and the earth itself. If the supply or return conductor betw the source and the load should be interrupted, as by the opening of a fuse, dangerously high ground currents may result in an earth grounded system. The first defense against this hazard requires that continuous metal objects (transmission lines, haulage rails, etc) be kept away from the immediate vicinity of electric blasting circuits. In addition, measurements for stray ground currents should be conducted before caps are introduced in a particular operation. The proper technique for conducting stray current tests requires the use of both AC and DC voltmeters capable of reading 0.05 volts. The AC or DC readings across the one ohm resistor should not exceed 0.05 volts and the maximum current that can be tolerated is 0.05 ampere, or one-fifth of the minimum firing for commercial blasting (0.25 amp). This is in accordance with the recommendations of The Institute of Makers of Explosives

The hazards which stray currents present to electric blasting caps can be greatly diminished by isolating all electric power lines from ground except at the power source and providing a separate, common bus, bonded to the frames of all electrical equipment. All rails, pipes, armored cables, ventilating ducts and other conductors not designed to carry power should be electrically bonded together at frequent intervals and connected to a single earth ground which should be isolated from the power ground or neutral bus. Isolation of all leading wires and blasting circuits from ground and from possible current carrying conductors are additional measures which will reduce the hazard. The insulated foil that covers over the shunted leg wires on DuPont EB Caps provides the final defense against stray current hazards and should not be removed until the loading operation is completed and caps are ready to be connected (Ref 27, pp 171–73)

V. Galvanic Action Electric Currents are those caused when dissimilar metals contact each other directly or thru a conductive medium. For example, when in seismic shooting an aluminum loading pole replaced the heavier wooden pole, two premature explns took place. This happened because the combination of aluminum and its steel casing in the alkaline drilling mud formed a sort of battery and this developed galvanic current causing prematures. This shows that no conductive devices should be allowed to enter a borehole contg electric caps. This rule of not allowing the presence of two dissimilar metals while loading a hole, is especially important to observe when working in salt water (Ref 27, p 173)

VI. High Voltage Power Transmission Lines Electric Currents. The chief hazard associated with operations near such lines is the possibility of being electrocuted should a cap leg wire or leading wire be thrown by the blast to make contact with the live power line

The following recommendations are offered when blasting near power transmission lines:

1) The shot point should never be located closer to a line than a distance equal to the length of both leg wires. Taking the length of both wires is necessary (except when duplex wire is used) because the single wires may part and form a single long conductor

2) If the shot point cannot be relocated to comply with the above "Ledcore" MS Delay Assemblies (See Ref 27, pp 107–09 & 184), "Primacord" (not wire bound) (pp 101–05 & 180–81) or "Primaline" should be substituted for most of the electric blasting caps and leading wires. Use of a cap and safety fuse assembly to associate the above cords provides a completely non-electric system

3) If "Ledcore" units, "Primacord" or "Primaline" are not available, the cap leg wires should be securely anchored at or near the shot point

4) If cap leg wire or a leading wire should happen to be thrown over a power line the blasting crew should not attempt removal of the wire, but request the power company to do this

Before conducting electric blasting operations in the vicinity of power transmission lines, it is advisable to check (in the presence of power company representative) for ground currents. Also it is advisable to determine whether or not measurable voltages can become more hazardous when peak power levels are transmitted. To conduct these tests, a leading wire is connected to a ground rod driven in the vicinity of the blasting site, while the other end of line is connected to one terminal of the voltmeter. The other terminal of the voltmeter is connected to another ground stake which may be used to probe the earth at different points around the blasting site. Voltages detected in this manner should be considered potential sources of stray current and tested in the manner described here under "Stray Electric Currents" (Ref 27, pp 173–74)

VII. Radio Frequency (RF) Energy. Before beginning any operation involving the handling of electric blasting caps, a survey should be made to investigate potentially hazardous sources of RF energy. The Institute of Makers of Explosives published a pamphlet which includes the identification and classification of RF energy sources, such as radio, television or radar transmitters. This publication is entitled: "Radio Frequency Energy–A Potential Hazard in the Use and Transportation of Electric Blasting Caps (Ref 27, p 174 and Ref 30)

VIII. Electrostatic Discharges and Sensitivity of Explosives to Initiation by Them, entitled in PATR **4319**(1972) as **Electrostatic Sensitivity Testing for Explosives**

In addn to a brief definition of static electricity and its discharge given under Introduction to this section in item II, entitled "Static Electricity Charge", the following discussion given in PATR 4319 (Ref 35, pp 4–5) may be included:

It is important to distinguish between two types of electrostatic discharges: sparks and arcs

A **spark** was defined by Loeb (Ref 1) as an "unstable and discontinuous occurrence marking the transition from one more or less stable condition of current between electrodes in a gas to another one. It may also occur that the transition process may start but fall short of achieving the transition owing to circuit conditions such as power supply (restrictions). This definition emphasizes the transitory nature of a spark, but it is too broad for the purposes of investigation discussed in PATR 4319 (Ref 35). For its purpose, a spark is defined as a "dielectric breakdown of a gas between two electrodes in which the liberation of secondary electrons from one of the electrodes is the major feedback mechanism necessary to sustain a discharge". The liberation of a secondary electron is usually due to both the impact of ions formed in the discharge region upon the cathode and to photoelectronic ionization from the cathode. Protons are produced thruout the gap by the discharge. Spark breakdown is governed by Paschen's Law, defined in Ref 1, but not in Ref 35. There is a minimum voltage below which a spark will not pass between two electrodes in a given medium. Such minimum is 275 volts for air. A spark is not stable because the cathode will heat up to a point where thermionic emission becomes the important mechanism for the liberation of electrons. The spark is then said to be forming an arc, but for this transition it is necessary that sufficient energy be supplied by the source, such as a charge capacitor

An **arc** may be defined as a "stable discharge between two electrodes in which thermionic emission is the feedback mechanism responsible for sustaining the discharge". An arc can be formed in two ways: 1) as a result of spark formation evolving into an arc (as mentioned above) and 2) as the result of an initial contact and subsequent separation of two electrodes carrying current. In the latter case, the arc is initially formed by intense Joule heating of the touching electrodes at the point of contact. Upon separation, the hot cathode emits sufficient electrons to maintain the discharge. An arc is usually accompanied by ejection of hot metal and emission of relatively high frequency radiation (into the ultraviolet). An arc may form even at very low voltages, while spark cannot form in air at voltages below 275, but always forms at voltages above it

One often speaks of sparks as "gaseous discharges", since contact betw the electrodes is not required, while arc can be defined as "contact discharges" or "metal-metal discharges" (Ref 34, pp 4–5)

Moore, Sumner & Wyatt (referred in PATR 4319 as MSW) were the first to recognize the distinction between the contact and gaseous discharges in igniting expls, as can be seen from their work at ERDE (listed here as Refs 16 & 17)

Historical. Accdg to information at our disposal, the earliest investigation of electrostatic discharge hazards associated with manuf, storage, and usesof expls was conducted beginning in 1942 at the USBurMines. This was reported in TechNote No 23 (Ref 3) and later in RI 3852 (Ref 4). Some work on electrostatic charges was also done as early as 1942 at the Bureau of Standards and reported in Circular C438 (Ref 2). The original method of investigation of electrostatic discharges, described in Ref 4, was modified and in such form described in Ref 13. In 1947 electrostatic charges were detnd by Fleischer & Burtle (Ref 5) for various Lead Azides and in 1949 Rathburg & Schmitz (Ref 6) determined sensitivity of primary and initiating expls to electrostatic charges. In the same year, Wilkerson (Ref 7) reviewed the literature describing electrically conductive materials in bridgeless electric primers. In 1950, Harvalick (Ref 8) prepd a literature survey on expl initiators in which conductive mixtures are described and some interpretation of results is given. Accdg to his survey, conductive mixts possessing good simultaneity must have the following characteristics: (a) one joule or less for initiation, (b) output energy sufficient to initiate HE's, and (c) two or more items contd in the mixt should fire with a spreadof 0.10 microsecond or a std deviation of 0.30μsec. In 1953, Henry (Ref 9) recommended exposure to radioactivity for discharging electrostatic charges of materials and the same method was recommended by Quinton (Ref 11). In the same year Bulgin described some precautions to be taken against electrostatic chges when handling expls. A brief resumé of his paper is given here under Ref 10. Peace (Ref 12) reported that RDX, Tetryl and some other expls developed electrostatic chges when being sieved thru 100-mesh after being dried at 50°. Damon and vanDolah reported in 1954 (Ref 14) the electrostatic spark test for 9 samples of expls. Braid et al (Ref 15) discussed prevention of accumulation of charges on powders and prevention of explns during hand operations on detonators (See also below under "Description of Methods"). In 1956, Moore, Sumner & Wyatt of ERDE (Refs 16 & 17) and Moore (Ref 18) described tests for detng electrostatic spark sensitiveness of initiators. Their work was continued at ERDE by Sciafe (Ref 11) and Wyatt (Ref 23). Two other papers of Wyatt are listed here as Refs 19 & 20 (Resumé of their work is given in Ref 35). Kirk (Ref 24) in his survey presented the problems in the field of electrostatic sensitivity and electrical initiation. Jackson of PicArsn (Ref 25) described the results of study aimed at: a) determining the electrostatic sensitivity of secondary expls and b) developing conductive mixtures which would give reproducible functioning times in the range of 0.10 microsecs or a standard deviation of 0.030 microsec. Résumé of his work is given below under "Description of Methods". Clear of PicArsn (Ref 26) described lab procedures for detng sensitivity of expls to electrostatic discharges. Blasters' Hdb (Ref 27) discussed hazards caused by various extraneous electricities, which is described in detail at the beginning of this section. Hannah & Polson of Burlington, Iowa (Ref 28) reported static electricity developed during LA handling. Montesi (Ref 31) described a fixed gap electrostatic discharge apparatus for characterizing expls. Perkins of PicArsn (Ref 32) gave a survey of methods of testing for electrostatic sensitivity of expls, while Gentner (Ref 33) described a test for Comp B. Satyavratan & Kurti of India (Ref 34) discussed reduction of static electricity in factories manufg expls. Westgate, Pollock & Kirshenbaum (Ref 35) gave a survey of electrostatic testing of expls as practiced at PicArsn and at NOL, as well as at some other establishments (See below under "Description of Methods, etc")

Description of Methods Used for Evaluating Electrostatic Charges and Sensitivities of Explosives to Electrostatic Discharges.

Accdg to Brown et al (Refs 4 & 13), the simplest and most useful means of testing samples of materials for thier sensitivity to spark initiation is to subject them to single discharges from a condenser that has been charged to a high voltage. The maximum energy of the spark discharge to

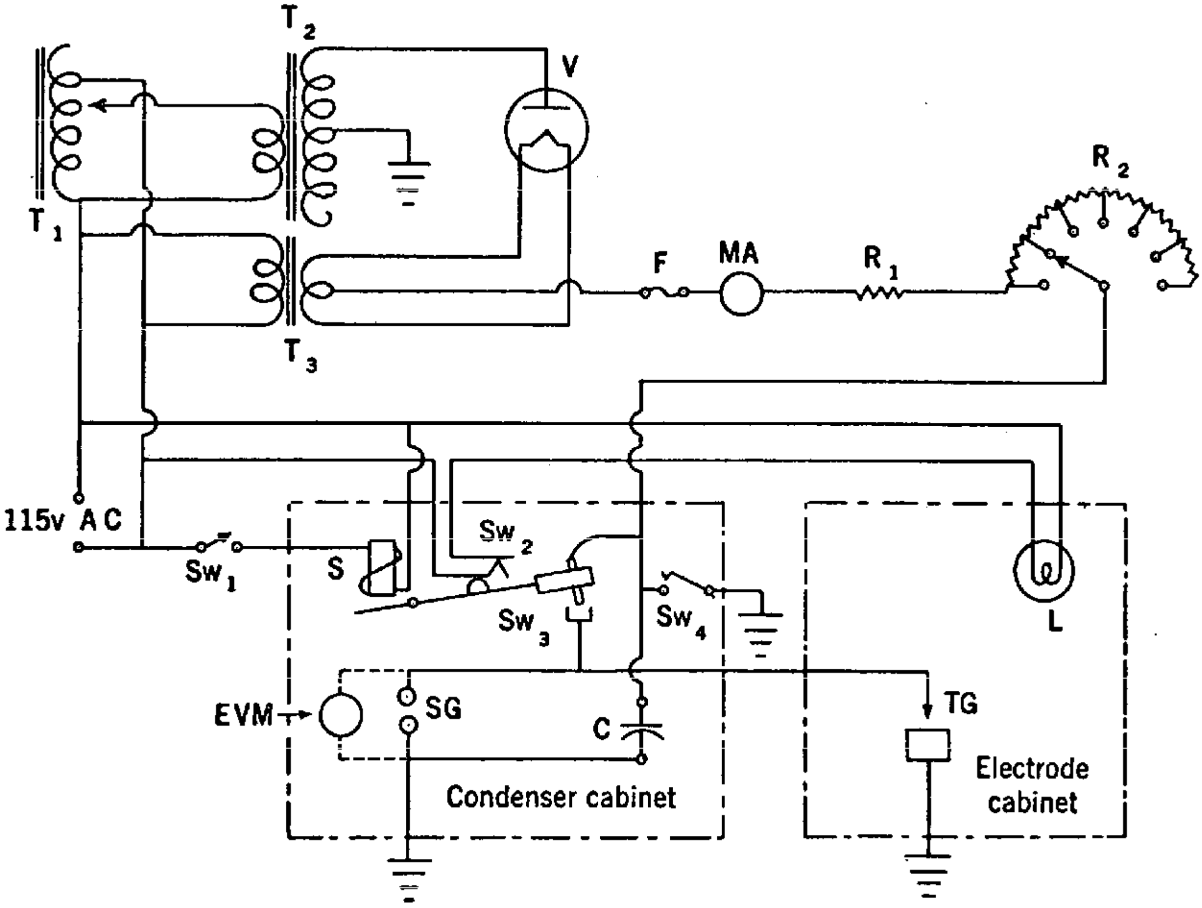


FIG E11 Spark sensitivity apparatus

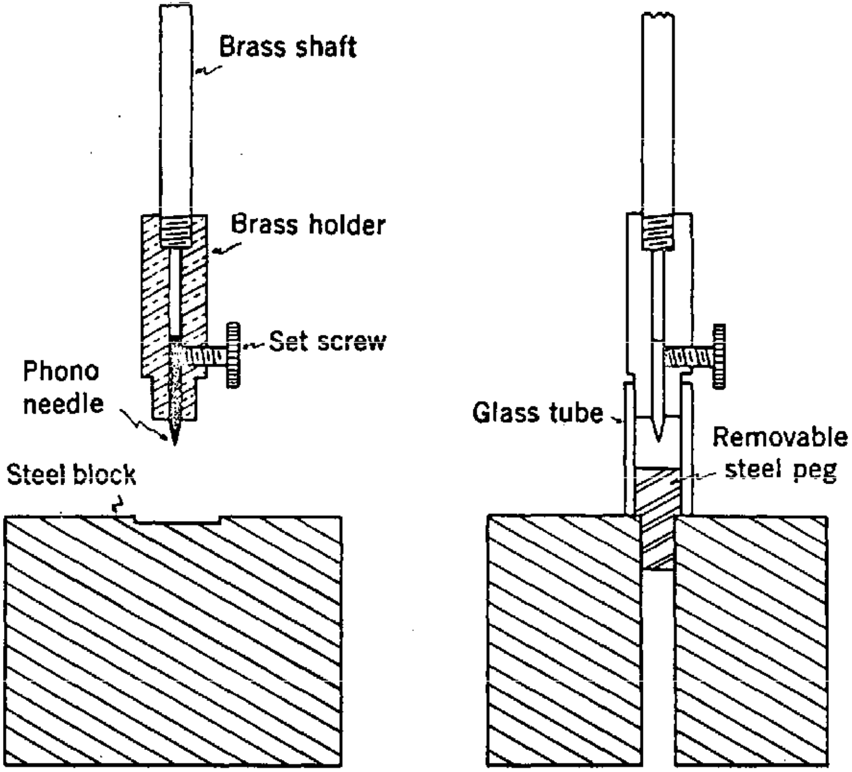
which the material can be subjected without being ignited has been chosen as a criterion of its sensitivity. Ignition of a material by an electrostatic discharge depends on other factors as well as energy of discharge, but present results indicate that the energy factor is the best single criterion

The energy E (joules) of the spark discharge from a condenser of capacity C(microfarads) charged to a potential V (volts), assuming negligible losses is:

$$E = 5 \times 10^{-7}CV^2$$

In order to understand the principle of the method of testing we are describing here the arrangement of Ref 4, which seems to be less complicated than its modification described in Ref 13

A schematic diagram of spark sensitivity apparatus, shown in Fig E11 contained a condenser (C) charged from a half-wave rectified high-voltage supply and is discharged thru the material placed on a steel plate or peg. The discharge occurs between a pointed electrode and the plate holding the sample (TG). For the pointed electrode, a steel phonograph needle is used because it is easily replaceable and allows one to use a new point for each test if necessary. Detail of electrode system using phonograph needle is shown in Fig E12 for unconfined and partially confined samples. The voltage can be adjusted by means of a variable transformer (T_1 of Fig E11) and measured with the electrostatic voltmeter (EVM) or



UNCONFINED

PARTIALLY CONFINED

FIG E12 Electrode systems

with the calibrated spark gap (SG). During a test, the condenser is left connected to the power source thru a high resistance (R_1 and R_2) between the source and the interchangeable condenser (mica or air dielectric)
Circuit Designations of Fig E11: C—condenser; TG—test electrode gap; SG—calibrated spark gap; EVM—electrostatic voltmeter; S—100V AC solenoid to operate mercury cup switch SW_3; L—electrode cabinet safety and illuminating lamp; SW_1—remote-control switch to operate solenoid; SW_2—safety light switch; SW_3—HV mercury cup switch; SW_4—condenser cabinet door interlock switch; R_1—protective water resistor; R_2—tap resistor – 1meg to 500meg; F—1/32amp HV fuse; MA—0-25 milliamperes; V—866-A rectifier; T_1—0-135 volt varitran; T_2—plate transformer, 7500 volts rms, each side, 30ma; T_3—filament transformer, 2.5volts/5amp/10000volts insulation

The resistor (R_2) is made sufficiently high so that several seconds are required to charge the condenser (C). During discharge the (C) remains connected to the power source, but the high resistance of (R_2) prevents any appreciable charge from flowing into the (C) during the time of discharge. To obtain a discharge, the high-voltage side of (C) is connected to the point electrode by means of remotely controlled mercury switch (SW_3), and the electrode is moved down by means of a screw until the gap is small enough for a discharge to occur. This procedure prevents any loss of energy by sparking at the switch and minimizes the effects of leakage in the system. The condenser must be shorted by the shorting bar (SW_4) before the door to the condenser cabinet can be opened. The light in the electrode cabinet is automatically turned off by the switch (SW_2)

when the condenser is connected to the electrode and it is unsafe to open the cabinet when the light is off

Procedure: Samples can be tested either unconfined or partially confined. In the unconfined test, a sample of approx 0.05g is placed into a shallow depression on steel block (See TG of Fig E11 and steel block of Fig E12) and flattened out with a spatula. In the partially confined tests the sample of approx 0.05g is introduced into a soft- glass tube (approx 7mm ID by 18mm long), which fits over a metal plug. Prior to introduction of samples, the needle-point electrode is raised up until the gap is greater than the critical gap for discharge at the test voltage. Then the high-voltage terminal of the charged condenser is switched to the needle-point electrode by means of mercury switch (SW_3) and the electrode is moved down rapidly until the discharge occurs

Note: In the procedure described in Ref 13, p 3, the needle is moved down by means of single-stroke, mechanically-operated electrode-control assembly, preadjusted so that the gap is less than that required for the discharge. The procedure with this device (which is reproduced here as Fig E13) prevents any loss of energy by sparking at the switch, minimizes the effects of leakage in the system, and allows only one spark to occur

For each type and condition of sample, an attempt is made to determine the maximum energy at a fixed voltage for which no ignition takes place. The energy at a given voltage is varied over a range by using condensers of different capacities so that a plot may be made of ignition probability versus energy, and the energy corresponding to zero ignition probability is detd from the curve. Most of the results reported by Bur Mines have been obtd by making 10 tests at each spark energy and such number of tests is considered sufficient to obtain fairly accurate results

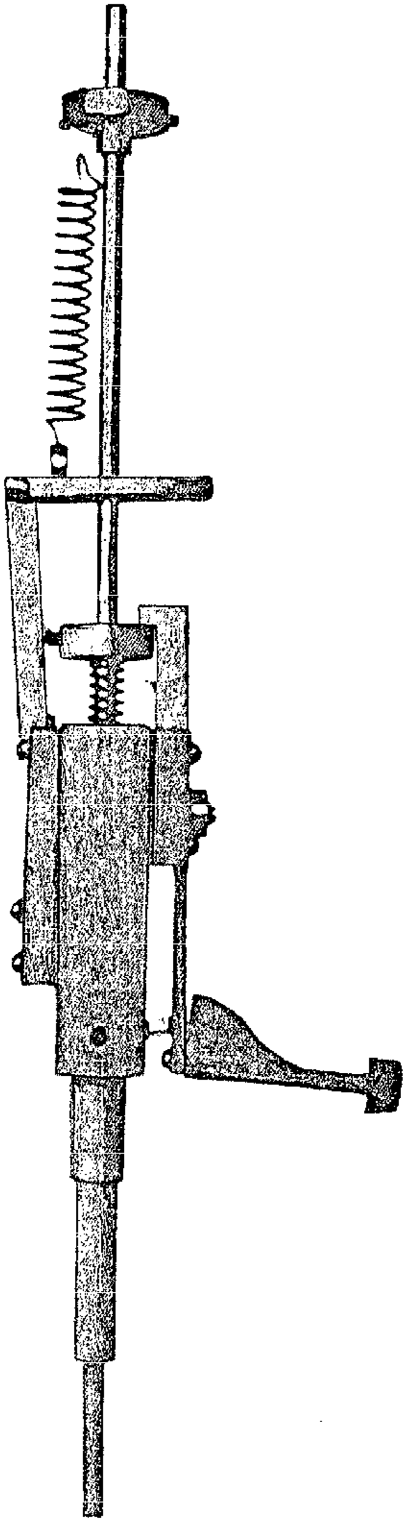


Fig E13 Closeup of single stroke, mechanically operated, electrode-control assembly

Table E6

Highest Electrostatic Discharge Energy at 5000 Volts for Zero Ignition Probability for Representative Materials

Material Description	Highest Energy (joules) for Zero Ignition Probability		Type of Ignition	
	Unconfined	Confined	Unconfined	Confined
Amm Picrate (as received)	>12.5	6.0	None	Deton
Amm Picrate (thru 100 mesh	0.025	6.0	Deflgrn	Deton
Black Powder (KNO_3 type) (thru 100 mesh)	>12.5	0.8	None	Deflgrn
Black Powder ($NaNO_3$ type) (thru 100 mesh)	>12.5	1.6	None	Deflgrn
Diazidodinitrophenol	0.012	0.012	Low-Order	Deton
Fusee Mixture	0.62	4.2	Deflgrn	High-Order Deflgrn
Igniter Composition	0.21	0.062	Deflgrn	High-Order Deflgrn
$KClO_3/Pb(SCN)_2$	<0.00075	<0.00075	Deflgrn	Deflgrn
Lead Azide	0.0070	0.0070	Deton	Deton
Lead Styphnate	0.0009	0.0009	Deton	Deton
Magnesium Powder, grade B	0.007 to >12.5	>12.5	Deflgrn	High-Order Deflgrn
Mercuric Fulminate	0.025	0.025	High-Order	Deflgrn
80/20-MF/$KClO_3$	0.054	0.054	High-Order	Deflgrn
Nitrocellulose (13.4% N)	0.061	3.1	Deflgrn	Deflgrn
Nitroglycerin (25°C)	>12.5	0.90	None	Deton
Nitroglycerin (60°C)	–	0.056	None	Deton
Nitrostarch	0.007	1.0	Deflgrn	Deton
PETN (as received)	>11.0	.21	None	Deton
PETN (100 mesh)	0.062	.21	Deflgrn	Deton
Smokeless Propellant (graphited; thru 100 mesh)	>12.5	>12.5	None	None
Smokeless Propellant (ungraphited; thru 100 mesh)	0.012	4.7	Deflgrn	High-Order Deflgrn
Tetracene	0.010	0.012	Low-Order	Deton
Tetryl granular (as received)	>11.0	4.68	None	Deton
Tetryl granular (thru 100 mesh)	0.007	4.38	Deflgrn	Deton
TNT granular (as received)	>11.0	4.68	None	Deton
TNT granular (thru 100 mesh)	0.062	4.38	Deflgrn	Deton
Tracer Composition	>12.5	6.2	None	High-Order Deflgrn

Note: The ignition of secondary HE's unconfined is apparently due to expln of fine dust dispersed into the air by the spark, whereas these expls under confinement detonate

Results of tests giving the highest electrostatic discharge energies at 5000 volts for zero ignition propagation for various materials, are given in Table E6, which is complied from table 1 of Ref 4 and Table 3 of Ref 13

Note: We described in detail the method of RI 3852 (Ref 4), because that report is out of print, but we do not think it is necessary to describe its modification (which is more complicated), given in RI5002 (Ref 13), because the report is still available from the Bureau of Mines. It would be sufficient to say that schematic diagrams of modified arrangement are shown in Fig 1&2 of Ref 13 and a photograph of the assembled equipment is shown in Fig 3. In Fig 4 is given a close-up of single-stroke mechanically operated elecrode-control assembly and in Fig 5 are shown electrode systems, same as given in Fig E12

The following general trends have been observed from the tests:

(1) The energy for ignition varies with voltage and overvoltage, but the trend of variation is not the same for all explosives

(2) Large particles ignite less readily than small ones, although for some expls under confinement this effect is not appreciable

(3) Except for primary expls, the degree of confinement usually has a marked effect on ease of ignition and completeness of propagation of the ignition

(4) The ignition of secondary HE's unconfined is apparently due to expln of fine dust dispersed in the air by the spark, whereas under confinement these expls detonate

(5) The ignition energies for unconfined samples of finely ground secondary HE's were invariably lower than for the same types of samples under confinement

(6) Metal powders are more sensitive when tested unconfined

(7) Black Powder is much more sensitive when tested under confinement

(8) Moist BkPdr (up to 7% moisture) is more sensitive than dry BkPdr when tested under partial confinement

(9) Less energy is required for ignition with a positively charged needle-point. This is probably because corona losses are lower (Ref 4, pp 4 & 7 and Ref 13, p 5)

Other Bureau of Mines investigations included comparison of some ignition energies with electrostatic charge energies that might be built up on the human body. Some capacitance measurements were made on several individuals in a building with a grounded steel framework. When an individual was insulated from the floor with either rubber-soled shoes or a 3/4 inch plate of glass, values of capacity ranging from 0.0001 to 0.0004 microfarad could be obtd. The value of 0.0004 μf was obtd when an individual wearing a leather jacket was leaning over a steel girder. Some tests were also made in a reinforced concrete bomb-proof shelter. Values of 0.00028 – 0.00032 μf were obtd consistently when the individual leaned against a wall. For two individuals holding hands, their capacitance was approx the sum of their separate capacitances. If we consider 0.0003 μf to be a reasonable value of capacitance under not too extreme conditions, then an individual charged to 10000 volts would have an energy of $5 \times 0.0003 \times 10^{-7} \times (10000)^2 = 0.015$ joule

On the basis of present information it is believed that precautions should be taken to prevent accumulation of electrostatic charges on individuals around operations involving handling of expls that may ignite with electrostatic energies of 0.015 joule or less. It must be emphasized that ungrounded pieces of equipment, wiring, or structures may acquire much larger energies than 0.015 joule and that precautions always should be taken to prevent accumulation of charges on such items around all operations involving expls, proplnts and pyrotechnic mixtures

It was also found that the capacitance of the human body depends on many factors, such as body structure, proximity of other conductors or insulators, etc and if there is contact between the body and some other object of high capacitance, the overall capacitance is approx the sum of the separate capacitance (Ref 4, p 7 & Ref 13, p 7)

Accdg to Braid et al (Ref 15), electrostatic charges which often occur when powders are stirred, sieved, etc may be prevented by using rough-surfaced rather than smooth-surfaced vessels or, alternatively, by coating the vessels spoons, sieves, etc by the powdered material itself using a suitable adhesive. Occurrence of explns during hand operations on detonators can be attributed to a spark discharge from the operator's body. Conditions favoring sparking are dry clothing combined with dirty moist hands which may contaminate supposedly insulated manipulating tools. Modification of the tool and /or positive grounding of the operator's body are recommended as preventive measures. Grounding of an operator can be done by means of a metal bracelet attached at a wrist. Other method suggested for preventing formation of sparks in handling detonators is the use of so-called " kittstick ", which is a piece of wood 5/16 inch in diam and 9 inches long with a slug of beeswax about 1 inch long attached to one end. With the sticky wax the operator picks up a paper disc and places it on the detonator. The other end of the stick must be kept clean and free from perspiration. Once in awhile, the stick and wax must be replaced by new ones. Combination of grounding of the body and use of kittstick is even better

It was found that an operator sitting on a wooden stool could generate (by frictional contact with the wood) sufficient electrostatic chge (such as 17000–20000 volts) to detonate sensitive expls if he approached them in such a manner that the chge becomes grounded thru it. A chge of this magnitude can be generated even in a room of high humidity (70–80% RH)

Table E7

Electrostatic Discharge Energy and Voltage for Zero Ignition

(Capacitance 500 MMF and Gap Setting 0.005")

Explosives	Voltage *	Energy * (Joules) (Jackson)	BurMines (1943) (Joules)	BurMines (1946) (Joules)	BurMines (1954) (Joules)	NOL (1959) (Joules)
B–HMX	40000	0.400	–	–	–	–
Composition B	12000	0.036	–	–	0.00062	–
Cu Chlorotetrazole	6000	0.009	–	–	–	–
Explosive D	40000	0.400	0.10	0.025	0.16	–
HEX M-24.1	20000	0.100	–	–	–	–
HEX–48	60000	0.900	–	–	–	–
MOX–2B	36000	0.290	–	–	–	–
PETN (2.6 microns)	12000	0.036	0.31	0.062	–	0.085
PETN/TNT	8000	0.025	–	–	–	–
RDX	20000	0.100	0.60	–	–	0.80
RDX/Acethylene Black 90/10	24000	0.144	–	–	–	–
Ditto 75/25	32000	0.256	–	–	–	–
Tetryl (on 100 sieve)	8000	0.025	0.020	0.007	0.0005	1.25
TNT (thru 100 mesh)	12000	0.036	0.077	0.062	–	–

* Maximum voltage and energy allowable above which ignitions occur

Abbreviations: MM – Micromicro
B–HMX – beta HMX – See AMCP 706-177 (1967), p 173
Comp B – See AMCP 706-177, p 46
Explosive D – Ditto, p 136
HEX–48 – Ditto, p 166
PETN – Ditto, p 276
RDX – See Cyclonite, p 69
TNT – Ditto, p 350
Cu Chlorotetrazole – Ditto, p 63
HEX M-24.1 – Ditto, p 164
MOX–2B – Ditto, p 215
PETN/TNT – Ditto, p 276
Tetryl – Ditto, p 335

within a short time after the operator entered the room.

Suggested (by *Braid*) means of avoiding explns caused by electrostatic discharges were to make the operator's body normally non-conductive, to have manipulating tools resistant to short-circuitry by perspiration and/or provide a positive grounding for the operator's body (Ref 15, pp 45–52)

Jackson of PicArsn (Ref 25) detd electrostatic charges of HE's and of some conductive mixture in the apparatus which seems to be similar to the one developed at the Bureau of Mines in 1942 and described in Ref 4 and in modified form in Ref 13 (See our Figs E11 and E12)

The powdered material (10-25 mg) was carefully placed in the standard die cup and leveled off with a wooden spatula. The loaded cup was placed directly under the needle point and on the base plate. A gap was set with a precision gauge of 0.005 inch thickness. The door of the test chamber was connected to the power supply by a micro switch which allowed the high voltage to be on only when the door closed. The charging voltage was set by adjusting the built-in powerstat. For each sample of a given material an attempt was made to ignite the sample by discharging the capacitors. Ten tests were made at each energy or voltage level. The results were recorded as to the number of ignitions per voltage level (See Table E7, which contains values of Table I and II, pp 5–6 of Ref 25 arranged in alphabetical order)

Note: The results in Table E7, besides giving the electrostatic sensitivity in terms of energy, also show the voltage **that is** necessary when a 500 MMF (micromicrofarad) capacitor is used. It should be noted that Tetryl has a zero initiation energy value lower than 0.015 joule. This amt of energy (0.015 j) is the most commonly accepted energy value that an individual may have with a chge of 10000 volts, assuming that the body capacity is 300 MMF. It is the opinion of some operators that the spark ignition energy varies with the voltage and that as voltage is increased the zero-ignition energy decreases. This means that the higher an individual is charged, the greater is the potential hazard. The results of Table E7 also show that the order of sensitivity of 6 expls tested at BurMines and NOL is nearly the same as obtd by Jackson, except for Comp B, Expl D and Tetryl

Jackson also detd functioning time of conductive LA-graphite mixts, placed on top of conductive PETN-graphite mixts fired in the fixture shown in Fig E14

The materials for conductive mixtures were finely pulverized by ball milling using chloroform or absolute methanol as a medium with steel balls 1¼-1¾ inch diam. The materials and medium were placed in a rubber container and rolled on a rolling mill for 2-3 hours. For blending the calcd wts of dry materials were tumbled in a rubber beaker which was rotated at 25–35 rpm by an air-driven motor. The resulting blend was weighed in required amt on a Roller-Smith Balance and poured into test fixture shown in Fig E14, held in a loading assembly. Then the sample such as PETN/graphite was consolidated with a Denison Midget Press (1 Ton on 1-1/2 inch diam ram) and on top of it placed a primary chge of conductive LA/graphite. The usual pressure for loading was 16000 psi and *firing energy 1.12 joules*

The functioning time in microseconds of three series of eight detonator test fixtures of 30 mg conductive LA/graphite-98.7/1.3 and 220 mg conductive PETN/graphite-95/5 loaded at 16000 psi are shown in Table III of Ref 25 (not reproduced here). The average times: 2.102, 2.105 & 2.114 are in good agreement, but their standard deviations (γ): 0.077, 0.075 & 0.069, respectively are greater than the desired 0.030 microsecond

The effect of variation in sample sizes of conductive LA/graphite-96.7/3.3 and conductive PETN-graphite-95/5 loaded at 16000 psi is shown in Table IV of Ref 25 (not reproduced here). The 1st three columns show the average functioning times of 30 mg LA/graphite and 220 mg PETN/graphite as 2.031, 2.036 & 2.120 with deviations 0.135, 0.142 & 0.068, respect-

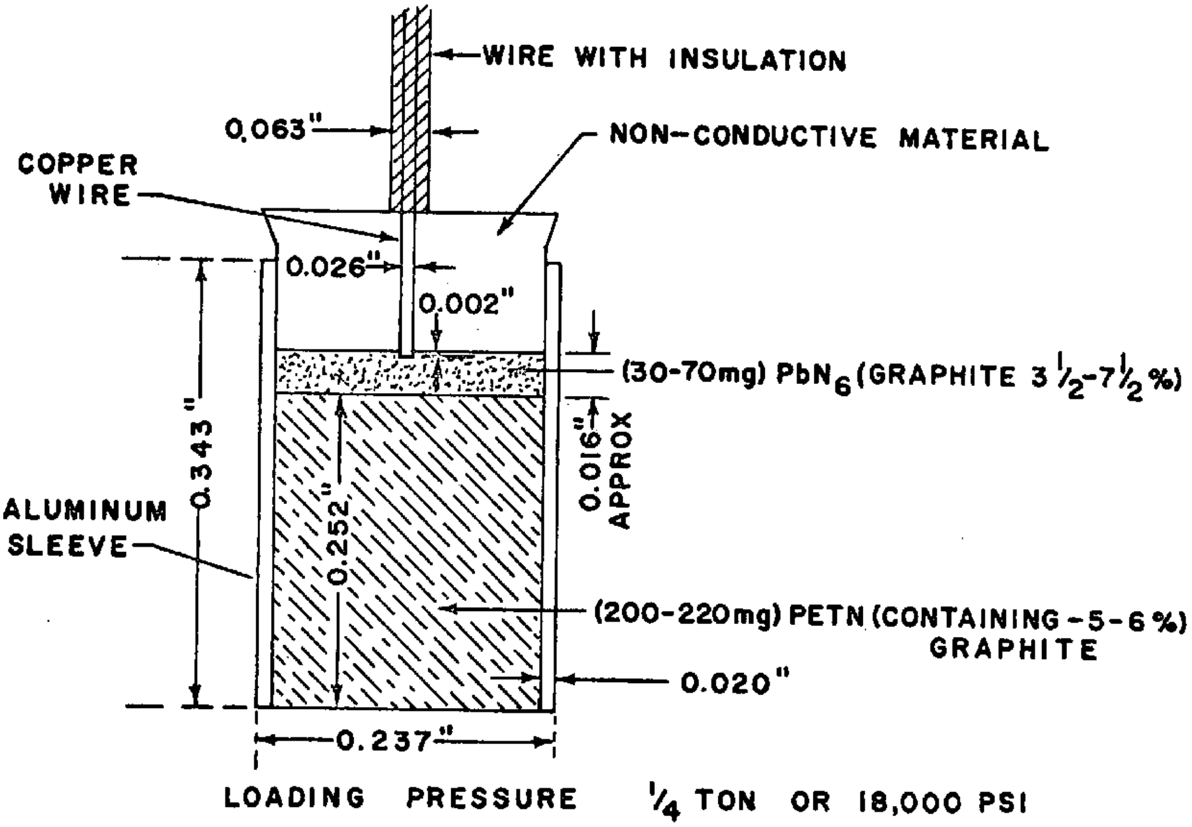


FIG E 14. FIXTURE USED IN CONDUCTIVE MIXTURE TESTING

ively which are greater than desired microseconds. The other three series show the functioning times of 50, 60 and 70 mg LA/graphite with 200 mg PETN/graphite as 1.809, 2.019 & 1.952 with corresponding deviations 0.068, 0.119 & 0.087

The evaluation of 60 mg of conductive LA/graphite-95/5 and 200 mg conductive PETN/graphite-95/5, loaded at 16000 psi is shown in Table V of Ref 25 (not reproduced here). The average time of tests (except for test 25 which is erratic) was between 1.751 to 1.787 with deviations between 0.032 to 0.074. This shows that increases of graphite content from 3.3 to 5.5% in LA/graphite resulted in improved std deviations. Still better results were obtd when graphite content in LA mix was increased to 7.5%. In two tests the functioning times were 1.692 & 1.706 with corresponding std deviations 0.031 and 0.030 microsecond

Jackson also conducted the so-called *simultaneity tests*. For this eight of the loaded fixtures (with Tetryl pellets attached) were mounted on a board and fired electrically; the light emitted by the Tetryl was recorded by a streak camera. The energy used to fire these loaded parts was approx 1 joule. This energy was obtd by discharging a 1microfarad condenser charged at 1500 volts. By comparing the values of functioning times and of standard deviations, listed in Tables III, IV & V of Ref 25 it becomes possible to judge simultaneity

Westgate, Pollock & Kirshenbaum (Ref 35) reviewed briefly the work done previously at USBureau of Mines (Refs 3,4,13,14 & 24); at Burlington, Iowa (as described by Hannah & Polson in Ref 28); at NOL (as described by Montesi in Ref 31); Picatinny Arsenal (as described by Jackson in Ref 25, Perkins in Ref 32 and by Gentner in Ref 33). A detailed review is given of work done at ERDE by Moore, Sumner & Wyatt (abbreviated as MSW) and collaborators, described in Refs 16, 17, 18, 19, 22 & 23

In "Introduction" of Ref 35, pp 2-3 it is stated that *spark* (See its definition and distinction from *arc* given on pp 4-5 and under item VIII of this section) sensitivity testing can provide an important measure of the electrostatic hazards associated with the handling of primary expls. The threshold (or minimum) energy required for initiation is of particular concern. The usual approach to determine this minimum energy is to discharge a capacitor thru a spark gap in or near the expl and to reduce the stored energy of the capacitor until no ignition occurs in a specified number of trials. Despite the apparent simplicity of this technique, the large number of variables present in this test makes a quantitative interpretation of the results very difficult. Two other methods were proposed. One was to measure and to control the energy actually delivered to the spark gap. This technique was followed by MSW and later by Genmer. The other, more traditional approach, was to abandon extensive analysis of the circuit energy delivery, but to design a test apparatus that was capable of producing comparison among expls and was safe and convenient. The rationale in this case was that it was considered feasible to determine the relative sensitivity or ranking of the expl tested among other expls despite the fact that an absolute minimum energy was not measured. This (approach has been followed in the tests conducted at PicArsn and at NOL, but none of these tests has been sufficiently evaluated to determine the fraction of stored energy actually delivered to the spark gaps or to det the delivery

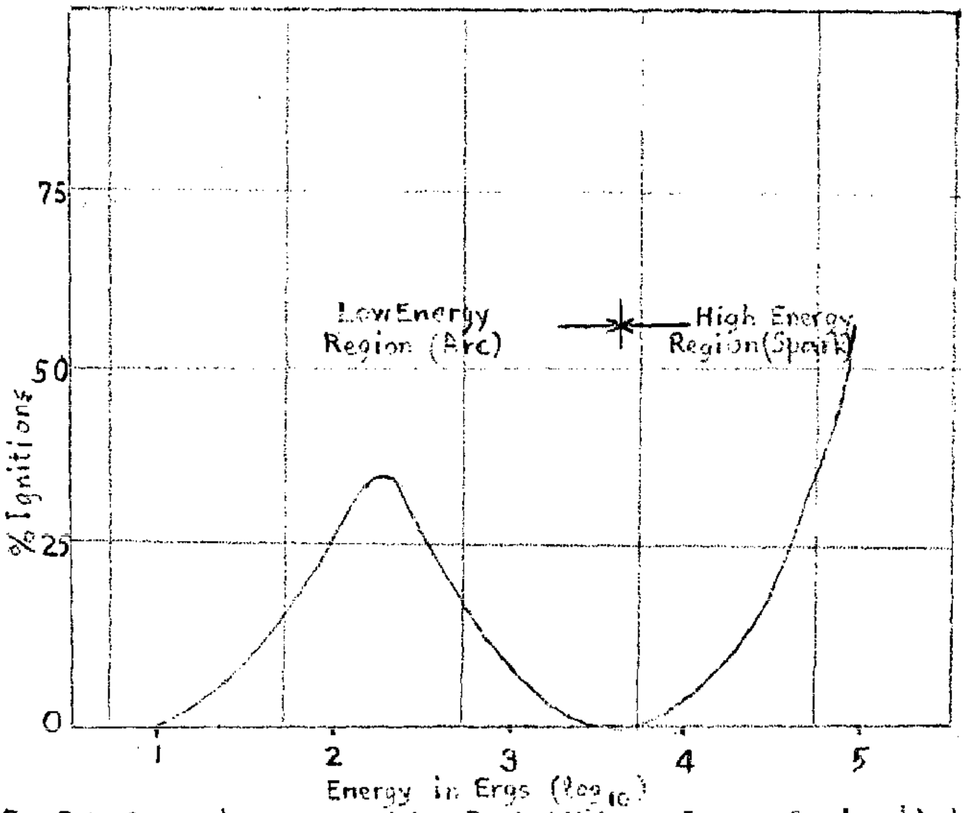


Fig E15 Dependance of Ignition Probability on Energy for Lead Azide
(C = 1000 pf)

rate. No attempts were made to correlate the results of methods used by MSW or by Gentner with those reported by PicArsn or by NOL. Consequently, there is an evident need to det whether the present sensitivity tests accurately reflect the true sensitiviness of expls and the purpose of rept listed here as Ref 35 is to review these methods and to outline a research program for developing improved testing procedures (Ref 35 pp 2–3)

Moore, Sumner & Wyatt (abbrd as MSW) (Refs 16, 17, 18, 19, 22 & 23) were the first to recognize the distinction between the contact (arc) and gaseous discharge (spark) in igniting expls. The ignition of powdered LA (Lead Azide) (as a result of these two mechanisms) is shown in Fig E15, which is a copy of Fig 1 of Ref 35, p 7. The behavior of the various factors, as shown in this Fig, can only be observed when the apparatus is arranged so that actual contact can occur between electrodes. Accdg to MSW, the peak of initiation probability at low energies occurs when the voltage across the storage capacitor is about 300 volts and is nearly independent of capacitance The peak thus occurs when spark formation is barely possible. Ignition at low voltages must therefore be due to contact discharge (arc). The decrease is probability of ignition above the low energy peak is due to energy loss from the arc to spark formation. Ignition in the high energy range is evidently due to spark discharge alone. LSt (Lead Styphnate) does not exhibit as large a difference in energy between the contact and gaseous discharge regions (Ref 35, p5)

MSW investigated the effects of a wide range of parameters. Two techniques were used: a capacitor discharge betwn electrodes with a fixed gap, and a capacitor discharge between moving or approaching electrodes. In the latter technique, actual contact betwn electrodes permitted arc discharge to occur. The moving electrode technique had the advantage of mechanical simplicity in that no gap needs to be set. The disadvantages were that the electrode spacing for arc initiation was necessarily ambiguous due to spark delays and corona losses. It is generally assumed that a spark discharge will occur betwn approaching electrodes when the gap voltage will equal the breakdown voltage. However there may be a delay in discharge of the order of microseconds to microseconds. This delay is statistical in nature and depends upon voltage, electrodes, illumination, and ambient gas. For an approaching electrode moving at 140 cm/sec and a spark delay of ca 10^{-4} sec, the uncertainty in gap width is ca 0.014 cm (0.006 inch). The corona losses may be as large as 10% of the energy stored in the capacitor. If electrode movement is very slow, powder may be attracted to the electrode, thus altering the explosive–electrode geometry. On the other hand, the fired electrode configuration allows one to set an accurate gap and eliminates corona losses; however, a fast low-loss switch is required to transfer energy from storage capacitor to the gap (Ref 35, pp5-6)

Following is a summary of some of the tests by MSW of ERDE:

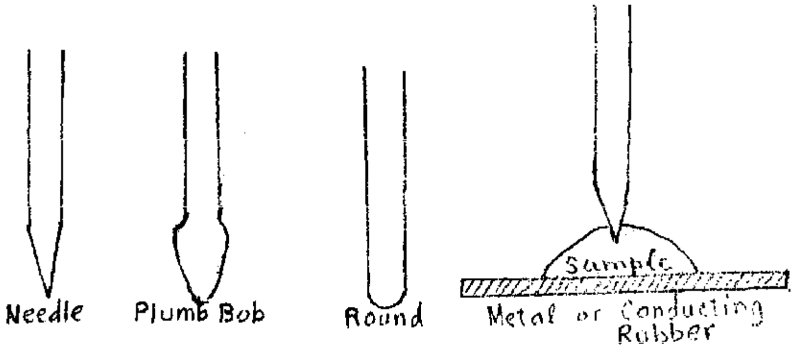
1) *Effect of Fixed Gap Vs Moving Electrodes.* In the gaseous discharge region, the curves of initiation probability vs discharge energy were very nearly identical and there seems to be no advantage in using moving electrodes (Ref 35, p6)

2) *Effects of Electrode Shape and Material.* In all experiments, one electrode was a flat metal plate, while the other was one of those shown in Fig E16. Generally, the probability for ignition was higher for the plumb-bob than for the steel phonograph needle or ball electrode. A study also was made of electrode materials, but no importance differences were observed among a variety of metals (Ref 35, p 9)

3) *Effect of Humidity.* Extensive data were not taken by MSW on the effects of humidity. Instead they chose to control the RH at approx 40% for all the tests. Since LA is not hygroscopic, no effects due to changes in surface conductivity would be anticipated for himities below 70% and this was shown to be true by the authors of Ref 35. Loeb (Ref 1), however, pointed out that impurities in the ambient gas can strongly affect the nature of the spark; hence, humidity can play an important role on that account (Ref 35, p 9). Cobine (Ref 21a) has shown that breakdown voltage across a gap decreased with increase of RH (See Fig 10 in Ref 35)

4) *Effect of Gap Length.* In the gaseous

FIG 16 – ELECTRODE SHAPES AND SAMPLE GEOMETRY



discharge region, the ignition probabilities as a function of energy were largest for gaps of the order of 0.003–0.006 inches. The probability functions decreased by factors of two to three for much wider gaps, i.e. in the range 0.020–0.040 inches. Some std tests use gaps as wide as 0.050 inch (Ref 35, pp 9 & 11)

5) *Effects of Size of Storage Capacitance.* Regardless of the stored energy, no ignitions were observed for capacitances below a certain limit. The minimum energies for both contact and spark initiation were nearly identical for a range of capacitance 100 to 1000 pf (Ref 35,p 11)

6) *Effect of the Series Resistance.* When a high series resistance was also present, a minimum capacitance effect was observed. Series resistance was inserted in two ways: as a lumped element in the circuit, and as a conducting rubber electrode w or w/o addnl resistance. The rubber electrode was supposed to simulate, in the laboratory, the resistance of a human. The lumped series resistance primarily affected the form of the discharge, i.e. changed an oscillatory current to a unidirectional one. The lumped element resistance also lengthened the duration of the discharge without a concomitant decrease in minimum energy. The rubber element lowered the minimum energies for spark discharges (Ref 35,p 11)

7) *Particle Size Effects.* Generally expls of smaller particle sizes were more sensitive. Colloidal LA was the most sensitive of all the expls investigated by MSW. The effects of particle size distribution and shape were not investigated by MSW (Ref 35, p 12)

8) *Energy Considerations and Spark Characteristics.* Many effects listed under items 1 to 7 play important roles in spark initiation because they affect the amt and rate of energy transfer from the storage capacitor to the spark gap. The energy delivery can be detd in part by observations made on the electrical circuit. MSW carried out these measurements for a number of tests and provided some analytical treatment of their circuits. The only quantitative result which can be drawn from their work is that only about 15% of the stored energy was actually delivered to the spark gap when a series resistance greater than 1000 ohms was placed in the circuit (Ref 35,p 13)

Note: A more quantitative analysis was made later by Gentner (Ref 33) whose work is also described in Ref 35, pp 13-21

The following (other than MSW) investigations are listed in Ref 35,p 12: Hannah & Polson (Ref 28), using an electrified vibrating probe detd a minimum energy of 10^{-2} erg for ignition of LA. They also described a more conventional test, but, based on their report, it was not possible to calculate the energy delivery. Crane, Smith & Bullfinch (ref 21) performed a statistical analysis to investigate a number of parameters in the ignition of Mg, but some of the conclusions are difficult to relate to electrostatic ignition of primary expls because Mg has high conductivity. However, one important point was made concerning the effects of humidity. Since one would not expect that humidity affects conductivity of such a conductive material as is Mg, it was concluded that humidity affected the nature of the spark itself. Another point made by the investigation was that it might be dangerous to extrapolate to probability for ignition curves to low energies, i.e. the tails of the distribution should actually be measured. This latter point has important implications in the detn of minimum energy, or for the estimation of acceptable energy levels in hazards analysis (Ref 35, p 12)

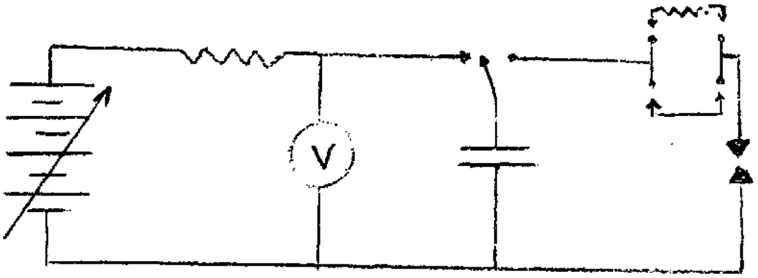
Gentner (Ref 33) studied the spark initiation of Composition B and LA. This work is particularly important because it treated the partition of energy among the elements of the circuit and gap in a quantitative manner. He showed that only 10% of the energy stored in the discharge capacitor actually was delivered to the spark gap when a series resistance was put in the circuit. Litchfield et al (Ref 29) studied the spark initiation of organic vapors and emphasized the effects of gap length and "quenching of the spark" by the electrodes, i.e. the electrodes conducting a large amt of heat from the discharge (Ref 35, p 12)

Under the heading "Present Testing Procedures", PATR 4310 (Ref 35, p 21) describes:

Picatinny Test No 1. It uses apparatus of Perkins (Ref 32) which consists of a motor-driven probe which moves to a set gap, remains stationary for a specified time, and then moves back to the starting point. The capacitance can be varied from 10^{-4} to 0.1 μf, and the voltage can be varied from 1.0 to 5k volts. No humidity control is present. The schematic circuit is shown in Fig E17, which is a copy of Fig 11 of Ref 35, p23

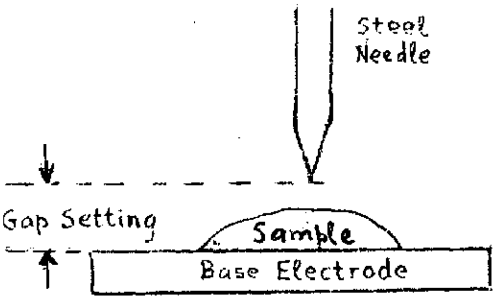
This test is used to characterize expls, (submitted by various directorates of the Arsenal) from the point of their relative sensitivity to electrostatic discharges. The test does not seem very reliable, since widely varying results are obtd, depending on the humidity, the operator and the test setting

A list of specific difficulties with the test is given on pp 21,24 & 25. The lack of humidity control is the most serious problem, and results



a Test Circuit

Fig E17 Picatinny Test No 1



b Electrode Configuration

of tests at RH above 20% are not reliable

In the present procedure, the operator chooses an intermediate capacitance and voltage, and reduces the latter until a minimum energy is obtd. Actually one should obtain a minimum energy for every given configuration and test setting. Wyatt (Ref 20) obtd a different minimum energy for every capacitance in his measurements, as shown in Fig 9 of Ref 35,p 25 (not reproduced here)

In order to develop testing procedures that will yield a more meaningful characterization of the sensitivity of primary expls, a testing method that involves the use of the so-called " sensitivity map" was introduced. This map which is a type of response surface is reproduced in Fig 12, Ref 35 and briefly described on p 25. No description is given here since the authors of Ref 35 admit that " obviously one needs a better procedure"

Picatinny Test No. 2. The apparatus consists of a fixed gap electrode which uses steel needles. The discharge circuit is similar to that of the Picatinny Test No 1, using the gap length of 0.005 inch set with a feeler gauge. In nearly very respect, this test is inferior to the test No 1. It has been used for distinguishing primaries from boosters (Ref 35, p 27)

Tests at The Naval Ordnance Laboratory. Two apparatuses have been used at NOL. The first was constructed by Wyatt (Ref 20) and is very similar to the approaching-electrode apparatus used in the ERDE laboratory. This apparatus has apparently not been used for recent tests at NOL, presumably because it is inconvenient and time consuming (Ref 35, p 27)

The second apparatus was constructed by Montesi (Ref 31) to provide a faster testing procedure. It was designed with simplicity and reproductivity in mind to determine relative sensitivity only. A fixed electrode configuration was used for all tests. Additional details are given in Ref 35,pp 27-8

The last section of Ref 35, entitled " A Proposed Research Program for Electrostatic Sensitivity Testing of Primary Explosives" is intended to lead to improved procedures and a better understanding of electrostatic spark initiation

Refs on Electricity, Extraneous and Hazards Associated with It: 1) L.B.Loeb, " Fundamental Processes of Electrical Discharges in Gases", Wiley, N Y(1939) 2) F B. Silsbee, USNatlBureau of Standards Circular **C438**(1942) [Methods for discharging materials: use of neutralizing charges and exposure to very moist air; detd the capacity of the human body as 3 x 10^{-4} μf); detd that surface resistance of an insulator, such as wood may decrease by a factor of 16^6 in going from an atm of 30% relative humidity to one of 80%; such materials acquire a surface layer of moisture which always contains enough dissolved material to provide a fair degree of electrical conductance. However, a few materials (including waxes) fail to acquire such films in humid atmospheres. Some body sweat (which is conductive because it contains NaCl, cholesterol, fatty acids, etc) may be present in such films]
2a) C.F. Wagner & G.D. McCann, ''Induced Voltages on Trsnsmission Lines", AIEE Transactions **61**, 916-29(1942)
3) D.J.Kusler & F,W.Brown," Ignitability of Explosives by Static Electricity'', USBurMines, Tech Note No **23** Nov 1943 4) F.W.Brown, D.J.Kusler & F.C.Gibson, ''Sensitivity of explosives to Initiation by Electrostatic Discharges", USBurMines Report of Investigations **RI 3852**,Jan 1946 and **CA41**, 6721 (1947) 5)J. Fleischer & J.B.Burtle, USP 2421778 (1947), p 5 & CA**41**, 5724 (1947) [Electrostatic charges were detd by means of an electroscope for pure LA (Lead Azide), DLA (dextrinated LA), and PVA-LA (polyvinyl alcohol-LA), before and after tumbling. Before starting the experiment each sample was allowed to stand for 16 hours before its electrostatic charge, prior to tumbling, was measured. Then each sample was subjected to a predetermined number of tumblings (same for each sample) and new charge immediately measured. It was found that in each case the charges were greater than before tumbling. Results of tests are given in Table E8 6) H. Rathsburg & L. Schmitz, Chem Ing Tech **21**, 386-89 (1949) & CA**44**, 1259 (1950) (Measurement of electrostatic charge and ignition sensitivity of various primary and initiating explosives) 7) R.F. Wilkerson ''Research and Development in the Field

Table E8

Electrostatic Charges of LA's

Explosive	Before Tumbling (Joules)	After Tumbling (Joules)
Pure LA	1.8	5.8
DLA	2.0	9.6
PVA–LA	0.2	0.7

of Electric Primers",Armament Research Establishment,GtBrit Rept **26215** (Nov 1949) 8) Z.V. Harvalik, "Explosive Initiators, Electric",A Literature Survey, NAVORD Rept **1487** (1950)
9) P.S.H.Henry, Brit JAppl Phys, Suppl 2, Vol 4, pp 578–583(1953) (One of the methods recommended for discharging electrostatic charges of materials was exposure to radioactivity) 10)D.Bulgin Ibid, S87–S91(1953) (It has been stated that cotton and other approved fabrics should be exposed to the humid atmosphere of the workroom dealing with expls for at least 10 mins to attain equilibrium or they may give rise to dangerous electrostatic charges. Furthermore frictional contact with ungrounded seats may generate charges of well over 15000 volts on the body)
11) A. Quinton, Ibid, S92–S94(1953) (One of the recommendations for discharging electrostatic charges was exposure to radioactivity) 12) A.G. Peace, Ibid, S94–S97(1953)(Many expls may be dried at 50° and sieved thru 100 mesh with little or no generation of electric charges. However, many other expls notable RDX and Tetryl, develop high degree electrostatic charges and adhere to spoons handling them and clog the sieves) 13)F.W. Brown, D.J.Kusler & F.C.Gibson, "Sensitivity of Explosives to Initiation by Electrostatic Discharge" USBurMines Report **5002**, Sept. 1953
13a) J.N.Ayres, "A Study of the Effects of Static Electricity on Low Input Energy Electric Initiators of the Carbon Bridge Type ",Proc Symposium on Electric Detonators, The Franklin Institute, AD-066001(Sept 1954) 14) G.H. Damon & R.W.Van Dolah "Electrostatic Spark Test on 9 Samples of Explosives", US BurMines Rept **3426** (Dec 1954) Project 2301–185 15) R.E. Braid, R.C. Langille & Anna M.Armstrong, "Studies in Electrostatics", Canad J Techn **34** (2), 45–52(1956) & CA **50**, 7461(1956) (Prevention of electrostatic charges on powders and prevention of explns during hand operations on detonators) 15a) R.C. Smart Explosivstoffe **1956**,67(Fire and expln hazards due to static electricity)
16) P.W.J Moore, J.F.Sumner & R.M.H. Wyatt, "The Electrostatic Spark Sensitiveness of Initiators: Part I–Introduction and Study of Spark Characteristics", ERDE(Explosives Research and Development Establishment) Report **4/T/56**, Waltham Abbey, Essex, England (Jan 1956)
17) Ibid, "Part II–Ignition by Contact and Gaseous Electrical Discharges", ERDE Rept **5/R/56**(March 1956)
18) P.W.J. Moore, Ibid, "Part III–Modification of the Test to Measure the Electrostatic Hazard Under Normal Handling Conditions", ERDE Rept **22/R/56** (May 1956) 19) R.M.H. Wyatt et al, "The Ignition of Primary Explosives by Electric Charges", ProcRoySoc **246A**, No 1245 (July 1958) 20) R.M.H.Wyatt, "The Electrostatic Spark Sensitivity of Bulk Explosives and Metal/Oxidant Mixtures"; NAVORD Rept **6632** (June 1959)
21) E.Crane, Ch. Smith & A. Bullfinch, "A Statistical Evaluation of the Pyrotechnics Electrostatic Sensitivity Tester", PicArsnTech Note **No 26** (July 1959)
21a) J.D. Cobine, Gaseous Conductors", Dover Publications, NY (1959),p 183
22) D.B. Sciafe, "The Electrostatic Spark Sensitiveness of Initiators, Part IV–Initiation of Explosion by Spark Radiation", ERDE Rept **9/R/59** (August 1959) 23) R.M.H. Wyatt, Ibid, Part V–Further Study of Ignition with Metallic and Antistatic Rubber Electrodes", ERDE Rept **24/R/59** (Sept 1959)
23a) A.Bleibtreu, Explosivstoffe **1960**, 27–31 (Danger of electrical ignition during transportation of explosives in

high mountains) 24) D.G.Kirk, "A Survey of Methods and Equipment for Electrostatic Sensitivity Testing of Solids", Frankford Arsenal, Preliminary Report (1960) 25) H.J.Jackson, "A Study of the Electrical Characteristics of Some Explosives and Explosive Mixtures", PicArsnTech Memorandum **1288** (Oct 1963) 25a) R.F. Ficci, Electrical Interference", Hayden Book Co, NY(1963) 26) A.J. Clear, "Standard Laboratory Procedures for Determining Sensitivity, Brisance and Stability of Explosives", PicArsnTechRept **3278**(1965), pp 14–15 27) DuPont & Co, "Blasters' Handbook(1966), 169–74: "Hazards of Extraneous Electricity" which includes on pp 170–71, "Static Electricity". The Pamphlet of the Institute of Makers of Explosives, entitled "Radio Frequency Energy – A Potential Hazard in the Use and Transportation of Electric Blasting Caps" is obtainable thru the DuPont Co 28) H.A. Hannah & J.R. Polson, "Investigation of Static Electrical Phenomena in Lead Azide Handling", Mason and Hangar-Silas Mason Co, Inc, TechRept **98-A**, Burlington, Iowa (1967) 29) E.L. Litchfield et al, "Minimum Ignition Energy and Quenching Distance in Gaseous Mixtures", USBurMines-Report of Investigation RI **7009** (August 1967) 30) C.T. Davey, "Hazards to EED's in Shipping and Handling with Emphasis on Lightning, Static, and RF Electricity", Minutes of Ninth Explosive Safety Seminar, Armed Services Explosive Safety Board, 15–17 (August 1967), p 657 30a) P.J. Klass, "Lightning Strike Threat Increases", Avionics **90**(1), 1969 [Abstracted in Expls & Pyrots **2**(6) (June 1969)] 30b) C.T. Davey, The Franklin Inst, "Why Ground", in Expls & Pyrots **2**(7), July 1969 (Grounding entails making an electrical connection to the earth so that electrical charges may be shared, bringing the grounded body to the same potential as the earth, which is equal to zero. When a body of an operator is not grounded and he wears thick rubber soles, he may accumulate a potential as high as 20000 volts. If, when so charged a person approaches an expl material, the stored energy of the body will discharge as a spark. This action will produce enough power in the form of pressure and heat to set off a number of EED's, including some so-called one-amp, one-watt devices. Whenever working with sensitive expl materials (such as primary expls) or devices contg them (such as primers, detonators, EED's, etc), operators should be connected electrically to the work bench, chamber or other surface on which they are standing. In fact, all objects in the vicinity should be grounded to avoid static charge accumulations. It must be noted, however, that grounding, as described above, does not protect the operators working near electrical power line, and there is a definite electrocution risk. A cure for this is to short out vital portions of the body. This may be done by grounding at both wrists with wristlet contacts similar to metal watch bands. These are connected to one another and to ground to avoid passage of current thru the chest area

In case of protection against lightning of expls plants, or places handling expls or devices (like EED's or detonators), well grounded metallic rods are considered to be effective. This problem was discussed in Ref 30) 31) L.J. Montesi, "The Development of a Fixed Gap Electrostatic Gap Discharge Apparatus for Characterizing Explosives", ProcsSixthSymp on Electroexplosive Devices, The Franklin Institute, Philadelphia, Pa, July 1969 (Description of work done at NOL) 32) W.E. Perkins, "A Survey of the Methods of Testing the Electrostatic Sensitivity of Solids", FrankfordArsnMemorandum-Rept **M69–29–1** (Dec 1969) 33) R.F. Gentner, "An Electrostatic Sensitivity Test of Composition B", PicArsnTechRept **4119** (Dec 1970) 34) P.V. Satyavratan & Kurti (Indian Detonators Limited, Heyderabad 500018, India), CurrentScience **41**(18) (Sept 10, 1971), pp 663–65. Abstracted in Expls & Pyrots **5**(11), Nov 1972, under the title of "Reduction of Static Electricity in Factory" [Bulk handling and sieving of dry PETN gives rise to accumulation of static charges of the order of few thousand volts. In order to minimize the build-up of such hazardous voltage, ionized air is circulated into the room where PETN is being handled. Ionization of air is achieved by means of suitable radioactive isotopes, selected with the help of BARC(?), Bombay]

35) C. R. Westgate (Johns Hopkins Univ), B.D. Pollock (PicArsn) & M.R. Kirshenbaum (Pic Arsn), "Electrostatic Sensitivity Testing for Explosives", PicArsn Tech Rept 4319, (April 1972), AMCMS Code 552C.12.55901, Dover, NJ, 07801 (Detailed résumé is given in this write-up)

Electric Matches for Use in Pyrotechnics. See Vol 4,p D761–R

Electric Powder. An older Amer Dynamite, which contd 28 to 33% NG. Other ingredients were not listed in Ref
Ref: Daniel (1902), 252

Electric Primer, Comparison with Percussion Primer. See Vol 4, p D794 with Fig 53

Electric and Percussion Primer Combination. See Vol 4, p D795 with Fig 54

Electric Primers (German,Military). Several types were employed during WWII in ammunition for AT (Antitank) guns above 5cm in caliber: also for tank guns of 5 cm and larger, for 7.5 cm StuG (Sturmgewehr), for 8.8 cm Flak 41 & 43 and for all caliber AAC(Antiaircraft) guns larger than 8.8 cm. One of such primers **EIZ C/22** is described, with Fig on p Ger 137-R of Ref 2, which is not reproduced here. This primer consisted of a brass body, a brass primer plug, its plastic insulator, an igniter assembly, a sheet brass igniter retainer, a brass retaining screw, a loose BkPdr chge, a cloth BkPdr disk, and an Al closing disk crimped in position to close the forward end of the primer. The igniter assembly consisted of two thin Al lead-ins placed on each side of a fiber strip and connected to each other by means of a Pt-Ir bridge. One lead-in was in contact with the primer plug, the other with the igniter retainer. The bridge and the fiber assembly were encased with a small quantity of LSt(Lead Styphnate) colored green with NC lacquer and around this was placed loose BkPdr chge

When the firing circuit was closed the current passed from the insulated primer plug, up one of the lead-ins, thru the wire bridge, and down the other lead-in, to the igniter retainer which grounded the current. The passage of the current heated the bridge wire sufficiently to ignite the LSt surrounding it and this, in turn, ignited the BkPdr chge (Ref 2,p Ger 137)

The following two primers were developed during WWII by the Deutsche Waffen- und Munitionsfabriken AG, Lübeck:
A)Bridgeless Type Electric Cap. It consisted of a cylindrical casing (Zn-plated steel)(See Fig E18), contg a primer mixture (presumably Pb dinitroresorcinate and Pb Azide), a pole piece, insulating cap, Pb/Sn foil washer(attached by shellac to an insulating material washer) and a contact ring. A current of 120 to 160 volts was required to fire the primer (Ref 1,pp 75 & 77 and Ref 2,p Ger 138-L

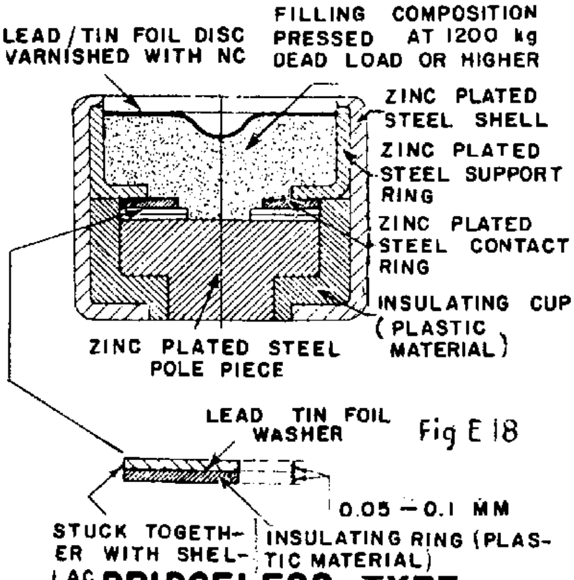


Fig E 18

BRIDGELESS TYPE ELECTRIC CAP

B) *Bridge Type Electric Cap* consisted of a cylindrical casing (Ni-plated brass or Ni-plated steel). See Fig E19, contg the following items: a)Bridge wire ("fuse") (such as of 80/20-Ni/Cr alloy was soldered to two leads consisting of two metal foil strips separated by a millboard, serving as an insulator; b) Fusehead; c) Molded Plastic insulator; d) Brass contact

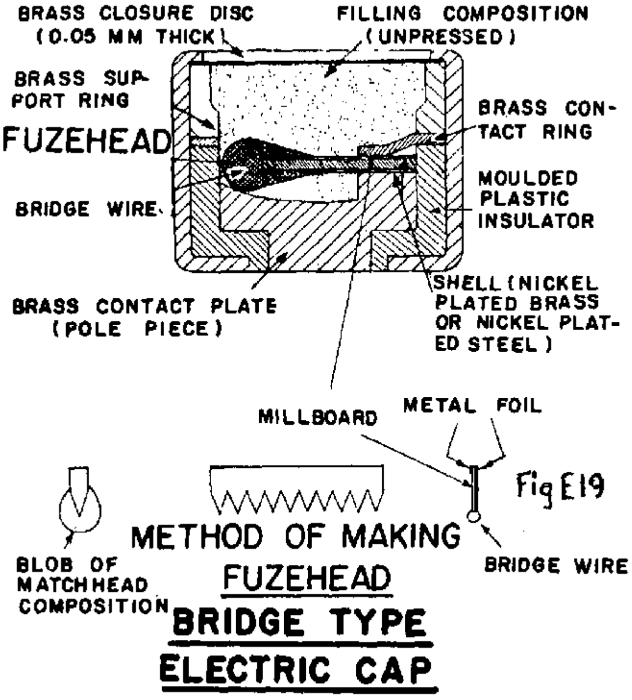


Fig E19

ring; e) Brass support ring; f) Brass contact plate serving as a pole piece; and g) Filling compn consisting of K perchlorate 47, Pb styphnate 23 & Ca silicide loaded loosely around the fusehead (Ref 1, pp 73-6 and Ref 2, p Ger 138)

For prepn of fusehead could be used the method "A6" developed at Troisdorf Fabrik of Dynamit AG, located near Köln. For this the tip of bridge wire was dipped succesively into the following liquid compns:

a) First dip which consisted of dry Pb picrate 90g & Si (20 to 40 microns) 10g, all suspended in 75 ml of a 2% soln of NC in amyl or butyl acetate was applied and then the tip was dried

b) Second dip which consisted of dry Pb picrate 50, dry Pb chromate 35g & Si 15g all suspended in 75ml of 3% solution of NC in amyl or butyl acetate was applied and the tip dried

c) Third dip which was a lacquer consisting of a 15% soln of NC in 75/25-butyl acetate/ethanol to which was added (20% by wt of NC) "Sipalin AOM" (methylcyclohexyl ester of adipic acid) was applied and the tip dried

d) Fourth dip was the same as the 3rd, except that it contd 0.8g of Sudan Brown for each 10 liters of lacquer

This produced a low-tension fusehead (Ref 2, p Ger 53-R)

Note: The "comb" shown in Fig E19 was used at Troisdorf Fabrik for mechanical production of fuseheads. It consisted of a strip of sheet steel from which the outline of a comb was stamped. The two legs of each fusehead were then bonded together with "Mipolan" (mixt of polyvinyl chloride, tricresylphosphate & butyleneglycolphthalate), the tips of the teeth suitably bent and the bridge wire soldered into position. After dipping the bridge wires into fusehead compns. the back of the comb was sheared off (Ref 2, p Ger 53-R

Refs: 1) H. Peploe et al, CIOS Rept 33-20 (1946), pp 73-76 2) PATR 2510 (1958), p Ger 137-38 and Ger 53-R

Electric Primers (Military) (US). There are the following two types of electric primers:

A. *Electric Primers for Igniting Propellants* in cartridge or separate-loading ammunition. They are also known as Primers-Igniters and described in Vol 4, Section 3, Part D, on p D794 with Fig 53, Combination Electric and Percussion Primers such as Mk 15 Mod 2 are on p D795 with Fig 54, Combination Electric and Percussion Primers M75 (T106E1) and Mk 34 Mod 0 are listed on p D795-L but not described. Electric Igniter-Primer M 74 is listed on p D1066, describing analytical procedure for its primary compn

B. *Electric Primers for Use in Fuzes* are described in Vol 4, Section 4, Part E, p D854 to D856:

a) Navy Electric Fuze Primer Mk 112 Mod 0 (p D854-R with Fig 1-43 on p D855)

b) Navy Electric Fuze Primer, Mk 121 (pp D854-R & D855-L with Fig 1-44)

c) Navy Experimental Spray Metal Electric Fuze Primer (pp D855-R & D856 with Fig 1-45)

In the Pamphlet of S. Odierno, "Information Pertaining to Fuzes", Vol IV, published in 1964 by Picatinny Arsenal are listed in Table on p VIA, four Army Electric Fuze Primers (See our Table E9)

Table E9

		Physical Characteristics			Elec Characteristics				Firing Time		Explosive Train Milligrams (Mean)		
					Capacitor Discharge						Priming		
Item No.	Nomenclature	Lead	Dia. Max in	Body Length Max in	Bridge	µf	Volts	Ergs	Max µsec	Spot	DLA	Base	Other
1	XM85	B1	.194	.300	W	16	2.5	500	250K	MLSt	40	–	I1 & D1
2	XM87	B [see Note (a)]	.194	See Note (a)	C	.004	100	200	5K	See Note (a)		–	–
3	XM88	B2	.194	.315	C	.004	100	200	50K	MLSt	40	–	I1 & D1
4	XM89	B [see Note (a)]	.147	See Note (a)	SG	.0016	500	2000	5	CLA	See Note (a)	–	–

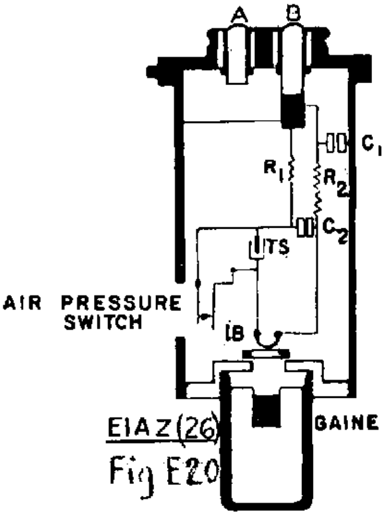
Symbols: Lead – B - Button with lengths B1 .065", B2 .120"
Bridge – W - Wire (2 to 10 ohms), C - Carbon (1K to 10K ohms), SG – spark gap
Explosive Train
MLSt – Milled normal Lead Styphnate mixed with Nitrocellulose lacquer
I1 – 25/75 zirconium/barium chromate
D1 – 72/23/5 barium chromate/zirconium-nickel/potassium perchlorate
CLA – Colloidal Lead Azide mixed with Nitrocellulose lacquer

Note: (a) As the primer was in Research and Development when the Data Sheet was prepared, this portion is not fixed

Addnl Refs: A) Remington Arms Rept AB-51-17(1951), Contr DA-19-059-ORD-15
B) L.C. Long & R.E. Donnard, FA Rept **R-1479A** (1959), "Development of an Improved Electric Primer for Satisfactory Ignition of Single Base, Extruded, Cool Burning Propellant in 20mm Aircraft Ammunition"
C) R.E. Donnard et al, F.A. Rept **R-1479** (1959, "Development of an Electric Primer (FAT-36E7) Compatible with Single Base, Extruded, Cool Burning Propellant"

Electric Primers, Wireless. This class of primers is not equipped with lead wires that are characteristic of other electric detonators but are likewise activated by an electric power source. This type of primer is exemplified by the M–52 A3 series which was standard for the M–99 cartridge or the 20mm ammunition. A typical composition for these primers is as follows:
Pb styphnate 40.0, TNT 10.0, $Ba(NO_3)_2$ 37.2, Ti 11.8, Acetylene black 1.0%, or FA composition 874: *N*-Pb styphnate 40.0, $Ba(NO_3)_2$44.2, Ca Si_2 13.0, Trinitroresorcinal (TNR) 1.0, acetylene black 0.8 & gum arabic 1.0%
Ref: Remington Arms Report 4 AB-51-17(1951) Contr DA-19-059-ORD-15

Electric Proximity Fuze (Ger). The type of fuze, described in Ref 1 and designated as ElAZ(26) was cylindrical in shape and contd (see Fig E20) the charging plungers AB (surrounded by insulating material) a charging condenser C_1, a firing condenser C_2, resistances R_1 & R_2, an igniter bridge IB, a trembler switch TS, and an air pressure switch, The latter switch consisted of fixed and movable plates. The switch was placed just inside of an opening in the fuze case and was aligned with the air tube leading from the nose of the bomb **C250Flam.** The base of the fuze was threaded to receive the gaine, which housed the electric primer (which contd a match compn and BkPdr), the detonator (which contd LA/LSt mixture over



EIAZ(26)

Fig E20

PETN & PETN/Wax and the booster (PA). Before the bomb was dropped from a plane, the current from the plane batteries was passed thru B (plunger A was a dummy) into C. and, at the release of the bomb, the current leaked slowly thru R_2 to C_2 where it accumulated. As the bomb approached its target the pressure of air built up in the tube leading to the air pressure switch, pushed the movable plate of this switch towards the fixed plate, thus closing the circuit thru IB and firing the gaine and eventually the main charge of the bomb. If the pressure of air should fail to operate the fuze the trembler switch TS was supposed to act on impact of the bomb *Note:* Accdg to G.E. Rogers, formerly of Pic Arsn, this type of fuze could be initiated by the air burst produced by other bombs exploding in vicinity and this would be undesirable if the bomb were not yet close to its target (Ref 1 & Ref 3)

Other German proximity types included, electronic fuze, produced by Telefunken Co and also acoustic and infra-red fuzes. They are described in Ref 2

Refs: 1) **TM E9-1983** (1942), File N2322.6
2) **TM9-1985-2** (1953), 216-17, 229 & 232
3) PATR **2510** (1958), pp Ger 151 & 152

Electric Purification of Nitrocellulose. K. Murakoshi, JapanP 100065(1953) & CA **28**, 2538 (1934) proposed to purify NC by passing thru its aqueous suspension an alternating current (and sometimes direct current in addition) at 50 cycles, 1000-2000 volts and 1 ampere/cm^2

Electric Squibs (Also known as *Electric Igniters*, or *Electric Actuators*). These devices are intended to fire deflagrating expls, such as a pellet or blasting Bk Pdr (Black Powder), NC (Nitrocellulose), or loose smokeless propellant. It operates on the same principle as an Electric Blasting Cap, except that it does not detonate but merely shoots out a small flame sufficiently hot to ignite the deflagrating material. By means of this device several charges may be fired simultaneously and any charge can be ignited in the center instead of at the end in order to obtain greater efficiency (Ref 2, p 37)

Some of the electric squibs are similar in construction to Single Component Blasting Caps (Such as described in Vol 2 of Encycl, p B185-R, except that the capsules may be made from paper, wood or plastic, instead of metal. There are also two-component electric squibs with shells made of metal (aluminum), such as manufd by the Du Pont Co and described in their Blasters' Hdb (Ref 7, pp 94-5). Their electric squibs designed to ignite a blasting or pellet BkPdr comprise an Al shell 7/8 inch long, with a chge of a deflagrating mixture in the bottom and with an electric firing element crimped into the other end. This firing element consists of plastic-insulated leg wires held together by a rubber plug, and a bridge wire across the end of leg wires. When electric current is applied to the leg wires, the ignition mixture fired by heated bridge wire, flashes and ignites the deflagrating chge which ruptures the Al shell. Under confinement, intense flame issues from the ruptured shell into the chge of blasting or pellet BkPdr to be ignited. These electric squibs will ignite pellet powder which is too wet for firing with safety fuze. In addition, they permit tight stemming of the borehole, ignition of the powder chge at any desired point, firing of a number of shots at the same time, and control of the time of firing the chge. Further, in contrast to safety fuse, they produce no smoke. Electric squibs and electric blasting caps are considered the safest and most effective means of firing BkPdr and their use was strongly recommended by DuPont Co

All-purpose Mk 1 Electric Squib

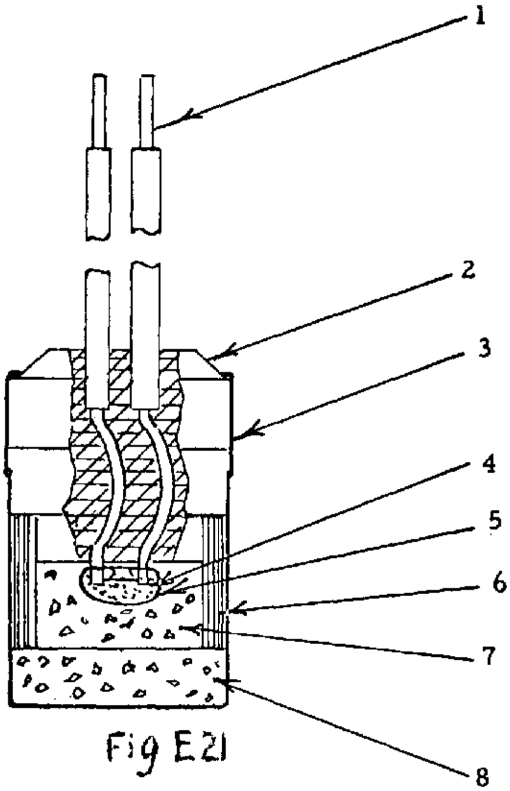


Fig E21

There are also electric squibs which are used for military purpose. One such device is the "US Navy All-Purpose Mk1 Electric Squib", shown as Fig E21. It comprises a gilding metal or Al case (3), contg Pt-Ir bridge wire (4), surrounded by the ignition bead (5), which may be LSt(Lead Styphnate), LA(Lead Azide) or LMNR(Lead Mononitroresorcinate) + $KClO_3$ (95/5). The bridge is connected to two leg wires (1), which pass thru a phenolic plug (2). The base charge is subdivided into booster chge (7) and main chge (8). Each of them consists of 45mg A5 BkPdr (Black Powder). This expl may be replaced by a mixture of Bk Pdr with Ball Propellant, or by other smokeless Propellant

In Fig E22 is shown a "Special Purpose M40 Pressure Squib" manufd by McCormic Selph Associates, sometimes used to initiate action in a gas generator. The squib of Fig E22 consists of the following parts:

1) **Shunt:** Positive spring clip type
2) Contact Pin: 0.125-inch-diameter steel with solder lug
3) Ceramic: Pressure stressed ceramic per Mc Cormick Selph Specification NS-161. Will withstand 25,000 psi applied in 10 msec. Insulation resistance is above 10 000 megohms
4) Case: 0.5-inch hex steel with .375-24 MF 24thread
5) Explosive Load**: 150 mg FFFG black powder
6) Bridge Wire*: Double safety welded nichrome Contact pin to case resistance, 1.07 ± 0.10 ohm
7) Closure: 0.002 crimped and sealed aluminum disc
8) Finish: Flow–melted electro tin plate

*Special bridge wires and squib resistances are available

** Alternate loads available for metal oxidant igniters

(Refs 1,3,4 and 5)

In the pamphlet of Odierno (Ref 6, p XVA, Table) are listed three US Military Electric Squibs. The Table is reproduced here as Table E10

M40 Pressure Squib

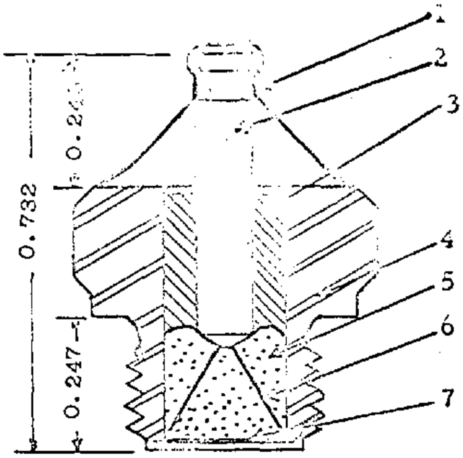


Fig E22

Table E10

Squib No	Diameter (In.)	Length (In.)	Type Bridge	Type Lead	Charge Weight	All Fire Current
M1A1	0.270	0.370	Wire	Wire	65 Mgs Note 1	0.545 Amp
M2	0.270	0.370	Wire	Wire	65 Mgs. Note 2	0.545 Amp
XM3	0.287	0.365	Wire	Wire	90 Mgs Note 3	5.0 Amps Note 4

NOTES:

1) 20% Diazodinitrophenol (DAzDNPh)
60% Potassium Chlorate, Grade I, Class C
15% Charcoal, Class D
3% Nitrostarch (NS)

2) 32% Lead Thiocynate
40% Potassium Chlorate Grade I
18% Charcoal, Class D
10% Egyptian Lacquer No 567X

3) 33% Granular Zirconium Type 2
8.9% Aluminum Atomized
3.0% Aluminum Flake
50.0% Barium Nitrate
5.0% Potassium Perchlorate Granulation 2
0.1% Binder Type 2

4) A Bead or Spot consisting of:
45% Zirconium Metal Powder Type 1
47% Potassium Perchlorate Granulation 1
1% Graphite Powder
7% Binder Type 1

Refs: 1) F.Short, Raymond Engineering Laboratory Rept No 271(1950), "Electrical Squibs BS-14b, BS–19b, Dimple Type Middletown, Conn 2) J.J. Berliner & Staff, "Explosives", Pamphlet (1953), p 37 3) Robert Betts, Ordnance Missile Laboratory Rept 4, No 3J14N1, "Compilation of Data on Army, Navy and Commercial Standard Electric Squibs", Redstone Arsenal, Huntsville, Alabama (1956) 4) C.E.Baugh, NAVORD Rept 6628 (1959), "Evaluation of Explosive Switches MK66 Mod O, and Mk 67 Mod O, White Oak, Maryland 5) J.C.Kenyon, Ordnance Mission Data Rept 4, DA Project 512-15-009 (1962), "No-Fire Level Test of TADM Electro-Explosive Devices, White Sands Missile Range, New Mexico 6) S. Odierno, "Information Pertaining to Fuzes", Pamphlet, published by Picatinny Arsenal in 1964, Vol 4, pp XVA & XVB 7) DuPont Blasters' Hdb (1966), 94–5 (Electric Squibs)

Electric Time Fuze (ETF). Its definition is given in Vol 4, p D881-L

Electric Torpedoes. These torpedoes are battery-powered and one of such torpedoes, Mark 18, was used during WWII. Much of the early development of electric torpedoes was hindered by lack of a satisfactory power source, but it seems that this difficulty has been overcome by now. Although this type of torpedo does not equal the power and performance of the steam-driven torpedo, its advantage is that it does not give telltale wake of bubbles, which is characteristic of steam-driven torpedoes. For this reason 65% of torpedoes fired by US submarines during the final 6 months of WWII were electrically-driven and they were responsible for the sinking of 300 Japanese vessels, well over one million tons

At the end of WWII, the Mark20 was ready for production. This torpedo was powered by a strong battery driving a 180 horsepower motor, capable of propelling the torpedo at 40 knots per hour to a distance of 8000 yards. Its warhead contd 1000 lbs of TNT or of Torpex
Refs: 1) Anon, Army Ordn 30, 100-102 (Jan-Feb 1946) 2) B. Rowland & W. Boyd, "US Navy Bureau of Ordnance in WWII", US-GovtPrintingOffice, Wash, DC (1953), pp110-16

Electroanalysis. See under Electrochemistry

Electroanalytical Analysis. See under Electrochemistry

Electrochemical Nitrations. A method developed in 1936 in Sweden by Öhman for prepn of nitric acid esters has been described in several patents. The method consists in anodic oxidation (using a bright platinum anode) in presence of nitric acid, or its salts (such as Ca nitrate). The compds to be nitrated are unsaturated hydrocarbons (such as ethylene, propylene, butylene, etc), which can be dissolved in nonaqueous solvents (such as acetone). The OH concn is maintained low during the reaction by adding either concd nitric acid or glacial acetic acid. Water should be absent to prevent the formation of various by-products

Refs: 1) V. Öhman, FrP 800944(1936) & CA **31**, 45(1937) 2) Ibid, ZElektrochem **42**, 862-72(1936) & CA **31**, 2106(1937) 3) Ibid, SwedP 87886(1936) & CA **31**, 2528(1937) 4) Ibid, SvenskKemiskTid **50**, 84-98(1938) & CA **32**, 6160(1938) 5) Ibid, USP 2130813 (1938) & CA **32**, 8959(1938) 6) Ibid, GerP 692415(1940) & CA **35**, 4292(1941) 7) N.M. Winslow & G.W. Heise, USP 2427433(1947) & CA **42**, 46(1948) (Org chem reactions by electrolytic processes) 8) G. Bionda & M. Civera, AnnChim **43**, 11-14(1953) & CA **47**, 5821(1953) (Electrolytic substitutions in aromatic nuclei-nitration of benzene) 9) P.V. Öhman, SwedP 150688(1955) & CA **50**, 93(1956) (Nitric acid esters of olefins by anodic esterification)

Electrochemistry, Electrochemical Engineering, Electroanalysis and Electrodecantation

Electrochemistry, according to the definition given in Perry's, is the science which treats of the chemical changes produced by an electric current and of the production of electricity from the energy of chemical reactions. Theoretically, the two branches are of equal importance. Industrially, however, the chemical and physical changes produced by the use of an electric current are by far the most important (Ref 19)

Electrochemical Engineering is primarily a branch of chemical engineering to which portions and viewpoints of electrical engineering and metallurgy have been joined

Electroanalysis is the application of electrochemistry to chemical analysis of various substances. It may be divided into electrodeposition(coulometry), internal electrolysis and electrosolution (Ref 16)

Electrodeposition depends on the power of the electric current to precipitate an element or its compound. The equivalence of the amount of an element precipitated with the amount of electricity expended, known as Faraday's Law, provides another means for the quantitative estimation of an element, and the method is known as **coulometry**

Note: Electrodeposition of metals has found practical application in electroplating

The term **internal electrolysis** was proposed by H.J.S. Sand, Analyst **55**, 309-12(1930) & CA **24**, 3726(1930) to apply to analysis of an alloy in which the metal to be determined is deposited on a Pt electrode without the application of any outside electrical current. Instead, an anode of the baser metal in the alloy is placed in contact with the solution containing its ions in a compartment separated by a parchment membrane from the solution to be examined

It should be noted that previous to this, Hollard, Bull(France) [3], **29**, 116(1903) proposed a similar method, but used Zn in a solution of a Mg salt as the anode. This method of analysis has been used in semimicro and micro-analysis. Several modifications of it have been described, some of them not employing the Pt electrode

Electric current has also been used as a means of dissolving samples, as well as for precipitating the constituents. This is known as **electrosolution** method. An important application of this principle has been for **electrographic analysis** (qv)

Of these three branches, **electrodeposition** is the most important, especially since the introduction (in 1906) of the controlled potential method. H.J.S. Sand was the pioneer in the development of this method

The science of electrochemistry was originated in the first half of the 19th century by Michael Faraday, who defined the following

rules, now known as Faraday laws:
1) The weight of an ion deposited electrically is proportional to the strength of the current passing thru the solution
2) Each 96489 coulombs (1 farad) liberates one gram equivalent of any ion during electrolysis

Electrodecantation is a stratification phenomenon that may take place when colloidal dispersions are subjected to an electric field between vertical membranes permeable to ions but not to colloids. This phenomenon was first observed in 1923 by W. Pauli of Austria (Ref 21)

Refs: 1) V. Engelhardt, Edit, "Handbuch der technischen Elektrochemie", AkadVerlagsges, Leipzig, 3 vols (1931-1934) (Reproduced during WWII by Edwards Bros, Ann Arbor, Mich) 2) M. Dole, "Principals of Experimental Theoretical Electrochemistry", McGraw-Hill, NY (1935) 3) R. Müller, "Allgemeine und technische Elektrochemie nicht metallischer Stoffe", Springer, Wien (1937) (Reproduced by Edwards Bros, Ann Arbor, Mich) 4) F. Fichter, "Organisch Elektrochemie", Steinkopf, Dresden (1942)(From the book "Die Chemische Reaktionen", vol 6) (Reproduced by Edwards Bros) 5) S. Glasstone, "An Introduction to Electrochemistry", VanNostrand, NY (1942) 6) J. Reilly & W.N. Rae, "Physico-chemical Methods", VanNostrand, NY, Vol **2** (1943), pp 341-55 (Electrochemistry") 7) H.J. Creighton & W.E. Koehler, "Principles and Applications of Electrochemistry", J. Wiley, NY (1943) 8) H.S. Harned & B.B. Owen, "The Physical Chemistry of Electrolytic Solutions", Reinhold, NY (1943) 9) J.J. Lingane, IEC (AnalEd) **16**, 147-52 (1944); **17**, 332-33 and 640-42(1945) 10) V. Tamburini, "Analisi quantitativa per electrolisi", Tipogr G. Frailoi, Alpino, Italy (1946) 11) Fischer-Schleicher, "Elektroanalytische Schnellmethoden", F. Enke, Stuttgart (1947) (3rd edition revised by A. Schleicher) 12) H. Diel, "Electrochemical Analysis with Graded Cathode Potential Control", C.F. Smith Chemical Co, Columbus, Ohio (1948) 13) F.T. Rabbitts, AnalChem **20**, 181-82(1948) (Mercury cathode cell for rapid electrolysis) 14) A.J. Lindsey, Analyst **73**, 67-73(1948) (Review of electrolytic methods of microchemical analysis) (55 refs) 15) A. Weissberger, Edit, "Technique of Organic Chemistry", Interscience, NY, vol 2 (1948), pp 143-202: S. Swan, Jr, "Electrolytic Reactions" 16) S.E.Q Ashley, AnalChem **21**, 70-75(1949) (Electroanalysis with 73 refs) 17) W. Bloom & G.B. Hogaboom, "Principles of Electroplating and Electroforming", McGraw-Hill, NY (1949) 18) C.L. Martell, "Industrial Electrochemistry", McGraw-Hill, NY (1950) 19) J.H. Perry, Edit, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950), pp 1771-1826: C.L. Mantell, "Electrochemistry" (with numerous refs) 20) H.S. Harned & B.B. Owen, "Physical Chemistry of Electrolytic Solutions", Reinhold, NY (1950) 21) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **5**(1950), pp 487-95: S. Ashley, "Electroanalysis (32 refs); pp 495-549: G.C. Akerlof, "Electrochemistry" (123 refs); and pp 549-551: P. Stamberger, "Electrodecantation" (10 refs) 22) G. Kortüm & J.O'M. Bockris, "Textbook of Electrochemistry", Elsevier, Amsterdam, Holland, Vol **1**(1951) and Vol **2** (1952) 23) B.E. Conway, "Electrochemical Data", Elsevier Press, Houston, Texas (1952) 24) E.C. Potter, "Electrochemistry: Principles and Applications", Macmillan, NY (1956) 25) G.W.C. Millner, "The Principles and Applications of Polarography and Other Electroanalytical Processes", Longmans, Green & Co, New York (1957) 26) K & O, 2nd edit **7** (1965), 726-84 (Electroanalytical Methods by D.J. Gross & R.W. Murray); 784-841 (Electrochemistry by G.C. Akerlof); 841-46 (Electrodecantation by P. Stamberger)

Electrode. The appliance by which an electric current passes in or out of a cell or apparatus. It may vary from a simple wire to complex devices, such as hydrogen-, calomel-, capillary-, dropping- (of Heyrovsky), glass-, etc electrodes. It may be the container of the cell itself, such as crucible, vacuum tube or valve

Ref: Hackh's Dict (1944), 297-R & 298-L

Electrodeposition. See under Electrochemistry

Electrodynamics. A branch of practical physics dealing with moving charges, such as electric current
Ref: Hackh'sDict (1944), 298-L

Electrodynamometer. An instrument for measuring the intensity of faradic and alternating currents
Ref: Hackh'sDict (1944), 298-L

Electroendosmosis. The production of endosmosis(qv) by an electric current (Compare with Electro-osmosis)
Refs: 1) Hackh'sDict (1944), 298-L 2) L. Page & N.I. Adams, "Electrodynamics", VanNostrand, NY (1945)

ELECTRO-EXPLOSIVE DEVICES (EED's). They may be defined as devices consisting essentially of a charge of explosive in contact with an "electro-explosive transducer"(qv)

Murphy & Menichelli of Jet Propulsion Laboratory, Pasadena, Calif stated in introduction to their paper published in Ordnance (See Ref) that "Electroexplosive devices storing and delivering large amounts of energy reliably and rapidly have many applications in defense technology and now can be checked accurately and nondestructively". This is followed by a cross section and a brief description of a typical modern device that presently is being used in aeronautical and space applications. Basically (See Fig E23) it is composed of a body, a header, wires (A) by which electrical current enters, a bridgewire (B) and an explosive or pyrotechnic charge (C). The body can be threaded at one end for insertion into a pyromechanical device and have a standard electrical connector at the opposite end. An electrically insulated header is glass sealed into the body with two or more pins extending into the connector shell. Inside the device, a fine-[typically 0.051mm (0.002-in)] diameter, high-resistivity wire is welded flush against the header to the pins. An explosive or pyrotechnic compn is loaded into the header and pressed at approx 6895N/m^2 (10000 psi) against the bridgewire. The unit is completed by hermetically welding a metallic end seal onto the body

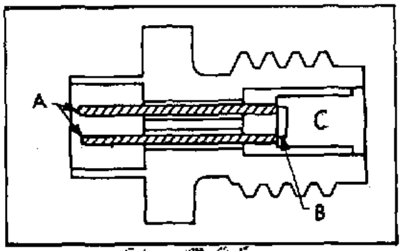


Fig E23

The EED is installed into a simple piston/cylinder-arranged mechanical device. When sufficient electrical energy is supplied thru the pins to the bridgewire, the wire becomes very hot and ignites the expl or pyrot chge. This is followed by rapid generation (usually in less than 1 to 2 milliseconds) of high-temp and high-pressure gas

The fact that these devices can store and deliver large amounts of energy very efficiently and quickly makes them prime candidates for use in spacecraft applications
Ref: A.J. Murphy, Jr & V.J. Menichelli, Ordn **57**, 236–37, Nov-Dec 1972
Addnl Refs: A) T. Warshall, "Electroexplosive Device", PicArsnTechnMemo **1509**(1964)
B) Collective, "Electroexplosive Devices", Proceedings of the Fifth Symposium on Electroexplosive Devices, held June 13 & 14, 1967 at the Franklin Institute, Philadelphia, Pa, 19103, typewritten 900pp, $10; Reviewed in Expls & Pyrots 1(3)(1968)
C) Pelmec Division of Quantic Industry, Inc, 999 Commercial St, San Carlos, Calif, 94070; Quoted from Expls & Pyrots **2**(11)(1969) (A new family of initiators has a microcircuit bridge, is at least one-amp, one-watt no fire, is hermetically sealed and heat sterilizable to 500°F. Pressure cartridges and gas generators are also available. Data sheets include description, drawing, design data and environmental specs met D) Collective, "Electroexplosive Devices", Proceedings of the Sixth Symposium on Electroexplosive Devices, held in San Francisco, Calif, July 8–10, 1969; 50 papers, 850pp, paper bound, $25 per copy. Can be obtd from Mr Gunther Cohn, The Franklin Institute Research Laboratories, Philadelphia, Pa, 19103; Listed in Expls & Pyrots **2**(8) (1969)

E) Anon, "Electroexplosives Handbook", published by Holex, Inc, 2751 San Juan Rd, Hollister, Calif, 95023, listed in Expls & Pyrots **2**(8)(1969) (A pocket size, 33pp, booklet contg EED technology terms, statistical terms, useful design formulas for expl components, notes on the packaging and shipping, safety notes on handling expl cartridges and bibliography) F) Collective, "Electroexplosive Devices", Proceedings of the Seventh Symposium on Explosives and Pyrotechnics, held at The Franklin Institute, Philadelphia, Pa on Sept 8–9, 1971. Announced in Expls & Pyrots **4**(8)(1971) (Available from NTIS AD 742150) G) R.K. Warner & D.L. Overman, "Explosive Train Technology for Electronic Fuzes", Harry Diamond Laboratories **HDL-PR-71-7**(Nov 1971) H) B.V. Frank, "Initiation Mechanisms of Electroexplosive Devices Due to Radiation Exposure (U)", PATR **4421** (Oct 1972) (SRD) (Not used as a source of info)

I) J. De Ment, USP 364647(1972), "A Cylindrical Array of Exploding Conductors Embedded in a Solid Dielectric for Pumping a Laser" (A rotatable phased array of explodable conductors is enstructured with a transparent cylinder of dielectric, blast-resistant solid, each of the explodable conductors fired in sequence by contact with a high-voltage source upon rotation of the cylindrical ensemble of exploding conductors, to sequentially release brilliant bursts of light including laser pumping light) (See also earlier patents by J. De Ment, USP's 3300734, 3537031, 3546623, 3646471 & BritP 1159144)

Electro-Explosive Devices (EED's); Operational Safety. It is known that EED's are sometimes sensitive to extraneous electrical energy and can function prematurely. Some documented accidental initiations are as follows:

a) In 1949 DuPont Co reported a blasting accident due to radio transmission

b) Naval Ord Test Station had a premature of a propelling motor caused by a mobile radio transmitter nearby

c) In 1955 there was a premature aboard the USS Ashland, presumably caused by a telemetering transmitter of the missile

d) A premature of 2.25 inch SCAR while it was being loaded on an aircraft on board the USS Kearsage, was believed to be caused by a transmitter

Listed below are some of the energy sources which are capable of unintentionally initiating EED's: electrically connected circuitry (induction), electromagnetic radiation, electrostatic charges, mechanical, thermal and chemical action

In order to prevent unintentional initiation due to the above mentioned causes, the following precautions should be taken:

a) For protection against electrically connected circuitry, the National Electrical Code should be observed in all equipment and wiring, especially with respect to grounding

b) For protection against electromagnetic radiation, complete shielding is required. No cracks or even the smallest apertures are allowed. When shielding is impractical, exposure to RF (radio frequency) fields should be kept at a minimum. Leads which act as a good antenna should be twisted together and these should not be a rabbit-ear configuration

c) For protection against electrostatic charges the use of grounded conductive materials is recommended. Clothing should be of cotton or linen, while wool, synthetic fibers, silk and rubber should be avoided. Utilization of static electricity indicators and grounded wrist bracelets should be considered

d) Mechanical energy, such as is supplied by a sharp blow, crushing, penetration by a sharp object, or a shock wave of a detonation can be hazardous and should be avoided

e) Thermal energy (heat), as provided by an open flame, oven or friction can be hazardous and should be avoided

f) As chemical energy is usually developing heat, it can be hazardous and EED's should be protected against it

Refs: 1) R.I. Gray, "Hazards to Electrically Initiated Explosives in Weapon Systems", Ministry of Supply Rept DGGW **58/6**(1958) (Conf)(Not used by us) 2) Anon, "Safety Principles for Operations Involving Electroexplosive Devices", NavOrdRept OD 10773(1959) 3) R.E. Grove, Naval Weapons Laboratory (NAVWPNLab) **TM**

No W-20/60(1960) HERO Testing; Techniques and Procedures, Dahlgren, Virginia. For testing and instrumentation used in the determination of the possible hazards of EED's) 4) R.N. Skeeters, "Design Techniques to Reduce the Hazard of Inadvertent Firing of Electroexplosive Devices by Electromagnetic Energy", NOTS TP **2629**(1961), China Lake, Calif 5) R.E. Betts, "Safety Consideration with Electroexplosive Devices", USArmy-MissileCommand Rept No **RR-TR-62-10**(1962), Redstone Arsenal, Alabama 6) The Franklin Institute Rept **FM-131977**(1963), "Operation and Maintenance Manual for the Franklin Institute Laboratories Initiator Test Set Model 3". This equipment measures: 1) bridge resistance, 2) voltage stored on a given capacitor and 3) functioning times, and is known as The Franklin Institute Laboratories Initiator Test Set, FILITS, Model 13

7) HERO Newsletter, "A Quarterly Review on Hazards of Electromagnetic Radiation to Ordnance", USNavalWeaponsLaboratory (Nov 1967), Dahlgren, Virginia

8) R.K. Warner, "Methods of Achieving Safe Stationary Explosive trains'', Harry Diamond Laboratories **HDL-TR-1481** (Feb 1970

Electro-Explosive Devices (EED's); Pulse Firing of. The energy required to fire an EED is an important characterization parameter. This mode of firing corresponds to delivering the energy in a time that is short compared with the thermal time constant in order that heat diffusion and losses to the expl environment surrounding the hot bridgewire are at a minimum. Practical firing systems, in most cases, operate in an adiabatic manner by using the discharge of the energy stored in a capacitor. An accurate measurement of this energy provides a useful sensitivity parameter that can then be used in the design of any impulsive firing system

The characteristics of 25 EED's to inputs of various pulses (condenser discharge, constant current, constant voltage) are found In Ref 1

See also Refs 2 & 3

Refs: 1) Anon, "Electric Initiator Handbook'', The Franklin Institute, Philadelphia, Pa, 3rd edit (Nov 1960) (AD 319980); First Suppl (April 1962) (AD 328852) 2) L.A. Rosenthal & V.J. Menichelli, "Half-Sine Wave Pulse Firing of Electroexplosive Devices", Jet Propulsion Laboratory Report **32-1534**, July 1971. Order as N71 31193 from NTIS, US Dept of Commerce, Springfield, Va, 22151 3) Gunther Cohn, Edit, Expls & Pyrots **5**(2), Feb 1972 (Review of Ref 2)

Electro-Explosive Devices (EED's); Sensitivity Electrostatic. The design and operation of a circuit to study the pin-to-case electrostatic sensitivity of EED's is described in Ref 1. This circuit provides a high voltage, low energy spark discharge that simulates the energy delivering capability of the human body. The circuit is then utilized to evaluate selected EED's that contain explosive and pyrotechnic flash charges. It is shown that properly formulated pyro chges can withstand repeated electrostatic discharges at 25KV. Exploding bridgewire detonators and No 6 electric blasting caps were also static insensitive at this potential. The role of a 500-ohm series resistance in the output of the circuit is discussed, and it is shown that the omission of the resistance has a drastic effect on output pulse profile but a small effect on the electrostatic sensitivity of EED's

Refs: 1) J.L. Austing & Richard Gortowski, "Circuit for Studying the Electrostatic Sensitivity of Electroexplosive Devices", Explostoffe **19**, No 2, pp 33–43 (Feb 1971)
2) G. Cohn, Edit, Expls & Pyrots **5**(3), March 1972(Review of Ref 1)

Electroexplosive Devices (EED's), Testing of. The output tests of EED's are the same as those for other detonators. See in this Encyclopedia, Vol **4**, pp D1084-L to D1088-L. The common input tests are condenser discharge, voltage sensitivity, and steady-current functioning. See Vol **4**, p D1081-L

A number of non-destructive tests have been proposed, but they are not standardized nor in general use as yet. See following article

Refs: 1) Anon, "Explosive Trains", Engineering Design Handbook, US Army Materiel Command Pamphlet **AMCP 706–179** (1965), 63, 67, 77 & 133 2) Anon, "Initiators, Electric, Design and Evaluation of", Military Standard **MIL-L-23659B** (May 1967) 3) R.K. Warner, "Measuring Output, Safety and Reliability of Explosive Components", Harry Diamond Laboratories **HDL–TM–71–34** (Nov 1971)

Electroexplosive Devices (EED's), Testing of, Non-destructive.

EED's being one-shot items, could not be tested functionally prior to actual use because they would be destroyed. However, devices of this type have for a long time been somewhat successfully qualified and tested on a small-sample statistical basis. That is, the functional reliability of such components, which were destructively ground-tested, was assigned to other identical units from the same manufd lot. The reliability and, hence, the expected failure rates of flight hardware of necessity had to be inferred. Testing or inspection of such critical components prior to actual flight usage typically was restricted to indirect examinations and various non-destructive tests, such as X-rays, leak checks, electrical resistance, measurements, weights, etc

To demonstrate, statistically, low failure rates on EED's requires that enormously large number of destructive tests be performed. And separate tests, in large numbers, are required for each new type of EED appearing on the market. It is evident that the destructive method is expensive and time consuming and for this reason, the need for alternative or, at least complimentary techniques, became recognized

Accdg to Ref 7, p 237, the work was begun at the NOL (Naval Ordnance Laboratory), White Oak, Md in 1959 for the purpose of developing new techniques for nondestructively examining what was recognized as the critical functional mechanism of EED's; namely the bridgewire-to-explosive interface (Ref 1)

Prof Rosenthal began in 1961 (Ref 2) experimentation to take advantage of two well known properties of nearly all EED's. The properties were, first that each of these devices had a known and reproducible firing energy vs time-to-fire relationship and, secondly, that most of the high-resistance bridgewire material used in the fabrication of such devices had a reasonably high temperature coefficient of resistance (TCR). Knowing that the most significant failure mechanisms (ie condition of bridgewire weld and intimacy of contact betw the bridgewire and expl or pyrotech chge) resided in the bridgewire/explosive/header interface, an electrochemical, nondestructive test technique for examining this critical area of EED was developed. The method is also described by Murphy & Menichelli (Ref 7, pp 237–38) and the equation is given defining the relationship between energy, ignition time and firing current

The next paper, in chronological order, was that of Harwood & Stewart (Ref 3) which is not listed in Ref 7. They developed a testing technique which allowed an examination of the thermal transfer characteristics from bridgewire to expl mixes in contact with them. The fundamental equation used in these measurements is:

$$C_P \frac{d\theta}{dt} + \gamma\theta = I^2 R_0 (1 + \alpha\theta)$$

where: C_p = Heat capacity of the system, watt sec/°C

θ = Temperature rise above ambient, °C

t = Time, secs

γ = Heat loss factor, watts/°C

I = Current in the form of step function, amperes

α = Temperature coefficient of resistance, ohm/ohm/°C

In the paper of Harwood & Stewart (Ref 3) are discussed methods for arriving at values for γ and for determining the thermal time constant. It is concluded that establishment of proper ranges of γ is empirical and requires accumulation of much more data

Davey of The Franklin Institute (Ref 4) who reviewed the paper, stated that the work reported there is the direct result of much earlier works at PicArsn, Naval Ordnance Lab, and The Franklin Institute, and he lists seven documents of years 1954 to 1963, which are available from the Defense Documentation

Center, Cameron Station, Alexandria, Va, 22314

Later work has been conducted at the Jet Propulsion Laboratory (Refs 5, 6 & 7) with a view toward developing techniques that would allow a nondestructive examination of the bridgewire/header/pyrotechnic interface of the EED. It was necessary for this to obtain thermal characteristics of the EED's. To achieve this a specific technique, consisting of introducing a low-level constant-step pulse into the bridgewire and observing the resistance change with time, was developed. By applying approx 0.4-amp, 50-millisec pulses to the bridgewire with 50-millisec cooling periods betw each pulse, a heating curve can be generated and displayed on an oscilloscope

A typical heating curve is shown in Fig 24. The thermal time constant displayed on this curve is the time in millisecs required for the wire to reach approx 63% of its steady-state average temp. Instrumentation techniques allow for detn of the thermal resistance, thermal time constant, thermal capacitance and the cold wire resistance of each EED. Besides quantitative measurements obtainable for each EED, a qualitative signature of each is obtd by photographing the heating curve. Irregularities on these curves have been investigated and the relationship betw these qualitative signals and faults that exist in the EED design or in manufg have been successfully detd. At the present time, most of the critical failure mechanisms within the EED bridgewire systems, has been correlated to heating-curve irregularities

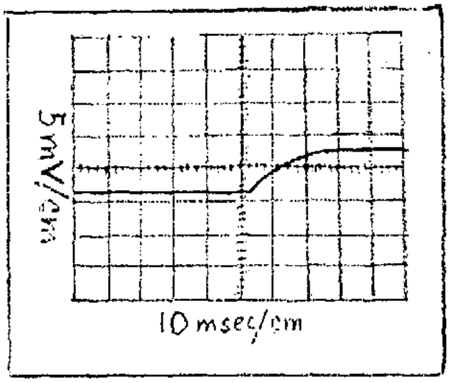


Fig E 24 Oscilloscope Trace of Typical Heating Curve Response from Normal EED

Essentially, each EED design has to be calibrated for fault type. Poor bridgewire welds have been identified and classified. Cases of air gaps existing betw the pyrotechnic material and bridgewire have been identified

Another interesting "fault" – that of the binder solvent existing in inordinately large quantities at the bridgewire surface – has been identified by a transient dip halfway across the heating curve. This is indicative of a cooling process caused by the solvent phase change from liquid to gas

The importance of nondestructive technique developed at Jet Propulsion Laboratory on the quality control of EED's, was proved. In the past, once an EED was completely assembled, little could be accomplished by way of examination of the bridgewire/pyrotechnic surface. It is important to note that failure mode, effect, and criticality analysis (FMECA) conducted on various types of EED's has shown consistently that the most critical area of EED design and manuf lies within this previously uninspectable area

This technique for nondestructively examining a completely assembled EED is now considered operational for EED flight applications at the Jet Propulsion Laboratory (Ref 7, pp 238–39)

Refs: 1) L.A. Rosenthal, "Electrothermal Equation for Electroexplosive Devices", NAVORD Report **6684**, US Naval Ordnance Laboratory, Silver Spring, Md, August 15, 1959. AD 230917 2) L.A. Rosenthal, "Thermal Response of Bridgewires Used in Electroexplosive Devices", Rev of SciInstrs **32**, 1033–36, Sept 1961 3) D.W. Harwood & L.C. Stewart, "Non-Destructive Measurement of the Quality of Electroexplosive Interfaces", Materials Evaluation **26**(12), Dec 1968 4) C.T. Davey of The Franklin Institute, Expls & Pyrots **2**(6), 1969 (Review of Ref 3) 5) L.A. Rosenthal & V.J. Menichelli, "Nondestructive Testing of Insensitive Electroexplosive Devices by Transient Techniques", TechRept **32–1494**, Jet Propulsion Laboratory, Pasadena, Calif, July 1970 6) V.J. Menichelli & L.A. Rosenthal, "Interrelationship of Nondestructive Testing to Fault Determination", Seventh Symposium on Explosives and Pyrotechnics, The Frank-

lin Institute, Philadelphia, Pa, Sept 8–9, 1971 7) A.J. Murphy, Jr & V.J. Menichelli, "New Test for EED's" Ordn **57**, 236–39, Nov-Dec 1972

Electroexplosive Devices (EED's); Thin Film Bridge for. A thin metal film, vacuum deposited on header surface (such as of glass) is used instead of welded bridge wires. There are no broken bridge wires and no poor weld joints with this system. Best results required deposition surface be flat and smooth, better than 100 μ inch. Lead Styphnate was used as first fire; resistance of film was 5–10 ohm, no-fire current 20ma for 2 mins and all-fire energy 1125 ergs from 100 μf capacitor charged to 1.5 volts. Produced in batches of over 1000 (See Fig E25)

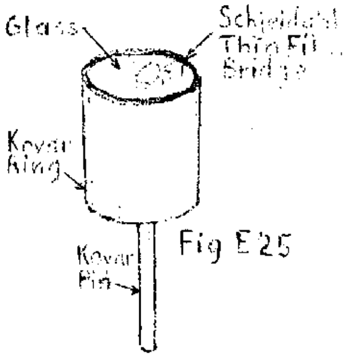


Fig E25

Refs: 1) G.T. Schjeldahl Co, Northfield, Mass, 55057 2) G. Cohn, Edit, Expls & Pyrots **1** (8), Aug 1968 (Review of Ref 1)

Electroexplosive Transducer (EET). It is a device that transforms electrical energy to heat energy. The heat energy is used to initiate an explosive charge and the combination is known as Electroexplosive Device (EED). There are at the present the following types of EET's:

a) Wire bridge type, consists essentially of two wire leads or contacts molded into a plug and a bridgewire which connects the contacts. When an explosive is introduced in contact with bridgewire, the device becomes EED

b) Deposited bridge (carbon or graphite) type is formed between two contacts molded into a plug made of a dielectric by coating the contacts and the surface of the dielectric between them with a conductive material, such as graphite or conductive paint

c) Conductive mixture type is made by adding a conductive material to the explosive; a current passing thru the mixture heats the explosive to detonation point

d) Gap type consists of two electrodes separated by a thin dielectric. A potential difference between the electrodes causes a spark to jump between them and this initiates the explosive. This type is the least common

Ref: Anon, "Safety Principles for Operations Involving Electro-Explosive Devices", NavOrdRept **OD 10777** (1959)

Electroforming. The reproduction of an object or a pattern by electrodeposition (See under Electrochemistry). A mold of the object to be reproduced is made in a soft metal or in wax (by impression). The non-conductor mold surface is made conductive by coating with graphite. Some suitable metal is then deposited electrolytically on the mold surface. This mold is then (in most cases) a negative of the object to be produced. Most extensive use is in the phonograph record industry

Ref: CondChemDict (1961), 434-R

Electroluminescence. The emission of light as a consequence of electrical discharge in gases. Typical examples are neon lights, mercury vapor lamps and lightning

Refs: 1) CondChemDict (1961), 434-R 2) H.K. Hanisch, "Electroluminescence", Pergamon Press, Oxford, New York (1962) (International Series of Monographs on Semiconductors, vol 5)

Electrolysis. Decomposition of a compd (liquid, molten or in solution) by an electric current, which results in the local and spacial separation of the ions of an electrolyte (qv)

and the transfer of their respective charges
Ref: Hackh'sDict(1944), 298-L

Electrolyte. Any substance which dissociates into two or more ions, to a great or small extent, when dissolved in water. Solutions of electrolyte thus conduct electric current and are decomposed by it. Examples of electrolytes are aqueous solns of acids, alkalies, or sslts
Ref: Hackh'sDict (1944), 298-R

Electrolytic Cell. A combination of a liquid or semi-liquid electrolyte (soln of a salt, acid or base) and two solids serving as electrodes. The cell generates an electric current when the electrodes are connected by an external wire. Flashlight batteries (dry cells), storage batteries and fuel cells (qv). When electricity is generated in such a cell chemical changes occur at the electrodes so that either they or both the electrodes and the electrolytes are gradually consumed
Ref: CondChemDict (1961), 434-R

Electromagnet. A piece of soft iron around which is would an insulated wire; so long as an electric current passes thru the wire, the iron has magnetic properties
Ref: Hackh'sDict (1944), 298-R

Electromagnetic Cannon (Elektromagnetische Kanone, in Ger). Prof C. Birkeland of Norway patented in 1906 an invention consiting of a cannon using as power for propelling a projectile an electromagnetic effect resulting from electric current flowing thru wire coils (solenoids) wound around the barrel

Accdg to information published in Russian military journal "Russkii Invalid", such a gun was built and tried on a Norwegian polygon in the summer of 1903. It is not known whether the test was successful or not, but if successful, such gun would have the following advantages over conventional cannons: noiseless shooting, absence of proplnt gases (usually accompanied by muzzle flash or smoke) and only a very small wear on the bore because there would be no overheating caused by burning proplnt gases. Absence of flash and smoke is a property particularly desirable for weapons operating in closed chambers, such as casemates in fortress defenses and in turrets of warships. The disadvantage of such a cannon was the requirement of tremendous amount of electric current for its operation. For example, calcn by an Austrian Captain Spačil showed that for propelling a 400kg shell with 500m/sec initial velocity from a 15cm cannon would require 54300 kilowatt per second which was much more than could be supplied at that time by the Vienna power station which had a total output of only 30000 kilowatt per second(Ref 1)

It seems that interest in electromagnetic cannon died (except possibly in Russia and America) until about 1935 when German Oberleutnant Justrow published an article entitled "Die elektrische Kanone", listedhere as Ref 2. Another paper by the same author was published in 1938 (Ref 3). Dipl-Ing E. Rogge reviewed the above paper (Ref 4) and stated that accdg to Justrow, the barrel of such cannon must be constructed of numerous closely connected in series, ring-shaped electromagnets, which, on account of their excitation winding, can be called electromagnetic coils or spools (Spulen)

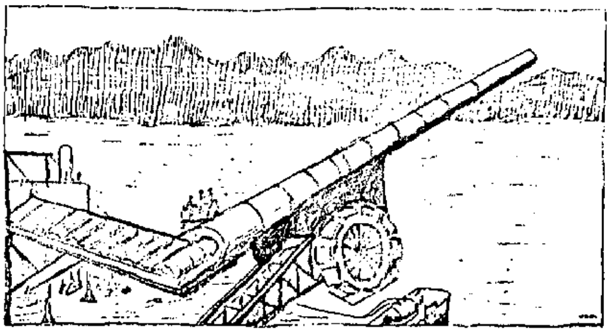


Fig E26

Our Fig E26 taken from Ref 4 consists of ten interconnected electromagnets. It is presumed that the cannon in Fig E26 was about 1/125th of actual size. Because Justrow's paper did not give a mathematical calcn of his cannon, Rogge undertook this work. He gave a detailed calcn on three pages of his paper for a cannon caliber 10cm and length of barrel 20 meters, propelling a projectile weighing 20kg with muzzle velocity 1500m/sec. The ten coils of the cannon were isolated by a heat-resistant material capable of withstanding a temperature as high as 70°C. For propelling the 20kg projectile with velocity of 1500m/sec it was required to have a generator capable of developing 13500 kilowatts and the duration of passage of the shell thru all ten coils was calcd to be 1/32.5 second

Rogge also calcd that for propelling by electromagnetic method of a projectile weighing 750kg with muzzle velocity of 1000m/sec from a barrel cal 38cm and 20 meters long, would require 100000 kilowatts, which is extremely high

Justrow in the forward to Rogge's paper stated that he considers that the length of 20 meters used by Rogge in calculating data for cal 10cm cannon corresponds to 200 calibers. No barrel of such length was ever constructed. The longest caliber length ever constructed was for ''Paris Gun'' (Parisgeschütz, in Ger) which was only 150 calibers, Justrow considered that for a cannon cal 10cm, the length must be 50–60 calibers and muzzle velocity 800 to 1000m/sec

In conclusion Justrow stated that as long as it is not known how the problem of stabilization of projectile flight by rotation can be successfully solved, the electromagnetic cannon will be less precise in hitting the targets than conventional cannons can achieve. Other difficulties of construction and high cost of electromagnetic cannons makes Justrow believe that such cannons were not practical at the time of appearance of Rogge's paper, which was in 1939

Refs: 1) Captain Alphons Spačil, "Mitteilung über Gegenstände des Artillerie- und Geniewesens'' (Austria), I. Heft 1906. Abstracted by W. Stavenhagen in SS **1**, 101(1906)

2) Oberleutnant Justrow, ''Rundschau Technischer Arbeit'', 1935, Nr 36 & 37 3) Ibid, ''Rundschau Deutscher Technik'', 1938, Nr 2, 4, 6, 7, 9, 11, 13 & 15 4) Dipl-Ing E. Rogge VDE, ''Probleme um Elektromagnetische Kanone'', SS **34**, 132–35(1939)

Electromagnetic Compatibility (EMC). Inhouse and site testing services are available for EMC. The Spectrum Control, Inc is equipped for testing to MIL-STD-461, MIL-I-6181 and similar RFI (Radio Frequency Interference) specs and also perfoms testing in accordance with FCC (Federal Communications Commission) and HEW (Health, Education & Welfare) requirements. The personnel are familiar with RADHAZ (Radiation Hazard) filter design andmeasurement problems

The company also manufactures a complete line of filters, feed-thru capacitors, variable ceramic capacitors, RFI shields, and knitted wire gaskets

Refs: 1) Spectrum Control, Inc, 152 E Main St, Fairview, Pa, 16415 2) G. Cohn, Ed, Expls & Pyrots 4(4), April 1971

Electromagnetic Field; Detonation Wave in. See Vol 4 of Encycl, p D729-R, Ref 96b

Electromagnetic Field Hazard Simulated. Susceptibility of munitions to unplanned deton from effects of high-powered electromagnetic fields can be tested with improved accuracy in one of the newest research facilities at Picatinny Arsenal, Dover, NJ. The electromagnetic hazard simulation chamber is believed unique in its capacity of creating concentrated power (radiation up to 10000 watts) and dissipating it at the chamber's center for test purposes. The chamber is 70ft long, 30 ft high and 30 ft wide

Thru electronic measurement of the power in the test environment, the chamber provides a means of estimating the amt of current that flows to the precise spot in a munition item where a detonator would be placed. Test results enable the munitions engineer to determine how susceptible a proplnt may be to the hazard from sources of EMF's. Included among such sources are nuclear bursts, lightning, static charge or communications and radar systems. Researchers say that the chamber is four orders of magnitude more efficient than an antenna for establishing high-quality electromagnetic fields for testing the RF susceptibility of munitions to undesirable deton

Field intensities of 100V/m can be established that are uniform over a 20-foot diam and can have a field impedance of 377 ohms or greater than 4000 ohms; also 266ma/meter with impedance less than 40 ohms. The system now operates over the frequency range from 300KHz to 3MHz and will soon perform down to Hz

Note: V means volts; m – meter; ma – milliamps; KHz – kilohertz and MHz – megahertz

Re/s: 1) Anon, Army R&D Newsmagazine **11**(3), March–April 1970, p 9 2) G. Cohn, Edit, Expls & Pyrots **3**(8), Aug 1970(Review of Ref 1)

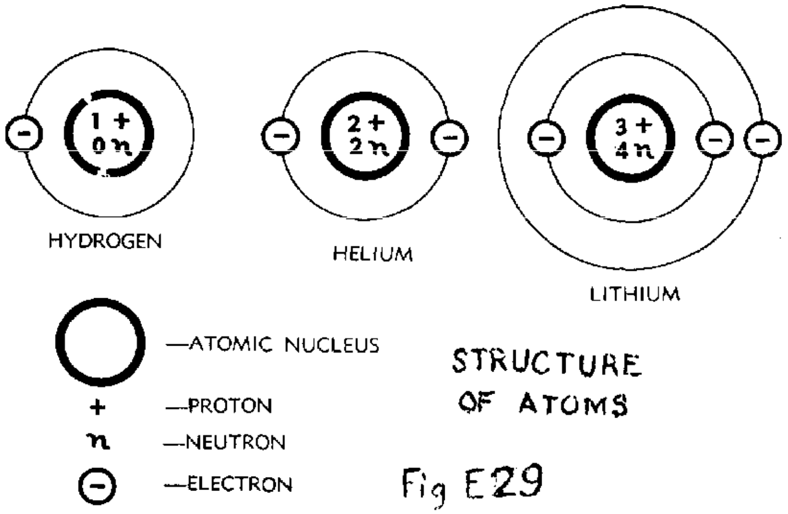
Electromechanical Fuze. Its definition is given in Vol 4, p D882-L, under Mechanical Fuze

Electron, Atom, Atomic Number, Atomic Weight, Electronic Shell, Element, Ion, Isotope, Mass Number, Molecule, Neutron, Proton and Valence.

Electron is the most elementary negatively charged particle which is the constituent of elements. Its weight is 1/1845 of the wt of the atom and its charge is equal to 4.77×10^{-10} cgs electrostatic units

Element is a substance (such as hydrogen, oxygen, nitrogen, iron, sulfur, etc) that cannot be decomposed into two or more substances by chemical means

Atom is the smallest unit of mass of an element that can participate in chemical changes. The relative weights of atoms of different elements are known as *atomic weights*. The atoms of the same element do not necessarily have the same weight. Atoms of the same element, but of different mass, are known as *isotopes*. In an arbitrarily established system, the atomic wt of oxygen is taken as 16. In this case the lightest of the elements, hydrogen, has atomic wt of 1.008. An atom of hydrogen weighs, however, 1.67×10^{-24} grams. While an atom is the smallest particle of an element that can be isolated by chem methods, it has been found that each atom is composed of subatomic particles (See Fig E29). An atom of hydrogen consists of a nucleus around which revolves one electron, while helium has two electrons revolving about and at the same distance from the nucleus. As no more electrons are found at this distance from the nucleus of any atom, these two electrons constitute the so-called first *electronic shell* of the atom. Lithium has three electrons, two of these constituting the first electronic shell and one in the outer electronic shell. The total number of electrons surrounding a nucleus of an atom is termed the *atomic number* of an element. The heaviest atoms have outer electronic shells contg 18 and 32 electrons. The number of electrons that can be gained or lost by an outer shell to form an *ion* (contg only completed shells or no shell in the case of hydrogen) determines the combining power or *valence* of an element. Hydrogen and lithium consequently have combining power. Helium with two electrons, has a full shell and hence has no chemical combining power. It belongs to the "inert elements" as do neon, argon, krypton, zenon and radon. The nucleus of the atom consists of positively charged



STRUCTURE OF ATOMS

Fig E29

protons and uncharged *neutrons*, except in the case of hydrogen which consists of a proton alone. An isotope of hydrogen, known as *deuterium* has a proton and neutron in its nucleus. Therefore, since the masses of a proton and of a neutron are almost the same, deuterium has approx twice the mass of an ordinary hydrogen atom. An atom of helium consists of a nucleus with two protons, two neutrons and two electrons. The total number of electrons in an atom is equal to the number of protons in its nucleus, and the sum of the number of protons and neutrons is termed the *mass number* of the element. Since the mass of a proton is very nearly the same as that of a neutron, the ".mass number" has almost the same value as the atomic weight. So small are protons, neutrons, and electrons that, like the solar system, most of the volume of an atom is space. For example, the diameter of a gold atom is approx 100000 times that of its nucleus

When atoms combine with each other there is formed a chemical entity termed a *molecule.* If two or more atoms of the same element combine there is formed a molecule of an element; for example oxygen (O_2), hydrogen (H_2), nitrogen (N_2), sulfur (S_8), phosphorus (P_4), etc, the subscript denoting the number of atoms in the elemental molecule. If two or more different atoms combine, there is formed a molecule of a *chemical compound,* such as water (H_2O), sodium chloride (NaCl) , sulfuric acid (H_2SO_4), etc

(See also "Electrons' and Neutrons' Action on Explosives")

Refs: 1) Hackh'sDict (1944), 299-L (Electron), 568-R (Neutron), 691-L (Proton) 2) Lange's "Handbook of Chemistry", McGraw-Hill, NY, 10th edition (1961), pp 1730-L, 1741-R and 1743-R 3) Kirk & Othmer **2** (1963), 806–14 (Atoms & Atomic Structure) 4) Anon "Military Explosives", **TM 9–1300–214 / TO 11A–1–34**, Depts of the Army and the Air Force, Washington, DC, November 1967, pp 3-1 and 3-2 ("Atoms and Molecules")

Electron. Engl name for Ger Elektron (qv)

Electron-Beam Welding. Such welding is being used in assembly of heat critical devices, especially EED's, and can be done in close proximity to expl charges. Production techniques including pulsed beam, beam deflection (for various weld shapes, e.g. round, square and elliptical) and multistation fixtures have been applied to reduce the number of pump-down operations

Refs: 1) Atlas Aerospace Newsletter No 4, Dec 1968, Atlas Chemical Industries, Inc, Valley Forge, Pa, 19481 2) G. Cohn, Edit, Expls & Pyrots **2**(4), April 1969 (Abstract of Ref 1)

Electron Bomb (Ger). See Elektronbombe

Electron Diffraction (Elektronenbeugung in Ger). It is the investigation of the structure of a surface of a substance by the diffraction (bending) of a stream of electrons directed upon it. Important differences between electron & X-ray diffraction arise from the lower penetrating power of electrons. This means that electrons are much more sensitive to small amts of matter and are preferred over X-rays for studying gases & surface films, which may be only a few atoms thick

The experimental technique when electron diffraction is used for detg the positions of atoms in solids requires the generation of a fine beam of high-speed electrons in a high-vacuum camera. In the study of gases, a jet of the vapor is directed across the electron beam

Refs: 1) Hackh'sDict(1944), 299-R 2) A. Weissberger, "Physical Methods of Organic Chemistry", Interscience, NY, Vol 1 (1949), pp 1109–1140: L. O. Brockway, "Electron Diffraction" 3) Encycl Britannica **8**(1967), 244–45

Electronic Flash Lighting High Speed Photography. Several systems of electronic flash tubes are known. For example, a flash unit known as a "Microflash Unit for Ballistic Photography" was developed in 1947 and described in 1948 by McCormick et al (Ref 1) for photographing large projectiles in flight. The light source combined high intensity with short duration. Several of these units were installed at the Aberdeen Proving Ground, Maryland

One of the electronic flash lighting high-speed photographic units described by Whelan (Ref 4) was installed at the US Naval Ordnance Laboratory, White Oak, Maryland. This system combined the desirable features of rotating prism-type motion picture cameras (such as Eastman Type III Camera) with those of electronic flash lighting. This resulted in a system which delivered extremely high overall definition and incorporated operating flexibility. With this system it was possible to obtain as many as 8000 frames per second without reduction of the illumination available per flash

Anderson & Whelan (Ref 2) described high–speed motion pictures with synchronized multiflash lighting. Lester (Ref 3) described electronic flash tube illumination for specialized motion picture photography

Some information on "electronic flash cameras", also known as "electric flash cameras" is given in Vol 2 of Encycl, under CAMERAS, HIGH-SPEED, PHOTOGRAPHIC, on p C14-R

When a visible light flash cannot be applied because extreme lighting obliterates the effect under study, one resorts to "X-Ray Photography", also known as "Radiography of High Speed Event". It is briefly described on pC17-R of Vol 2. Clark (Ref 5) described flash photography applied to ordnance problems. Quinn et al (Ref 6) described Kerr-cell camera and flash illumination unit for ballistic photography. Sultanoff (Ref 7) described a 100 million frame per second camera. Courtney-Pratt (Ref 8) described fast multiple plate photography. Aspden described electronic flash photography in his book (Ref 9)

The late F.R.Schwartz of Picatinny Arsenal presented at the 14th Meeting of PicArsnScientific Advisory Council, on April 26, 1957 a paper entitled: "High-Speed Photographic Facility for Study of Detonation"

Refs: 1) W.W. McCormick, L. Modansky & A.W. Farbanks, JApply Phys **19**, 221-25 (1948) 2) R.A. Anderson & W.T. Whelan, JSocMotPict Engrs **50**, 199-208 (March 1948) 3) H.M. Lester, Ibid **50**, 208-33(March 1948) 4) W.T. Whelan, Ibid **52**, 116-29 (March 1949) 5) C.Clark, JAppl Phys **20**, 363-70 (1949) 6) H.F.Quinn et al, Ibid **21**, 995-1001 (1950) 7) M. Sultanoff, RevSciInstr **21**, 653-56 (1950) 8) J.S. Courtney-Pratt, JPhotoSci **1**, 21 (1953) 9) R.L. Aspden, "Electronic Flash Photography", Temple Press, London (1959)

Electronic Fuze. Same as Electric Fuze defined in Vol 4, p D881-L

Electronic Power Pack. Under this title is given the following abstract in Expls & Pyrots **4** (12), Dec 1971:

"The ESD Power Pack is a capacity–type energy with the environmental and shelf life characteristics of a good quality capacitor, but at the same time an energy storage capability somewhat like a secondary battery. The cap consists of encapsulated series-connected ESD cells. It is offered as a standby power source for long shelf life and extreme temperature applications. Claims are that it will stay charged indefinitely without trickle charging, provide hundreds of identical discharge cycles plus >10 year shelf life, performing well beyond military semiconductor environments, cannot leak (no liquids involved), has no catastrophic failure mode, decouples...... absorbs line transients, and can be mounted on pc boards".

Note: The meaning of ESD and of pc board are not given in the abstract. They are:
ESD= Electronic Storage Device and
pc= printed circuit board

More detailed description is given in Bulletin 71922 or Report 71-E-57 entitled "ESD Applications to Storable Weapon Systems", published by Gould Ionics, Inc, P.O. Box 1377, Canoga Park, Calif, 91304

Electronic Shell. See above under Electron, Atom, Atomic Number, Atomic Weight, Electronic Shell, etc

Electronics. The branch of physical science that deals with study of electrons, especially with reference to industrial application. The devices which are studied in electronics are electron (or vacuum) tubes, electron microscope, photo-electric devices, x-ray apparatuses, audio-amplifiers, and audio-transformers

Most of these devices, are extensively used in Ballistics and other branches of Ordnance and for this reason the subject of electronics is mentioned in the Encycl and references are listed

Refs: 1) A.H. Hughes & L.A. duBridge, ''Photoelectric Phenomena'', McGraw-Hill, NY, (1932) 2) J.T. McGregor-Morris, ''Cathode-Ray Oscillography'', Chapman & Hall, London (1936) 3) G.Parr, ''The Low-Voltage Cathode Ray Tube and Its Application'', Chapman & Hall, London (1937) 4) J. Millman & S. Seeley, "Electronics", McGraw-Hill, NY (1941) 5) Massachusetts Institute of Technology, Dept of Electrical Engineering, "Applied Electronics", J.Wiley, NY(1943) 6) R.C. Walker, ''Electronic Equipment'', Chem Pubg Co, Brooklyn (1945) 7) V.K. Zworykin & E.D.Wilson, ''Photocells and Their Applications'', J. Wiley, N Y(1945) 8) G.M. Chute, "Electronics in Industry", McGraw-Hill, NY (1946) 9) W.I.Bendz & C.A. Scarlott, ''Electronics for Industry'', J. Wiley, NY(1947) 10) B. Lovell, Edit, ''Electronics and Their Application in Industry and Research'', Pilot Press, NY (1947) 11) A.L. Albert, "Fundamental Electronics and Vacuum Tubes", Macmillan, NY(1947) 12) L. Marton, "Advances in Electronics", Academic Press, NY, Vol 1(1948) 13) Westinghouse Electric Corp, ''Industrial Electronics Reference Book, J.Wiley, NY (1948) 14) T. Soler, Edit, ''Cathode-Ray Tube Displays'', McGraw-Hill, NY (1948) 15) A.V. Austin, ''Fundamentals of Vacuum Tubes'', McGraw-Hill, NY (1949) 16) P.G.Andres, ''Survey of Modern Electronics'', J. Wiley, NY (1950) 17) J.C. Slater, ''Microwave Electronics'', Van Nostrand, NY (1951) 18) F.P. Mitchell, ''Fundamentals of Electronics'', Addison-Wesley-Press, Inc, Cambridge, Mass (1951) 19) Encyclopedia Britannica, London, Vol **8** (1952), pp 323-340 V, ''Electronics'' 20) A.L. Albert, ''Electronics and Electron Devices'', Macmillan, NY (1956) 21) M. Wind, Edit, ''Handbook of Electronic Measurements'', PolyInst of Brooklyn, Brooklyn, NY (1956) 22) R. Herzberg, ''Electronics Handbook'', Fawcett Publications, Greenwich, Conn (1956) 23) R.W. Landee, D.C. Davis & A.P. Albrecht, ''Electronic Designers Handbook'', McGraw-Hill, NY (1957) 24) N.M. Cooke & J. Markus, ''Electronics and Nucleonics Dictionary'', McGraw-Hill, NY(1960) 25) H.V.Malmstadt & C.G.Enk with the assistance of E.C. Toren, "Electronics for Scientists'', Publd by W. A. Benjamin, Inc, New York, NY, 10025 (1962) 26) Encycl Britanicca **8**(1967), p 246

(See also refs under Electron Microscope and under Electron Tube)

Electronic Time Fuze. Development of an electronic time fuze which would be substantially more accurate than exisitng time fuzes is described in the report of Texas Instruments, Inc (Ref). A programmable digital circuit is driven by an oscillator based on a Picatinny Arsenal design. As power source, two types of battery, a silver-zinc primary battery and a reserve battery which uses ammonia as the electrolyte, were tried. A detailed manufacturing plan is included as an appendix

The fuze system consists of the fuze & a fuze setter. The fuze circuit consists of a stable low-frequency oscillator, a time base divider circuit, a programmable time accumulator, and a control circuit. The fuze setter consists of control switches and decade selector switches for programming fuze functioning time. The setter is a completely passive programming device which attaches to the fuze via a nose-mounted connector

The fuze was designed to be compatible with all artillery weapons from 105mm thru 8 inches. The fuze itself contains no expl components. An electrical output is provided as a firing signal at the end of the

timing sequence. An adapter, housing an electrically initiated detonator, was designed to allow existing safety & arming mechanisms to be used during evaluation firing tests
Ref: Anon, Texas Instruments, Inc, Final Report "Development of Electronic Time Fuze, XM574" (22 June 1964)

Electronites (Brit). Accdg to Daniel (Ref 1), original Electronite was *Amberite No 1* (See Vol 1 of Encycl, p A166-L) to which Amm carbonate was added. As this mixt was very unstable, the mixt of *Blasting Amberite* with Ca carbonate was proposed, but this did not find much use

More satisfactory Blasting Explosives were patented in 1895, 1897 and 1899 by Curtis and André

Following formulations are given by Daniel (Ref 1) and by Gody (Ref 2) (See Table E11)

Table E11

Components	No 2 (Ref 1)	No 2 (Ref 2)	No 3 (Ref 1)	No 3(Ref 1) (Modified)
AN	94–96	91–90	70–75	71–75
$Ba(NO_3)_2$	–	–	16–21	18–20
Woodflour (slightly roasted) with starch	6–4	9–10	6–9	7–10
Pine resin (purified)	–	–	6–9	–

Refs: 1) Daniel (1902), 175 & 253 2) Gody (1907), 706

Electron Microscope and Electron Microscopy
('Application of Electron Microscopy in Chemistry, Physics and Industry, Including Explosives)

The electron microscope is an optical instrument with magnification much higher than any other known instrument (magnifications of up to 100000 are obtainable, compared with 1000 for the ordinary compound microscope)

Essentially, an electron microscope consists of a source of electrons, sometimes called an electron gun (negative high voltage), and a series of devices serving as "lenses"; the first is a condenser lens, then an objective and a third lens which functions as a projection eye piece. The lenses may be operated either by electromagnetic or electrostatic arrangement. If the sample to be examined emits electrons, or can be made to emit them (e.g., by heating, ultraviolet illumination, etc), it can provide an electron beam and the microscope will be called an "*emission electron microscope*". If the sample cannot be made to emit electrons, then it has to be placed between a regular source of electrons (such as an electron gun) and the projector lens. Such microscopes are called *transmission electron microscopes*

In all of these microscopes, the image may be either photographed or rendered visible by projection onto a fluorescent screen
Historical:

The first electron microscope was built in Germany in 1931 by Knoll and Ruska (Ref 2). Its principles were based on previous works of L. de Broglie (1924), Busch(1926) and others. The first electron microscopes gave images inferior to those obtained by optical microscopes, but by 1934 a quite satisfactory instrument was obtained by B. von Borries, E. Ruska and M. Knoll. Commercial production of electron microscopes was begun in 1939 by Siemens and Halske, AG, Berlin. These instruments (the total number built was about 30) used electromagnetic lenses

The first instrument using electrostatic lenses was produced in 1932 by E. Brüche and H. Johannsen. The first practical application of the electron microscope was made in Belgium in 1934 by L. Marton, who built his own instrument. The first one on the American continent was built in 1939 in Canada by E.F. Burton at the University of Toronto

All of these instruments were designed to give enlargements of the source emitting electrons. The first electron microscope which could magnify objects not being electron sources was built in 1940 by H. Mahl and H. Brüchs. It was a transmission type electrostatic microscope

During WWII, great improvements in the electron microscope were made in the US by V.K. Zworykin and collaborators, working at RCA (Radio Corp of America), Camden, NJ. As result of this, the electron microscope has grown from a complex, highly specialized instrument, difficult to operate, into a simple apparatus capable of operation by relatively inexperienced personnel

The electron microscope serves as a means of conveying to the brain thru the sense of vision, qualitative information regarding the organization of structures in the range of dimension 10 Å to 10000 Å. Its major limitation at present is the lack of suitable criteria by which the accuracy of the representation presented by the microscope may be judged

(See also in this Vol: Electron, Electron Diffraction, Electronics, Electron Theory of Matter and Electron Tube)

Refs: 1) H. Busch, AnnPhysik **81**, 974(1926); ArchElektrotechnik **18**, 583(1927) 2) M. Knoll & E. Ruska, ZTechnPhysik **12**, 389 (1931) and AnnPhysik **12**, 607(1932) 3) V.K. Zworykin, JFrankInst **15**, 535(1933) (Electron Optics) 4) E.P. Burton et al, PhysRev **56**, 1171(1939) 5) V.K. Zworykin, Instruments **13**(6), 151(1940); Science **92**, 51 (1940) 6) M. von Ardenne, "Elektronen-Uebermikroskopie", J. Springer, Berlin(1940) (Reproduced by Edwards Bros, Ann Arbor, Mich) 7) Siemens & Halske AG, "Das Uebermikroskop als Forschungsmittel", De Gruyter, Berlin(1941) (Reproduced by Edwards Bros, Ann Arbor, Mich) 8) V.K. Zworykin et al, TransAmInstElecEngrs **60**. 157(1941) 9) V.K. Zworykin, IEC **35**, 450 (1943) (Electron Microscope in Chemistry) 10) C.H. Bachman & S. Ramo, JApplPhys **14**, 155(1943) (Electrostatic electron microscope developed by General Electric) 11) J. Alexander, "Colloid Chemistry", Reinhold, NY, vol 5, pp 152-235(1944); A.F. Prebus, "Electron Microscopy" 12) V.K. Zworykin, G.A. Morton & E.G. Ramberg, "Electron Optics and Electron Microscope", J. Wiley & Sons, NY, 1945 13) C. Magnan, A. Ertaud & P. Chanson, CR **220**, 28 May (1945) (Sur un projet de mecroscope protonique) 14) P. Chanson, Thesis, Masson, Paris (1946) 15) E.F. Burton & W.H. Kohl, "The Electron Microscope", Reinhold, NY(1946) 16) J.B. Le Poole, Philips Tech, Rev **9**, 33(1947) (New electron microscope with continuously variable magnification 1000-80000x) 17) M.E. Haine, JInstElecEngrs (London) **94**, 447(1947) (British electron microscope, the Metropolitan-Vickers type E.M.2) 18) J. Hiller, AnalChem **20**, 390(1948) (Analytical application of electron microscopy) 19) D. Gabor, "The Electron Microscope", Chemical Pub Co, Brooklyn(1948) 19a) R.C. Extermann, "The Electron Microscope, Theories and Application", Täuber & Co, SA, Zürich (1948) 20) V.E. Cosslett, Research **1**, 293(1948) (Recent advances in electron microscopy in the United Kingdom) 21) A. Weissberger, "Physical Methods of Organic Chemistry", Interscience, NY, 2nd ed, vol 1, part 1(1949), pp 971-979: E.E. Jelly, "Electron Microscopy" 22) C.J. Burton, AnalChem **21**, 36-40(1949) (Application of electron microscopy to analytical problems; includes 159 references) 23) R.W.G. Wyckoff, "Electron Microscopy, Technique and Applications", Interscience, NY(1949) 24) V.E. Cosslett, Ed, Bibliography of Electron Microscopy", Arnold, London(1950)(350 pages devoted exclusively to bibliography; many entries contain short abstracts) 25) Louis de Broglie, "L'optique électronique et corpusculaire", Hermann, Paris (1950) 26) V.E. Cosslett, "Electron Microscope", Interscience, NY(Sigma series) (1950) 27) V.E. Cosslett, "Introduction to Electron Optics", Oxford Press, NY (1950) 28) W.G. Berl, Ed, "Physical Methods in Chemical Analysis", Academic Press, NY, vol 1(1950), p 535-586: R.D. Heidenreich, "Electron Microscopy" (63 references) 29) V.E. Cosslett, "Electron Microscopy-Practical", Academic Press, NY(1951) 30) P. Chanson, MémArtilFr **25**, 175-220 (1951) (Microscope électronique) 31) S.J. Gregg, "The Surface Chemistry of Solids", Reinhold, NY(1951), p 159-72 (The electron microscope and electron diffraction; their application to surface problems) 32) F. Jobin, Vide **16**, 231-34(1961) & CA **56**, 5780 (1962) (Electron emission microscope) 33) J.W. Menter, RoyInstChemJ **86**, 415-24 (1962) & CA **58**, 4175(1963) (Electron microscopy) 34) F. Leonhard, "Electron Optical Patterns for Velocity Analysis", Ztschr für Naturforschung **9a**, 727-34 & 1019-31(1954). Engl transln by RIC (Redstone Information Center) is located in PicArsnLibrary as RSIC-42 and RSIC-43 (5 Aug 1963) 35) W.A. Ladd & M.W. Ladd, EncyclIndChemAnal **1**,

685-702(1966) & CA **64**, 16594(1966) (Electron microscopy, a review with 57 refs)

Electrons' and Neutrons' Action on Explosives *(Irradiation with Electrons and Neutrons of Explosives).* Kallmann & Schränkler (Ref 1) have observed the initiation of TNT, MF and of some azides when they were bombarded with intense beams of hydrogen, argon and mercury ions. They suggested that explosions were due to the activation of a few neighboring molecules. No expln took place if the vacuum pressure was very low

Muraour (Ref 3) has also observed the explosion of Silver Acetylide when irradiated with high-speed electrons. On the other hand, Trillat & Muraour (Ref 2) reported that they could not initiate expls, such as TNT, PA, Amm Picrate, NC, Smokeless Propellants and even NI_3 (one of the most sensitive expls) when they were exposed to bombardment of electrons in a special apparatus equipped with a Coolidge-Lenard Tube

Meldrum (Ref 4) studied the thermal decompn of nitrogen iodide at low pressure and showed the reaction to be:

$$2NH_3.NI_3 \rightarrow 2NH_3 + N_2 + 3I_2$$

Its activation energy is 18-19 kcal and its heat of formation is -35kcal/mole. The decompn reaction is retarded by ammonia & water and also by an increase in pressure over the solid

Bowden & Singh (Ref 5) found that when crysts of LA were irradiated with an electron beam of 75KeV and 200 microamps, an explosion took place, but it was suspected that this expln was probably due to bulk-heating of the sample. In order to prove that considerable heat is evolved on irradiation with electrons, crysts of K nitrate (mp 334°) and also some metallic wires were treated in the same manner as LA. During these experiments, the crysts of K nitrate readily melted and as some metallic wires fused, it was proved that there was considerable evolution of heat. In order to avoid this bulk-heating, Bowden & Singh bombarded the crysts of expls with slow neutrons (flux 10^8 neutrons per cm^2 per second). Bombardments of Silver Acetylide and of Pb, Cd and Li Azides produced no explns, although some changes (such as darkening or changes in color) were observed in the crysts

In a second series of experiments, the crysts of LA were mixed with uranium oxide and irradiated with neutrons so that fission of U-235 took place. Again no expln was produced. Bombardment with slow neutrons of NI_3 did produce an expln, but this was not due to neutrons, but simply to the well known fact that NI_3 usually explodes at ordinary temp as soon as ammonia (which is usually mixed with NI_3) becomes evaporated

Kaufman (Ref 6) studied the resistance of expls to the effects of low energy gamma rays. Data are reported for Pb styphnate, TNT, Tritonal, Baratol, Tetryl, Tetrytol, Pentolite, RDX, M15 Propellant, PETN, Ballistite, JPN Propellant, LA, DDNP, NG & MF. No correlation was found between radiation resistance & thermal stability, mech sensitivity or ignition temp

Refs: 1) H. Kallmann & W. Schränkler, Naturwissenschaften **21**, 379-82(1933) 2) J. Trillat & H. Muraour, BullFr **53**, 443-44 (1933) 3) H. Muraour, Chimie et Industrie **30**, 39(1933) 4) F.R. Meldrum, ProcRoySoc **A174**, 410(1940) 5) F.P. Bowden & K. Singh, Nature **172**, 378-79(1953) 6) J.V.R. Kaufman, ProcRoySoc **246A**, 219-25(1958) & CA **52**, 21105-06(1958) 7) J. Eggert, JPhysChem **63**, 11-15(1959) & CA **53**, 12680 (1959) (Ignition of expls by radiation) 8) E. Piantanida & M. Piazzi, Chim e Ind (Milan) **43**, 1389-93(1961) & CA **56**, 10437 (1962) (Behavior of expls under gamma irradiation)

Electron Theory of Matter. An atom is pictured as a nucleus around which are arranged electrons, or negative charges, equal in number to the net charge of nucleus. The nuclear charge and the number of external (outer) electrons vary from element to element, increasing by one for every increase in the atomic number of the elements. The total number of external electrons is thus identical with the atomic number of the element. These electrons account for valency and some other properties of elements. They can be liberated from the atom by radioactive disintegration, and transferred from one atom to another in oxidation-reduction reactions (electronation). Electrons can be made visible by the Wilson track method and by Millikan's fog chamber

The lowest number of electrons in an atom are: one for hydrogen, two for helium, while the highest numbers are: 92 for uranium, 93 for transuranium (ekarhenium) and 94 for ekaosmium

Refs: 1) Hackh'sDict (1944), 299-L and 629-30 2) Lange'sHandbook of Chemistry, 10th Edn (1961), 1730-R

Electron Tube, also known as *Cathode-Ray Tube, Radio Tube, Vacuum Tube* or *Valve*. It is a nearly-evacuated, sealed, glass vessel (such as tube) that contains a heated cathode, an anode (plate), and a third electrode or grid serving to control the negative circuit flowing between cathode and anode. Such a device is also known as *Three Electrode Electron Tube* or *Triode*. Its operation depends essentially on the flow of electrons emitted from the hot cathode. This tube is used for the detection of radio waves, for the amplification of electric currents, for the generation of alternating currents of a very great frequency range, and (since it transmits the current only in one direction) as a rectifier of alternating currents

A vacuum tube which contains only anode (cold) and cathode (heated), but no grid is known as *Two Electrode Electron Tube* or *Diode*. Such a tube can be used to rectify alternating current

Electron tubes found extensive use in Ballistics and other branches of Ordnance, as was mentioned under Electronics, where the following refs for these tubes are given: 1, 2, 3, 6, 7, 11, and 14

Historical development of electron tube is described in Encyclopedia Britannica, 1952 edition, Vol **8**, p 340H

Ref: Kirk & Othmer **8**(1965), pp 1–23 (Electron Tube Materials by R.J. Bondley)

Electron Tube Chronographs (Elektronenröhrenchronographe, in German). They are devices for measuring time intervals which incorporate in their electrical circuits one or several electron tubes. One of the first of such instruments was developed in 1927 by Büge (Ref 1) and is described in the book of Cranz (Ref 4). Another early electron chronograph was by Steenbeck & Strigel (Ref 2) and still another by Dodds & Fucks (Ref 3). All of them are described by Cranz (Ref 4)

In Vol 3 of Encycl, under CHRONOGRAPHS, are briefly described the following devices using electron tubes: Photoelectric-Screen (p C307-L), Sky Screen (p C307-L), Lumiline Screen (p C307-L), Ottawa Lumiline Screen (p C307-R) and Counter Chronograph, also known as Electronic Stop Watch (pp C309-R & C310-L)

Refs: 1) W. Büge, ZElektrotech **18**, 616–28 (1927) and **19**, 480–85(1928) 2) M. Steenbeck & R. Strigel, Ibid **26**, 831–40(1932) 3) J.M. Dodds & W. Fucks, Ibid **27**, 597(1933) 4) C. Cranz, "Lehrbuch der Ballistik", J. Springer, Berlin, Ergänzungsband(1936), 171–73

Addnl Refs: A) J.F. Roth, SS **28**, 42–6(1933) (Electron Tube Chronograph for Laboratory Method of Determining Detonation Velocities) B) C.R. Niesewanger & F.W. Brown, USBurMines, Rept of Investigation, **RI 3879**(1946) (Electronic Condenser Chronoscope for Measuring Detonation Velocities of Explosives) C) T. Soler, Edit, "Cathode Ray Tube Displays", McGraw-Hill, NY (1948) D) P.B. Sharpe, Ordn **34**,, 434–36(1950) (An electronic apparatus "Counter Chronograph" of Potter Instrument Co for measuring bullet velocities) E) K.J. Brimley, TransSocInstrTechnology(London) **2**, p 2 (1950) (Electronic Condenser Chronoscope) F) P. Fayolle & P. Naslin, MAF **26**, 767–817(1952) and **27**, 7–75 & 294–346(1953) (Description of various instruments used in France for measuring, in ballistics, short-time intervals; included are Electromechanical and Electronic Chronographs) G) H. Crenn, MAF **29**, 817–29(1955) (Electronic Chronograph used in France) H) H. Rumpf, Explosivstoffe **1957**, 261–67 and **1958**, 1–13 (Chronographs for measuring short-time intervals in ballistics; a review with refs) I) J.F. Rider & S.D. Uslan, "Encyclopedia of Cathode-Ray Oscilloscopes and Their Uses", J.F. Rider Publisher, Inc, NY (1959) J) J. Berger & J. Viard, "Physique des

Explosifs Solides", Dunod, Paris (1962), 133–37 (Chronographe à Oscillograph Cathodique) K) EncyclBritannica 5(1967), p 718 (Electronic-counter chronograph)

Electroösmosis and Its Application for Purification of Water. Electroösmosis is the diffusion of a liquid or a gas thru a porous wall or semi-permeable membrane caused by an electric field (potential gradient)

Inasmuch as the usual method of purification of water by distillation is expensive, it was proposed that the impurities be removed by electrolysis. For this, water is placed in a cell, divided by means of porous diaphragms into three compartments, a large one in the middle and two small ones on either side. Each outer compartment contains an electrode, connected to terminals of DC current. When the current is switched on, the electrolyte substances which are dissolved in the water, decompose, the positively charged metallic ions (such as Ca, Na, Mg, etc) move to the cathode, while the negatively charged ions, such as Cl^-, SO_4^{--} etc move to the anode. Eventually, all the electrolytes are removed from the center compartment, leaving pure water in it

Industrial application of electroösmosis has been described by Illig & Schwerin (Ref 1), Behrman (Ref 2), Patin (Ref 3), Barstow & Jebens (Ref 4) and by others. Definition of the term of electroösmosis is given in Hackh's (Ref 5), Stauffer in Weissberger's book (Ref 7) and by W.K.W. Chen in Kirk & Othmer's Encyclopedia (Ref 8)

(Compare with Electrophoresis)

Refs: 1) H. Illig & B. Schwerin, USP 1133967(1915) 2) A.S. Behrman, IEC **19**, 1229(1927) 3) P. Patin, Chimie et Industrie **19**, 205(1928) 4) E. Barstow & R.H. Jebens, IEC **22**, 1020(1930) 5) Hackh'sDict(1944), 300-R 6) A. Weissberger, "Physical Methods of Organic Chemistry", Interscience, NY, Vol 3(1950), p 319: R.B. Stauffer defined electroösmosis as "the movement of a liquid phase thru capillary orifices or along the walls of a solid phase, under a potential gradient" 8) Kirk & Othmer **7**(1965), 846–65 (Electrodialysis)

Electrophoresis, Electrophoretic Analysis and Electrophoretic Deposition. *Electrophoresis* may be defined as the phenomenon of migration of colloidal particles in a liquid due to the effect of an emf or potential difference across immersed electrodes. Most solids, being negatively charged, migrate to the anode, but there are some exceptions. Migrated particles lose their charge at the electrode, and are deposited on it

Electrophoretic Deposition consists in application of a DC electric field between two electrodes immersed in a suitable colloidal suspension, thus causing migration of the suspended phase toward one of the electrodes and the deposition of a coating at that electrode. This technique is usually applied when it is desired to deposit a uniform layer (coating) of a material on an irregularly shaped form. For example, deposition of rubber or synthetic polymers on various articles may be done by this method (Refs 5, 6 & 8)

Electrophoresis is important in the study of proteins because the molecules of such materials act like colloidal particles and their charge is positive or negative, according to whether the surrounding soln is acidic or basic. Thus, the acidity of the soln can be used to control the direction in which a protein moves upon electrophoresis (Ref 2)

Electrophoretic Analysis of colloidal mixtures was developed by Tiselius (Ref 1). His method was used by Longsworth & MacInnes (Ref 2) for analysis of proteins. Electrophoretic analysis was also used by Svensson (Ref 4) and was described in the book of Weissberger (Ref 5).

Electrophoretic Deposition is described in Kirk & Othmer's Encycl (Refs 6 & 8). The so-called *paper electrophoresis* is described in Ref 7. It consists of applying the sample with a micropipet at the center of a pencil line made on strips cut from Whatman No 1 filter paper. About 0.4–0.5ml of appropriate solv saturated with water is placed in a test tube. After the solv ascends, the strip is dried and sprayed with an indicator

(Compare with Electroösmosis)

Refs: 1) A. Tiselius, TransFaradSoc **33**, 524(1937) (A new apparatus for electrophoretic analysis of colloidal mixtures) 2) L.G. Longsworth & D.A. MacInnes, Chem-

Revs **24**, 271(1939) (Electrophoresis of proteins by Tiselius Method) 2a) Ibid, JACS **62**, 705(1940) (Interpretation of simple electrophoretic patterns) 3) H.A. Abramson, L.S. Moyer & M.H. Gorin, "Electrophoresis of Proteins and the Chemistry of Cell Surfaces", Reinhold, NY(1942) 4) H. Svensson, AktivKemiMineralGeol **A22**, No 10(1946) (Electrophoresis by the moving boundary method) 5) A. Weissberger, "Physicochemical Methods of Organic Analysis", VanNostrand, NY, Vol **1**(1949), pp 1685–1712 6) R.E. Kirk & D.F. Othmer, Edits, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **5**(1950), pp 606–10: M.A. Coler & M. Feinleib, "Electrophoretic Deposition" (18 refs) 7) M. Lederer, "An Introduction to Paper Electrophoresis and Related Methods", Elsevier, Amsterdam (1955) 8) Kirk & Othmer **8**(1965), pp 23–36 (Electrophoretic deposition by M.A. Coler)

Electroscope. An apparatus for detecting electrical charges consists in its simplest form of two strips of gold-leaf attached to an insulated conductor suspended from the stopper of a glass container (See Fig on p 300 of Ref 1). When desired to determine the potential of an *electrostatic charge*, a so-called "measuring electroscope" is used. It consists of an insulated gold-leaf mounted in a closed case having an adjustable insulated plate which can be rotated so that an electrostatic and gravitational balance is established. The position of the leaf is read in a low focus microscope (See Fig on p 301 of Ref 1)
Refs: 1) Hackh'sDict (1944), 300–01
2) EncyclBritannica **8**(1967), p 263

Electrostatic and Electrostatics. *Electrostatic* pertains to static electricity or electricity at rest. It can be produced by friction or atmospheric electricity. *Electrostatics* is the science of static electricity. Electrostatic discharges are frequent causes of fires and explosions. This problem is discussed under ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT
Refs: 1) Hackh'sDict(1944), 301-L
2) "American College Dictionary", Random House, NY(1952), 588-R & 1180-R
3) EncyclBritannica **8**(1967), pp 143–56 (Electrostatics)

Electrostatic Capacity. The ratio of quantity of static electricity to the difference of potential
Ref: Hackh'sDict(1944), 301-L

Electrostatic Characteristic of Explosives. See under ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT

Electrostatic Charges of Explosives and Their Prevention. See under ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT

Electrostatic Concentration. See under ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT and under Electrostatic Separation and Concentration

Electrostatic Currents Caused by Galvanic Action. See under ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT

Electrostatic Currents Caused by High Voltage Power Transmission Lines. See under ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT

Electrostatic Currrnts Caused by RF (Radio Frequency) Energy. See under ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT.

Electrostatic Current, Stray. See under ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT

Electrostatic Discharge. See under ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT

Electrostatic Elimination. Means of defeating static, including ionization of surrounding air and both air and chemical sprays, are currently available. Ionization is created by high-voltage power sources applied to "static bars" that act as ionization chambers. Placed near areas where static is either generated or stored, the bars supply positive ions to negatively charged objects or supply negative ions or electrons to materials that are positively charged. This process neutralizes the charged objects. A brochure "The Fascinating Story about Static" of Simco Co, Inc, 920 Walnut St, Lansdale, Pa, 19446, also lists ionizing air guns, nozzles, dust collector, static locator & generators, and antistatic spray
Ref: G. Cohn, Edit, Expls & Pyrots 1(4) April 1968

Electrostatic Hazards from Lightning. See under ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT

Electrostatic Hazards in Pneumatic Loading Systems. See under ELECTRICITY, EXTRANEOUS AND HAZARDS ASSOCIATED WITH IT

Electrostatic Law of Coulomb. The force between two electrostatic charges varies: a) inversely as the square of their distance apart; and b) directly as the product of their electrical charges. Hence, $F = (1/k)(qq^1/r^2)$, where F is the force, k a constant depending on the constants used, and q & q^1 the two charges which are a distance r apart

This law was formulated in the 18th century by French physicist Charles Augustin de Coulomb (1736–1806), in whose honor the unit of electrical quantity, signifying the number of electrons transferred by a current of one ampere in one second, was named **Coulomb.** This means that 1 Coul = 10^{-1}EMU = 10^9 x 3ESU = (VoltSec)/Ohm = Amp/Sec = 1/96489 Faraday, where EMU is abbr for "electromagnetic unit" and ESU for "electrostatic unit"
(See also under Electrostatic Units)
Ref: Hackh'sDict (1944), 229-R (Coulomb)

Electrostatic Precipitation. It may be defined as "the use of an electrostatic field for precipitating or rapidly removing solid or liquid particles from a gas in which the particles are carried in suspension"
Historical. The gases from a blast furnace, the smoke-stacks from boiler furnaces, cement plants, etc carry with them solid particles in the form of dust. While the greater part of this dust may be recovered by passing the gases thru a "dust collector", such as shown in Fig 242 of Ref 3, p 720 (our Fig E27) there are dusts, however, too fine to settle in the collector. The dust collector provides a space wider than the flue, in which therefore the velocity of the gases is reduced, and their carrying capacity correspondingly lessened, so that the dust deposits in the cone of collector and may be drawn out at intervals. Besides dusts too fine to settle in dust collector, many furnace gases carry fumes, finely divided droplets, or metallic particles just condensing from the vapor state which no dust box will retain. There are also, in some gases, fine toxic, solid particles which do not leave the slightest deposit in a dust box. This applies to the concentrators of sulfuric acid plants because their fumes carry par-

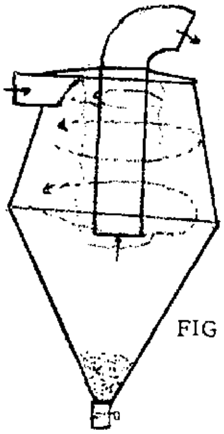


FIG E27

ticles of sulfur trioxide (SO_3) which cannot be precipitated by dust collectors (See Ref 2)

Ever since the fact of impossibility of collecting fine dusts and fumes in ordinary dust collectors was realized, attempts were made to improve the methods of separation of solid and liquid particles from gases. One such device is "Flick centrifugal catchall and entrainement separator", shown in Fig 241, p 721 of Ref 3. None of the devices proved to be successful until the invention of "electrostatic precipitation" in the USA by F.G. Cottrell (1877–1948)

The idea of using electrical discharges for the removal of particles suspended in air originated with Hohlfeld, who tried to apply it, apparently unsuccessfully, as early as 1824. Guitard (1850) and then Sir Oliver Lodge (1886) independently described methods essentially similar to that of Hohlfeld. Slightly later, A.O. Walker of England and K. Moeller of Germany secured patents for electrical precipitation, but it does not appear that their methods were successful. Cottrell began the study of the problem of electrostatic precipitation at the beginning of the present century and by 1906 succeeded in constructing a workable apparatus. The first practical demonstration of the process occurred at the Hercules Works of the DuPont Co at Pinole, California. After this, especially during WWII, many Cottrell installations were constructed at sulfuric acid plants equipped with a certain type of acid concentrator (Ref 2)

Description of Cottrell Precipitator. The principle of Cottrell method can be learned from Fig 243, p 722 of Ref 3, which is reproduced here as Fig E28. The dirty gas enters at pipe (3), connected at right angle to the vertical pipe (2), in which a copper or lead-covered copper wire (1) is suspended axially. The pipe (2) is 3 to 12 inches in diam and from 5 to 15 feet long. Its lower part is attached to a dust-collecting box. A high-tension direct current passes from the wire (1) to the surrounding pipe, as shown in Fig. This causes the particles of solids present in the dirty gas to be electrified by the silent discharge of wire and then to be attracted by the pipe, which has the opposite polarity. The gas issued from the top of the pipe is known as "clean gas" because it is free of suspended solid or liquid particles. The solids deposited on the walls of the pipe (2) are caused to drop off into the dust box beneath by rapping (Ref 3, p 722)

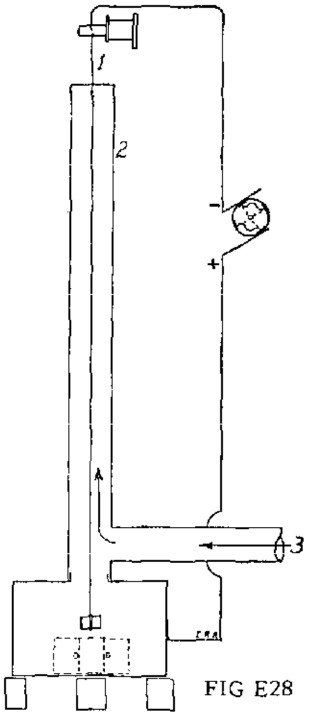


FIG E28

When a number of such units are nested in a shell with connections that permit the gas to enter at the bottom and emerge clean at the top, the system is called "pipe-type Cottrell" (Ref 4, p 471)

The Cottrell Apparatus of the Western Precipitation Corp, Los Angeles, Calif, shown in Fig 358, p 471 of Ref 4 and reproduced here as Fig E29 has no pipes but numerous wires suspended vertically in a box-like casing. The ensemble is known as the "precipitator" and it is located on the left hand side of the Fig E29. To the right of precipitator are located: *rotary rectifier*, transformer, and switchboard. The precipitator is *shown operating on a solid*

In this system, a high-tension current, ranging from 7500 to 100000 volts, is applied to one electrode in the form of a wire, while the other electrode, a flat surface, is grounded. The current is direct, mechanically rectified

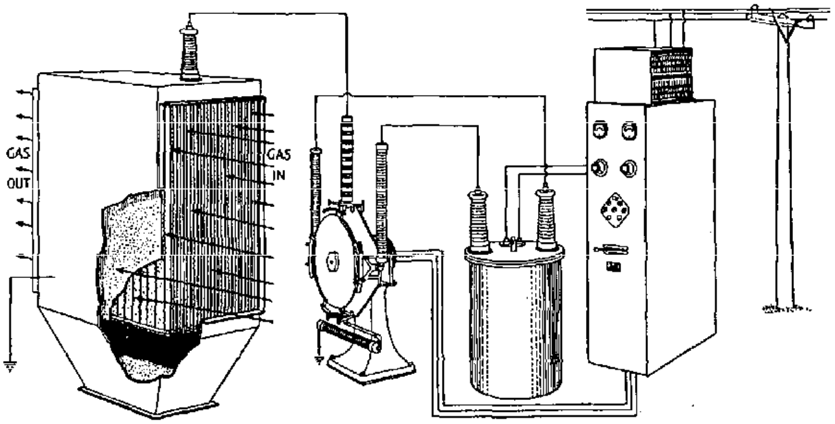


FIG E29

and best results are obtd when the ionized electrode (here the wire) is made negative in polarity. The suspended matter acquires a charge, partly from high-speed electrons from the current, partly from ionized gas, which precipitates them at the extended, grounded surface. Droplets so precipitated unite to form a stream of liquid, while solids form a coating which must be rapped off to a dust-collecting box below, at intervals (Ref 4, pp 470–71)

There is also the "plate-type Cottrell". It has a series of parallel plates in a casing which forms the channels thru which the gas flows. The discharge electrodes are suspended between the plates from an insulated framework. The flow of gas between the plates may be horizontal or vertical

The Cottrell process proceeds in four definite steps!
1) charging the particles by means of gaseous ions or electrons 2) precipitating or transporting the charged particles thru the gas to the collecting electrode by the force exerted on the charged particles by the elec field
3) discharging the charged particles and
4) removing the pptd material from the electrode to a suitable receptacle
(See also a brief biography of Cottrell with some refs given in Vol 3 of Encycl, p C547-R)

Refs: 1) F.G. Cottrell, IEC **3**, 542(1911) (The electrical precipitation of suspended particles) 2) A.M. Fairlie, "Sulfuric Acid Manufacture", Reinhold, NY (1936) (ACS Monograph Series No 69), pp 138–42 & 296 3) E.R. Riegel, "Industrial Chemistry", Reinhold, NY (1942), pp 722–23; 5th edit (1949); and (1962) by J.A. Kent 4) E.R. Riegel, "Chemical Machinery", Reinhold, NY (1944), pp 470–71; 2nd edit (1953) 5) R.E. Kirk & D.F. Othmer, Editors, "Encyclopedia of Chemical Technology", Interscience, NY., Vol **5**(1950), pp 646–62: L.M. Roberts, "Electrostatic Precipitation" (22 refs) 6) Kirk & Othmer **8**(1965), pp 75–92 (Electrostatic precipitation by L.M. Roberts & A.B. Walker)

Electrostatic Sensitivity of EED's. The design and operation of a circuit to study the pin-to-case electrostatic sensitivity of electroexplosive devices is described by Austing & Gortowski (Ref 1). This circuit provides a high voltage, low energy spark discharge that simulates the energy delivering capability of the human body. The circuit is then utilized to evaluate selected EED's that contain expl and pyrotechnic flash charges. It was shown that properly formulated pyrotechnic chges could withstand repeated electrostatic discharges at 25000 volts. Exploding bridgewire detonators and No 6 Electric Blasting Caps were also static insensitive at this potential. The role of a 500-ohm series resistance in the output of the circuit was discussed, and it was shown that the omission of the resistance had a drastic effect on output pulse profile but a small effect on the electrostatic sensitivity of EED's

Refs: 1) J.L. Austing & R. Gortowski, Explosivstoffe **19**(2), 39–43(Feb 1971) 2) G. Cohn, Edit, Expls & Pyrots **5**(3), March 1972 (Abstract of Ref 1)

Electrostatic Separation and Electrostatic Concentration. The principle of this operation is based on the fact that, if one or more of the materials in a granular mixture can receive a surface charge on or just before entering an electrostatic field, the grains of that material will be repelled from the active electrode or attracted toward it, depending upon the sign of the charge. By causing such grains to fall into separate chutes from other grains not so affected, a separation or concentration results

The ability of the grains to receive and to hold a charge, varies with the material. Some non-conducting substances, such as ebony or hard rubber, can take a charge by friction ("contact potential"), while others acquire it if they are passed thru a strong electrostatic field. If electrical conductors have received a charge, it is immediately dissipated when they come in contact with an electrode of the opposite sign

Refs: 1) F. Fraas & O.C. Ralston, IEC **32**, 600(1940) (Electrostatic separation of solids) (17 refs) 2) Ibid, "Contact Potential in Electrostatic Separation", USBurMinesRepts of Investigations **RI 3667**(1942) 3) Ibid, "Electrostatic Separator for Fine Powders", **RI 3677**(1942) 4) H.B. Johnson, ChemMetEngrg **50**, 130–33(Nov 1943) (Recovery of valuable materials by electrostatic separations) 5) J.H. Perry, Edit, "Chemical Engineers' Handbook", McGraw-Hill, NY(1950) pp 1093–95: J.L. Gillson, "Electrostatic Methods of Concentration" 6) Perry (1963), pp 21–67 to 21–70

Electrostatic Units (ESU or esu). See under Electrical Units and also under Electrostatic Law of Coulomb

"Elefant" (Elephant). Ger tank (Panzer) of WWII, known as *Jgd Pz VI Ausf Porsche* or *SdKfz 184s*. Accdg to Jarret (Ref 1), it weighed 75 short tons, was equipped with one MG and one 88mm A/T Gun, 71 calibers long. Accdg to von Senger & Etterlin (Ref 2), it weighed 75.6 short tons and was equipped with one MG and one 128mm A/T Gun, 55 calibers long (12.8cm Pak, L/55). Its max speed was 35km/hour (22 miles/hour)

Refs: 1) G.B. Jarrett, "Achtung Panzer". "The Story of German Tanks in WWII", Great Oaks, RD1, Aberdeen Md (1948) 2) Dr. F. von Senger & Etterlin, "Taschenbuch der Panzer 1943–1954", Lehmanns Verlag, München (1954) 3) Fedoroff et al, PATR **2510**(1958), p Ger 125-R & 126-L

Elektra (Èlectra). A WWII, German continuous wave navigation system using radio beacons providing *multilobe equisignal pattern*

Ref: Glossary of Ord(1959), 105-L

Elektron (Electron). Accdg to All&EnExpls (Ref 1) it is an alloy consisting of 86/14-Mg/Al, which burns at 1500°, while Mg alone requires 4200°. Accdg to Fisher (Ref 2), it is an alloy contg 86% Mg and 13% Al, invented by Germans in 1909 as a light metal substitute for structural steel, but since 1917 it was used for construction of incendiary bombs (See next item)

Refs: 1) APG, "Allied and Enemy Explosives", Aberdeen Proving Ground, Md (1946), p 49 2) G.J. Fisher, "Incendiary Warfare", McGraw-Hill, NY (1946), p 33

Elektronbombe (Electron Bomb). An incendiary bomb invented in Germany in 1917, but not used until WWII. Its description is given in Vol 2 of Encycl on pp B234 to B237 and on p B235 is a drawing and cross-sectional view

Refs: Same as under Elektron and several Refs on p B237 of Vol 2, which include description of methods of extinguishing fire produced by Elektronbombe

Elektronmetall Staub (Electronmetal Dust). An explosion of dust produced during machining operations with electron metal took place before WWII at one of the German plants. Bäck, who reported the accident (Ref), recommended applying, during machining operation, a constant stream of water to remove any Mg/Al dust

Ref: W. Bäck, Oberflächentech **13**, 55–6 (1936) & CA **30**, 3238(1936)

Element. See in this Vol under Electron, Atom, Atomic Number, Atomic Weight, etc

Elements, Delay in Fuze Detonators and Fuze Primers. See Vol 4, pp D856 to D863 and Figs 1-46, 1-47, 1-48, 1-49, 1-50, 1-51, 1-52, 1-53, 1-54 and 1-55. See also pp D866 to D867 and Figs 1-60 and 1-61

Eleostearic Acid (Eleomargaric Acid or Octadeca-9.11.13-trienoic Acid), $C_{17}H_{31}COOH$; mw 280.44; colorless, rhombic scales; mp 48°(α-form) & 71°(β-form); bp 235° at 12mm pressure (α-form); while β-form slowly decomp. This unsaturated, monobasic, aliphatic acid occurs in oils of the seeds of *Elaeococca vernicia* (Japanese wood oil) and tung oil. It was first investigated in 1875 by Cloëz (Refs 1 & 2) who named it oleomargaric acid. The name was changed to oleostearic acid by Maquenne (Refs 1 & 3). Kametaka (Ref 4) obtd it by treating tung oil with NaOH in water & alcohol, and refluxing for one hr on a water bath. Morrell (Ref 5) gave method of prepn of β-isomer, while Nicolet (Ref 6) gave method of prepn of α-form

Eleostearic acid is insol in w and sol in alc; its α-form is very sol in ether, while β-form is sol in CS_2

It is used in prepn of pentaerythritol oleostearate, a substance suitable for use in paints and varnishes, being superior to tung oil. It might be suitable as a binder in expl mixtures

Refs: 1) Beil **2**, 497, (212), [465,467] & {1515, 1518} 2) S. Cloëz, CR **81**, 469 (1875) 3) L. Maquenne, CR **135**, 696(1902) 4) T. Kametaka, JCS **83**, 1043–45(1903) 5) R.S. Morrell, JCS **101**, 2083–89(1912) 6) B.H. Nicolet, JACS **43**, 938–40(1921) 7) Kirk & Othmer **1**(1963), p 231(Table); **8**(1965), p 815 (Table)

Elephant Brand Gunpowder (Brit). A type of BkPdr manufd at the end of the 19th century by Curtis & Harvey, Ltd of London. It consisted of KNO_3 74.5–76.0, charcoal 14.5–15.5 & sulfur(sublimed) 9.0–11.0%. It was authorized for use in gaseous & dusty coal mines, provided 50% by wt of cooling agent, such as NH_4 oxalate or Na (or K) bicarbonate was placed in upper part of the cartridge, being separated from the expl by a diaphragm sufficiently strong to prevent mixing. Cartridges were wrapped in brown, so-called "spark-proof" paper

Refs: 1) Daniel (1902), 254–55 2) Thorpe (1917), 414 3) Molina (1930), 183–84

Ellagitannic or Ellagotannic Acid, known also as **Ellagotannin,** $C_{14}H_{10}O_{10}$, mw 338.1. It is a pale yellow crystalline, tannin-like substance, sl sol in water, extracted from the pods of *Caesalpinia coriaria,* a bark of *Punicia granatum* (Ref 2) and fruits of *Myrobalanus cebula* (Ref 1). Richmers (Ref 1)patented the use of organic substances contg ellagotannic acid as ingredients of expl mixts, together with NH_4ClO_4 alone or mixd with KNO_3 or NH_4NO_3. These mixts may be called *Rickmers' Explosives*

Refs: 1) W. Rickmers, BritP 10510 & CA **3**, 839(1909) 2) Hackh'sDict(1944), 303-R

Eluant or Eluent, also called **Elution Agent.** A liquid, such as alcohol, ether, acetone, methylene chloride, ethylene chloride, chloroform, petroleum ether, etc used in separation of ingredients adsorbed on a chromatographic column. This treatment, known as *elution,* is briefly described in Vol 3 of Encycl, p C291-R

Elution. See under Eluant or Eluent

Elutriation. A process of washing, decantating and settling which separates the suspension of a finely divided solid into parts according to their weight. It is especially useful for very fine particles below the usual screen sizes and is used for pigments, clay,

dressing and ore flotation
(Comp with Decantation described in Vol 3 of Encycl, p D22-R)
Ref: CondChemDict(1961), 436-R

Elvanol. DuPont Co trademark for polyvinyl alcohol, PVA, white to creamy-white, odorless, synthetic resin, sol in water. Used as an adhesive and binder (Ref 1) and desensitizing LA by coating it (See Ref 2, p A558-R)
Refs: 1) CondChemDict (1961), 436-R
2) Vol 1 of Encycl, p A558-R (PVA–LA)

Elvax. DuPont Co trademark for a series of high molecular weight vinyl resins. They are translucent white, amorphous copolymers, wax-compatible but insol in aqueous mixts and in most polar solvents. Used as a wax additive to improve its toughness, its heat-seal bond strength and to reduce flaking. Also used in laminating formulations, etc
Ref: CondChemDict(1961), 436-R

Emerald Powder (Brit). One of the varieties of *Cooppal Powder* (See Vol 3 of Encycl, p C512-R), manufd in England ca 1889. It contd NC, Ba nitrate & resins and was colored green by malachite (oxalate of tetramethyldiamidotriphenyl carbinol)
Ref: Daniel (1902), 255 (Émeraude)

Emery Paper Figure (Brit). See EPF

Émilite. An explosive patented by Audouin (EnglP 5899 of 1887), which was prepd by nitrating the fraction of coal tar boiling at 185–200°, after resinous compds had been removed
Ref: Daniel (1902), 256

Emission Spectrometry, Spectrography & Spectroscopy and Spectrochemical Analysis. Emission spectrography is devoted to the study of wavelengths and intensity of the radiations emitted by atoms and molecules under various physical conditions, and emission spectrometry is devoted to actual measurements of wave lengths and intensities

When emission spectroscopy or spectrometry is applied to the analysis of various compositions, this is known as *Spectrochemical Analysis*

In the emission spectrum analysis, a small piece of material to be analyzed is heated (burned) in an electric arc or spark and its spectrum recorded by means of a spectrometer. By comparing the wave lengths of the spectrum with those produced by known materials, it is possible to determine rapidly the composition of the sample

If quantitative measurements are desired, it is necessary to determine the intensities (brightness) of the spectra

For the arc method, carbon or iron electrodes are used. In the spark method, the "simple spark", Feussner spark, Hasler spark and Wolfe spark are used

Spectrochemical analytical methods received a tremendous impetus during WWII, because of the time and labor saving involved

(Compare with "Absorption Spectroscopy" in Vol 1 of Encycl, p A6)

Refs: 1) Jacques Bardet, "Atlas de Spectre d'Arc", G. Doin & Co, Ed, Paris (1926) (Pamphlet of 54pp and 54 plates) 2) W.C. Boettger, "Physikalische Methoden der Analytischen Chemie", AkadVerlagsgesel, Leipzig, Vol 1(1933) (Lithoprinted during WWII by Edwards Bros, Ann Arbor, Mich)
3) A. Heinrich & G. Scheibe, "Chemische Spektralanalyse", AkadVerlagsgesel, Leipzig (1939) (Reprinted by Edwards Bros, Ann Arbor, Mich) 4) W.R. Brode, "Chemical Spectroscopy", J. Wiley, NY (1942) 5) R.A. Sawyer, "Experimental Spectroscopy", Prentice-Hall, NY (1944) 6) G.R. Harrison, R.C. Lord & J.R. Loufbourow, "Practical Spectroscopy", Prentice-Hall, NY (1948)
7) N.H. Nachtrieb, "Principles and Practice of Spectrochemical Analysis", McGraw-Hill, NY (1950) 8) W.G. Berl, Ed, "Physical Methods of Chemical Analysis", Academic Press, NY, Vol 1(1950), pp 255–332: J. Sherman, "Emission Spectrography (31 references) 9) N.S. Sventitskii, "Visual Methods of Emission Spectroscopy", Davy, NY(1965), 344pp (Translated from Russian)

10) R.N. Dixon, "Spectroscopy and Structure", Wiley, NY (1965), 205pp 11) Kirk & Othmer, 2nd edit (Not found)

Emmens Acid. A yellow substance prepd in 1888 in the USA and in England by Dr S. Emmens of New York by dissolving an excess of PA (Picric Acid) in warm fuming nitric acid, followed by cooling. This gave crystals of mp 114–15°, called in the book of Daniel *l'acide Emmens*. Then the mother liquor was partly evaporated and cooled. This yielded a 2nd crop of crysts of different system than the 1st crop. They were called *le résidu* by Daniel. Both crops were used for an explosive known as *Emmensite* (qv)
Ref: Daniel (1902), 256

Emmensite (Amer & Brit). Accdg to Daniel (Ref 1), the Emmensite of 1888 (USP 376145 & EnglP 370) was prepd by melting together a mixture of L'acide d'Emmens & le résidu with equal parts of AN and Na nitrate. Accdg to Colver (Ref 2), Emmensite was prepd by melting together 5 parts of yellow crysts of Emmens Acid, melting at 114–115°, with 5p of AN and 6p of PA. This expl was tested by the USWarDept and found to be suitable as a bursting chge for projectiles, and as a demolition expl

Accdg to USP 422415 (1890), Dr Emmens proposed, in order to lower the mp of Emmensite, incorporating some aromatic mononitro-compd, such as Nitrobenzol. The same method was patented in Germany by the Chemische Fabrik Griesheim (Ref 1, p 256)

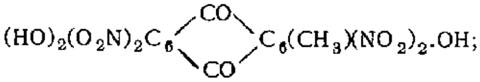
Another expl proposed by Dr Emmens (USP 423235 of 1890) was prepd by nitrating paper pulp, followed by drying and impregnating with a mixture of ammonia and PA. This expl was known as **Gelbite** (Ref 1, p 257)

Pérez Ara (Ref 3) stated that one of the Emmensites was prepd by mixing together PA, AN and concd sulfuric acid. It was a spongy, yellow, odorless mass of density 1.47
Refs: 1) Daniel (1902), pp 256 & 257
2) Colver (1918), 323 3) Pérez Ara (1945), 222

Emmet. Code name for 1,1,1-Trimethylpropane Trinitrate

Emodin or **1,3,8-Trihydroxy-6-methylanthraquinone,** $C_{14}H_4O_2(OH)_3CH_3$; mw 270.23, orn-colored crysts (from glac acet ac), mp 250-60°, sublimes w/o decompn; sol in alc, eth, chlf & benz; sl sol in petr eth, hot alc; v sl sol in chlf, glac acet ac, benz & acet; insol in w. It can be prepd by oxidation of emodinanthranol with chromtrioxide in acet acid at 50°, and by other methods (Ref 1)

2,4,5,7-Tetranitro-1,3,8-trihydroxy-6-methylanthraquinone,



$$(HO)_2(O_2N)_2C_6\begin{matrix} \diagup CO \diagdown \\ \diagdown CO \diagup \end{matrix}C_6(CH_3)(NO_2)_2.OH;$$

mw 450.23, N 12.45%; orn-colored crysts (glac acet ac or benz + petr eth), mp – deflagrates on heating & explodes mildly by impact; turns red on exposure to light; sol in water with an orn-red color; becomes violet in aq ammonia soln; red-brn in aq Na_2CO_3; and is unstable in aq (2N) NaOH. It can be prepd by treating Emodin with fuming nitric acid (d 1.5) & concd sulfuric acid (Refs 2 & 3)
Refs: 1) Beil **8**, 520, (743), [563] & {4154}
2) Beil **8**, (745) & {4158} 3) O.A. Oesterele & W. Sypkens-Toxopéus, Archiv der Pharmazie **249**, 314 (1911) & CA **5**, 2818 (1911)

Empfindlichkeit (Ger). Sensitivity or Sensitiveness, such as E. gegen Reibung (S. to Friction), E. gegen Stoss (S. to Impact), E. gegen Wärme (S. to Heat), etc

Empire Powder (Brit). One of the sporting proplnts: Guncotton 48, Collodion Cotton 34, petroleum jelly (vaseline) 7, nitrates of K and/or Na or Ba 9 & moisture 2%
Ref: Thorpe 4 (1940), 519

Emulsification, Emulsion, Foaming and Foam. *Emulsification* may be defined as the process of size reduction in which two or more immiscible liquids are intimately mixed, one as the dispersed or discontinuous, the other being the dispersing or continuous medium. The resulting fluid consisting of a microscopically heterogenous mixture of two phases both of which are liquid (or semi-liquid) at ordinary temperatures is called *emulsion*. For instance, if a small amount of an oil or soft fat is very rapidly agitated in a larger amount of water, the resulting emulsion will consist of minute droplets of oil or fat suspended in water. If on the contrary, a small amount of water will be agitated in a larger amount of oil or fat, the resulting mix will consist of minute water droplets, suspended in oil or fat. Milk is an example of an oil-in-water type of emulsion, while unworked butter, mayonnaise or creams are examples of water-in-oil emulsions

Foaming is the formation of a two-phase system where a dispersed phase is a gas and dispersion phase is a liquid. This is known as a *foam* and may be considered as the inverse of *aerosol*, described in Vol 1 of Encycl, pp A108-R & A109-L. Foam, also known as *froth*, usually forms on the surface or inside of some liquids, as for example during manuf of expls. Excessive foaming is objectionable in many industries because it might cause losses either by overflowing of the expended liquid (such as in boilers or nitrators); poor separation (as in separation of NG or MNT from spent acids); or formation of porous cast material (as in the case of filling projectiles with liquid expls discussed in Vol 1 of Encycl, pp A463 & A464-L, under "Antifoaming or Antifrothing Agents")

In many cases foam may be prevented by some mechanical means, such as by properly designed stirrers, while in other cases chem methods must be used. The earliest antifoaming agent was ether and it found use in laboratory practice. Following are some other antifoaming agents used in industry: vegetable oils (such as pine oil), animal oils, mineral oils, octyl alcohol, fatty acids and their esters, alkyl lactates, org phosphates, metallic soaps, sorbitan esters, silicones, highly polymerized glycols (such as "Ucon Brand Fluid"), higher fatty acid amides, particularly polyamides, etc. (See also next item "Emulsifiers or Emulsifying Agents")

Refs: 1) J.H. Perry, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950), pp 1167–69, "Emulsification" by C.K. Sloan 2) R.E. Kirk & D.F. Othmer, "Encyclopedia of Chemical Technology", Interscience, NY, Vol **5**(1950), pp 692–718, "Emulsions", by W.C. Griffin 3) Ibid, Vol **6**(1951), pp 772–78, "Foams", by E.I. Valko 4) Paul Becher, "Emulsions, Theory and Practice", Reinhold, NY(1957)(ACS Monograph No 135) 5) K & O, 2nd edit **8**(1965), pp 117–54, "Emulsions" by W.C. Griffin and **9**(1966), pp 884-901, "Foams" by L. Shedlovsky

Emulsifiers or Emulsifying Agents. Accdg to definition given by W.C. Griffin in Kirk & Othmer (Ref 2 of previous item, p 701), the term "emulsifier" is often misused. They are a subdivision of the general class of surface active materials. Other subdivisions are: wetting agents, solubilizers, detergents and suspending agents. "An *emulsifier* is employed in an emulsion formulation to increase the ease of formation of the emulsion or to promote the stability of the emulsion. These actions are usually accompanied by reduction of interfacial tension between the two phases and by protective colloid, respectively"

Emulsifiers may be divided according to their behavior into *ionic (anionic* and *cationic)* and *nonionic*

Examples of *anionic* emulsifiers are: Na, Li, or Al stearates; K laurate, Mg oleate, Na lauryl sulfonate, etc

Examples of *cationic* emulsifiers are: quarternary ammonium salts, amine hydrochlorides, etc

Examples of *nonionic* emulsifiers are: polyoxyethylene, polyoxypropylene, fatty alcohol ether, polyethylene (or polypropylene), glycol fatty acid esters, lecithin, lanolin, cholesterol, etc

In addition to the above, there are emulsion and suspension stabilizers that act as protective colloids and in some cases as thickeners: gums (such as acacia and tragacanth), alginates, starch and starch derivatives, casein, glue, egg albumin, methyl cellulose, hydrated Mg and Al silicates, etc

Refs: Same as in previous item

Emulsified NG and NC Explosive. Slurries of NC (CP_2 Bal) in emulsified NG were prepd at 15 & 30° with 0.5 & 1.0% emulsifier (CO 10) based on NG by using mechanical or air agitation. No stabilizer was added and %N detd for NC and the NG in rolled sheet (80–85°

rolling temp) after 5, 10 & 20 days. Best sheet was obtd at 30° & 0.5% emulsifier; denitration was less for all samples than for usual proplnt (GAL Bal 58/42) w/o stabilizer
Ref: J. Tranchant, MP **35**, 209–12(1953) & CA **49**, 12831(1955)

Emulsifiers in Preparation of Solventless Nitroglycerine Propellants. In prepn of these propellants, known in France as "poudres SD", where SD stands for "sans dissolvant", the usual practice is now to mix NC with an aqueous emulsion of NG and in order to facilitate the formation of emulsions, some emulsifier ("émulsionnant", in French) may be used

Tranchant investigated an emulsifier, known as "Emulphor CO-10" of General Aniline and Film Corp, and found that the addn of this material in quantities of the order 0.5% per total NG not only facilitated the gelatinization of NC (which was done by rolling, known as "laminage", in Fr) but also improved the quality of the resulting proplnt, such as its homogeneity and stability
Refs: 1) J. Tranchant, MP **35**, 209–12(1953) 2) CondChemDict (1961), p 438 (Emulphors are nonionic emulsifying & dispersing agents; the compn of Emulphor CO-10 is not reported)

Emulsion as an Explosive. D.R. Wiggam (USP 1999828 & CA **29**, 4180(1935) prepd an emulsion suitable as an explosive by rapidly agitating NG with a water-soluble cellulose ether, such as methyl cellulose
Ref: Clift & Fedoroff, Vol **2**(1943), p E1

EMW. Hercules castable double-base rocket proplnt described in conf Propellant Manual SPIA/M2(1969), Unit No 1071

"en". Designation of ethylenediamine as used in formulas for coordination compds, such as cobalt complex $Co[en](NO_3)_3$
Ref: CondChemDict (1961), 439-L

Encapsulation of Hazardous Materials by Ultrasonic Welding. The methods are described in Refs 1, 3 & 4 with abstracts given in Refs 2 & 5
Refs: 1) Technidyne Inc, PO Box 553, West Chester, Pa, 19380 2) Explosives & Pyrotechnics, Vol 1, No 9(1968) 3) Welded Metal Capsules for Pyrotechnics", Aeroprojects, Inc, 310 E. Rosedale Ave, West Chester, Pa, 19380 4) Charles Zglenicki & H.L. McKaig, Jr, "Ultrasonic Weld Encapsulation", Ordn **55**, July-Aug 1970, pp 68–70 5) Expls & Pyrots **3**(9), Sept 1970

Encapsulants, Unconventional. Epoxies and other potting materials conventionally used for electronic assemblies are so difficult to dissolve that they are useless in R & D (Research & Development) applications. The search for temporary encapsulants has led to several household items: paraffin wax, soapstone, rice and brown sugar
Refs: 1) Sandia Science News **6**(3), Sept 1971. Sandia Laboratories, Albuquerque, New Mexico, 87115

Enclosure (or Inclusion) Components. See under Clathrates and Other Inclusion Compounds in Vol **3** of Encycl, p C327

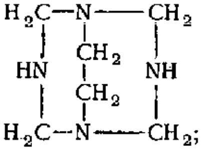
Endecacellulose Nitrate or **Endecanitrocellulose.** $C_{24}H_{29}O_9(ONO_2)_{11}$. See Endecanitrate in Table of Vol 2, p C100-R

End Effect in Detonation (and Explosion). See Vol 4, p D264

End Effect in Detonation Wave. See cross-ref in Vol 4, p D684-L and in Cook(1958), 91 & 98

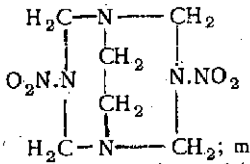
End Effect in Rockets is discussed in Wimpress, Ballistics(1950), 92

Endoethylene-tetraaza-cyclooöctane and Derivatives. *1,5-Endoethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane,*



a hypothetical substance which may be considered as the parent compd of its dinitro derivative:

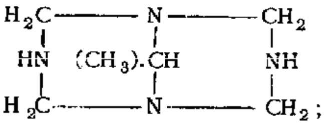
1,5-Endoethylene-3,7-dinitro-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane, code name **homo-DPT.** Its other names are **3,7-Dinitro(bicyclo)-2,4,6,8,9,10-hexamethylene-1,3,5,7-tetramine** or CA name, **3,7-Dinitro-1,3,5,7-tetraazacyclo[3.3.2.] decane,**



mw 232.20, N 36.20%; ndls (from acet), mp 140°(with frothing); resembles DPT in properties, sol in warm acetone and NBz; insol in w, ethanol, methanol, benzene, dioxane, AcOH and CCl_4; decomp in boiling w with evoln of formaldehyde. For its prepn Chute et al (Ref 2) treated a cold soln of 0.62g of dimethylolnitramide in 3.0cc of 37% formalin at 0°C with 3.3cc of 10% ethylenediamine soln, plus 5cc water. After 12 hrs the slowly formed ppt was filtered off, water-washed and dried at 50°. It weighed 0.52g (45% theory, nitramide basis) and melted at 134° to 139° with violent frothing. Recrystn from acet raised the mp to 140°. Its expl props were not investigated

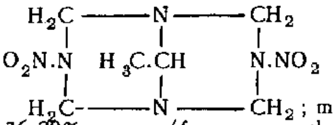
Refs: 1) Beil, not found 2) W.J. Chute et al, CanadJRes **27B**, 225 & 232(1949) & CA **43**, 8354f(1949) (No later Refs found thru 1966)

Endoethylidene-tetraaza-cyclooöctane and Derivatives. *1,5-Endoethylidene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane,*



a hypothetical substance which may be considered as a parent compd of its dinitro derivative

1,5-Endoethylidene-3,7-dinitro-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane or **3,7-Dinitro(bicyclo)-1,5-ethylidene,2,4,6,8-tetramethylene-1,3,5,7-tetramine** or CA name, **3,7-Dinitro-9-methyl-1,3,5,7-tetraazabicyclo[3.3.1] nonane,**

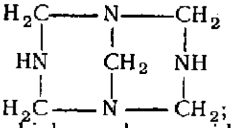


mw 232.20, N 36.20%; crysts (from warm ethyl acetate); mp 175° with a brown residue remaining up to 245°; sl sol in warm acetone and methyl ethyl ketone; insol in hot benz, ethanol, AcOH, chlf and dioxane. For its prepn Chute et al (Ref 2, p 234) cooled a soln of 0.124g of dimethylolnitramide in 2.75ml of 20% acetaldehyde by an ice bath, while 0.56ml of 10% ammonia was added. After 20-min digestion at 0°, 0.30ml of 37% formaldehyde was added and the mixture maintained at 0° for 2 hrs. Then the ppt was filtered off, water-washed and dried at 50°. The resulting product weighed 0.09g (yield 30% theory on formalin basis). Its mp was 173° but crystn from ethyl acetate raised it to 175°. Its expl props were not determined

Chute et al (Ref 2, p 235) treated Dinitroendoethylidene with 99% nitric acid in order to obtain impure **HMX** (called by them *1,3,5,7-Tetranitro-1,3,5,7-tetraazacyclooöctane*) of mp 261° to 271° with the yield of 22% of theory (1:1 mole basis)

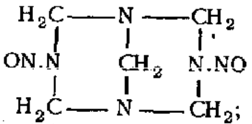
Refs: 1) Beil, not found 2) W.J. Chute et al, CanJRes **27B**, 225, 234 & 235(1949)

Endomethylene-tetraaza-cyclooöctane and Derivatives. *1,5-Endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane,*



a hypothetical substance which may be considered as a parent compd of its dinitroso- and dinitro-derivatives

1,5-Endomethylene-3,7-dinitroso-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane or **3,7-Dinitroso-(bicyclo)-2,4,6,8,9-pentamethylene-1,3,5,7-tetramine.** Its CA name is **3,7-Dinitroso-1,3,5,7-tetraazabicyclo[3.3.1]-nonane.** Called by Duden & Scharf, *Dinitrosopentamethylene* *tetramine* (DuPont's code name *Unicel-100)*. Another name is *Perform* and our abbrn, **DNsoPMT)**



; mw 186.18, N 45.14%; solid; mp – expl on heating (204–205°). It was first prepd by Duden & Scharf (Refs 1 & 2) in a small yield by nitrosation of molar ratio soln of formaldehyde-ammonia, contg some water and NaCl. Henry (Ref 2a) obtd DNsoPMT by nitrosation of a similar, but more concd, formaldehyde-ammonia soln. Richmond et al (Ref 4) repeated the prepn and confirmed experiments of Duden & Scharf and of Henry. Later they obtd the dinitroso, mp 198° in 36% yield (HCHO basis), by nitrosation of Henry's Solution. They gave on p 3663-L of Ref 4 the following method of prepn of Henry's Solution. Into 25g (0.31 mole) of 37% formalin at 0° was passed 6.5g (0.38 mole) of gaseous ammonia. To this cold soln was added 8g of K carbonate with stirring at 10° until two layers separated. The top layer, decanted after centrifuging, weighed 14.5g. To this was added 5g K carbonate and after stirring thoroughly, cooled in refrigerator, centrifuged and the top layer decanted. After another repetition with 5g K carbonate, the top layer, weighing 4.8g, was a viscous liquid, which did not freeze to a crystalline mass at -30°. For prepn of DNsoPMT, Richmond et al (Ref 4, p 3663-R) added (with stirring) to this freshly prepd, cooled to 0°, Henry's soln contg 0.031 mole ammonia and 0.031 mole formaldehyde in 20cc ice-water, a soln of 2.5g (0.036 mole) Na nitrite and 2.92cc (0.036 mole) concd HCl in 12cc ice-water. The resulting slurry was filtered and the ppt, when dried, weighed 0.34g and melted at 198°

Chute et al (Ref 5) did not describe the prepn of DNsoPMT, but just mentioned that its oxidation did not produce the dinitro derivative, DPT, described here as the next item

Bourjol (Ref 6) prepd the dinitroso deriv by nitrosation of HMTA (hexamethylenetetramine) by adding sulfuric acid, drop by drop, to an aq soln of HMTA and NaNO$_2$ at <10°

In ADL Rept (Ref 7a) is described the method of prepn of dinitroso deriv based on description given by P. Aubertein in MP **33**, 227(1951): Reaction of Na nitrite & hexamethylenetramine in aq soln at pH 2.0

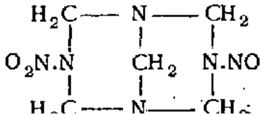
The effect of acids on lowering the decompn temp of DNsoPMT was studied by Ikuta & Nishimoto (Ref 7)

Schwab & Othmer (Ref 8) described a dust expln of DNsoPMT

It has been known that a very small quantity of DNsoPMT added to crude rubber rendered it, after heating, more pliable and spongy. It is probable that on the strength of this knowledge, the US Agency of International Development shipped in 1966 50 tons of DuPont's DNsoPMT (code named *Unicel-100*) at $1800 a ton to South Vietnam, presumably for manuf of soles for tennis. This quantity of DNso would be sufficient for manuf of many millions of soles and also to explode during shipping or in storage. The Agency evidently did not know that DNsoPMT is a high explosive, just slightly less powerful than TNT. Fortunately no accident occurred and all 50 tons of material were recalled after some Inspectors of the US State Department learned about the dangerous properties of DNsoPMT (Ref 9)

Refs: 1) Beil **1**, 589 2) P. Duden & M. Scharf, Ann **288**, 218(1895) 3) L. Henry, BullAcadRoyMédBelg **1902**, 721 4) H.H. Richmond et al, JACS **70**, 3663(1948) 5) W.J. Chute et al, CanJRes **27B**, 220(1949) 6) G. Bourjol, MP **34**, 7–12(1952) 7) S. Ikuta & A. Nishimoto, JSocRobberIndJapan **28**, 270(1955) & CA **50**, 6828(1956) 7a) Arthur D. Little, SynthHE's, 4th Rept (1956), p 112 8) R.F. Schwab & D.F. Othmer, ChemProcessEng **45**(4), 165–74 (1964) & CA **61**, 4141(1964) 9) Anon, "Mystifying Labels", "The Morning Call", Allentown, Pennsylvania, Tuesday, May 10, 1966

1,5-Endomethylene-3,7-dinitro-1,3,5,7-tetraaza-2,4,6,8-cyclooctane; or 3,7-Dinitro(bicyclo)-2,4,6,8,9-pentamethylene-1,3,5,7-tetramine. CA name **3,7-Dinitro-1,3,5,7-tetraazabicyclo [3.3.1]-nonane.** Code named **DPT** or **DNPT**, which is abbrn of *Dinitropentamethylenetetramine*,



; mw 218.18, N 38.52%, OB to CO_2 minus 80.7%; wh crysts (from acetone or other solvents), mp 206–208°(dec); explodes at higher temp; hydrolyzed by w; sol (besides acet) in ethyl acetate, Nitromethane, dioxane, acetic anhydride and acetic acid. It dissolves in weak nitric or sulfuric acids at pH 5.6 and can be reprecipitated at pH 3.4. DPT was prepd by several methods, such as:

1) In the course of RDX prepn by nitrating hexamine (hexamethylenetetramine, $C_6H_{12}N_4$) with 98% nitric acid (Hale's method) (Ref 3). An 18% yield of DPT was obtd when yield of RDX was 79%. The highest yield of DPT was 23.4%, but the yield of RDX was only 47% (Ref 7, pp 219 & 230)

2) A slurry of hexamine dinitrate (HADN), $C_6H_{12}N_4.2HNO_3$, in 4 moles acetic anhydride gave a 30.6% yield of impure RDX, mp up to 200°(Ref 7, pp 220 & 230)

3) Interaction of hexamine dinitrate with 90% aq sulfuric acid gave a 31% yield of impure DPT, mp 199–201°(Ref 7, p 230)

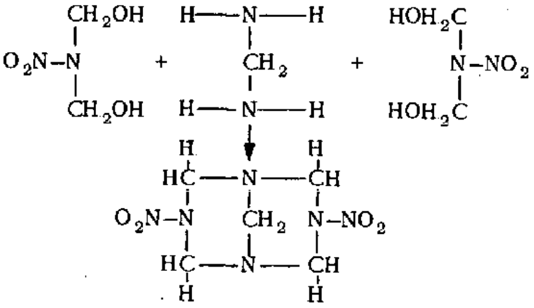
4) Interaction of dimethylolnitramide with formaldehyde-ammonia gave an 83% yield (on formaldehyde basis) of DPT, mp 206–08°. The method is described in Ref 7, p 230

5) The highest yield was obtd by Chute et al (Ref 7, p 231) by interaction of dimethylol nitramide and methylenediamine sulfate conducted as follows: The dimethylolnitramide was prepd from 2 moles of CH_2O and 1 mole of $NO_2.NH_2$ as described on p 68 of Ref 4.

The methylenediamine sulfate was prepd by method of Knudsen (Ref 2) from methylenediformamide and sulfuric acid using a sulfuric acid ratio of 5:6, while he suggested 3:4 (Ref 7, p 231)

Procedure: To a cold (at 0°C) nitramide-formaldehyde soln, prepd by mixing 0.10g (0.0016 mole) of dimethylolnitramide and 0.12ml (0.0016 mole) of 37% formaldehyde, was added 0.20g (0.0011 mole) of methylenediamine sulfate dissolved in 7.5ml water. The resulting soln was cooled to 0° and neutralized to pH 7 with satd aq soln of Na carbonate. The wh ppt which appeared at once was filtered after 40 mins, washed with 10ml water, and dried at 50°C. The product weighed 0.085g (yield 97% CH_2O basis) and melted at 206° to 208° with decompn

The reaction proceeded as outlined in Ref 7, p 223:



Other methods of prepn of DPT described by Chute et al (Ref 7) included:

6) Interaction of hexamine dinitrate (HADN) with a soln of AN in 99% nitric acid, as described on p 236. Yield of crude DPT (mp 190–92°) was 38% on the basis of one mole of HADN giving one mole of DPT

7) Interaction of *Dinitroxydimethylnitramide*, $O_2NO.CH_2–N(NO_2)–CH_2.ONO_2$, N ca 26.4% (powerful impact-sensitive explosive) with AN and formaldehyde, conducted as described on p 237, gave crude DPT of mp 200–201° with decompn. Yield calcd as DPT was 60% of theory (basis of 2 moles of Dinitroxydimethylnitramide giving one mole of DPT)

Richmond et al (Ref 6) prepd DPT as a test for the presence of $HCHO.NH_3$, $\overline{HN.CH_2.NH.CH_2.NH.CH_2}$, or $CH_2(NH_2)_2$, by addg to 37% HCHO at 0° nitramide; after dilution with water or with soln to be tested, the mixt was adjusted to pH 7.0 with NH_4OH or dil HCl. The yield was 21%

Attempts to prepare DPT by oxidation of Dinitrosoendomethylenetetraazacycloöctane did not succeed (Ref 7, p 220)

DPT is a rather unstable powerful explosive, less sensitive to impact than TNT (Bruceton No 5 Machine gave with 5kg weight 50% positive at 37cm, vs 30cm with TNT) (Ref 5)

DPT is a by-product in the manuf of RDX, Type B and may be isolated in yields 5.6 to 18.3% after removal of RDX

DPT was used by Chute et al (Ref 7, p 234) for prepn of **HMX** (High Melting Explosive), called by them *1,3,5,7-Tetranitro-1,3,5,7-tetraazacycloöctane*, mp up to 280°, with decompn.

DPT was treated with 99.6% nitric acid and the resulting slurry drowned in ice water after allowing to stand for 25 mins

McKay et al (Ref 8, p 467) used DPT for prepn of HMX. They treated a stirred mixt of DPT and acetic anhydride with a soln of AN in 99.6% nitric acid

McKay et al (Ref 8, pp 467–68) also showed that DPT can be used for prepn of a mixture of HMX and RDX. They added DPT to a stirred soln of AN in 99.6% nitric acid heated to 70–75° on a water bath. After 15 mins the mixt was drowned in ice-water, filtered and the ppt washed and dried. Its mp was between 187.5 & 189.5° and, accdg to thermal analysis by the mp compn diagram, it consisted of 30% HMX and 52% RDX

Aristoff et al (Ref 9) prepd DPT in 18% yield by nitrolysis of hexamethylenetetramine with Schiessler-Ross reagents (a mixt of AN, acetic anhydride & paraformaldehyde in molar ratio of 1.25/2.7/1, respectively)

HACS & IR (Ref 10) described the prepn of DPT of mp 205–06° from hexamethylenetetramine dinitrate with Ac_2O at RT for 3 days (yield 31%) or with 90% sulfuric acid at 15° for 45 min; also as a by-product of manuf RDX from hexamine

HMX, which is called *Homocyclonite* by HACS of IR was prepd by treating DPT with concd sulfuric acid (Ref 10)

Bachmann et al (Ref 11) prepd DPT in 48% yield by mixing instantaneously hexamine, 98% nitric acid & acetic anhydride in acetic acid at 25–30°. After addn of water, the DPT was collected on a filter & washed with water. Product had mp 198° (dec)

Refs: 1) Beil – not found 2) P. Knudsen, Ber **47**, 2698(1914) 3) G.C. Hale, JACS **47**, 2754(1925) 4) H.S. Booth, Ed, "Inorganic Syntheses", Vol 1, p 68, McGraw-Hill, NY (1939) 5) A.H. Blatt, OSRD **2014**(1944) 6) H.H. Richmond et al, JACS **70**, 3659–64 (1948) & CA **43**, 1316(1949) 7) W.J. Chute et al, CanJRes **27B**, 218–37(1949) & CA **43**, 8354(1949) 8) A.F. McKay et al, CanJRes **27B**, 462–68(1949) & CA **43**, 9072(1949) 9) E. Aristoff et al, CanJRes **27B**, 520–44 (1949) & CA **43**, 9075(1949) 10) HACS & IR, BritP's 615419(1949) & 615793(1949) & CA **43**, 9079(1949) 11) W.E. Bachmann et al, JACS **73**, 2771 & 2778(1951)

Addnl Refs for DPT:

A) A.H. Lamberton et al, JChemSoc **1949**, 1650–56 & CA **44**, 1413(1950) (Prepn of DPT)

B) R.N. Jones & G.D. Thorn, CanJRes **27B**, 828(1949) & CA **44**, 2848(1950) (UV absorption spectrum)

C) J.L. Boivin & G.F, Wright, CanJRes **28B**, 213–14(1950) & CA **44**, 9959(1950) (Prepn of DPT)

D) L. Berman et al, CanJChem **29**, 774(1951) (Prepn of DPT)

E) W.E. Bachmann et al, JACS **73**, 2769(1951) & CA **46**, 2084(1952) (Prepn of DPT)

F) W.E. Bachmann & E.L. Jenner, JACS **73**, 2773(1951) & CA **46**, 2085(1952) (Reaction of DPT with nitric acid & acetic anhydride)

G) W.A. Schroeder et al, AnalChem **23**, 1741, 1742(1951) (UV & visible absorption spectra in ethyl alcohol)

H) C. Holstead & A.H. Lamberton, JChemSoc **1952**, 1893 (Action of DPT on treatment with acetanilide in sulfuric acid)

I) E. Malmberg et al, AnalChem **25**, 901(1953) & CA **47**, 12095(1953) (Detection & estimation of DPT & other impurities in RDX)

J) G.F. Wright & W.J. Chute, USP 2678927 (1954) & CA **49**, 7606(1955) (Prepn of DPT and of HMX from DPT by treating with nitric acid or nitric acid-acetic anhydride-Ammonium Nitrate mixt)

K) R. Reed, JOChem **23**, 775(1958) & CA **53**, 14113(1959) (Prepn of DPT)

L) T.C. Castorina et al, JACS **82**, 1617(1960) & CA **54**, 18542(1960) (DPT as intermediate in nitrolysis of hexamethylenetetramine)

M) J.G.L. Harthon, ActaChemScand **15**, 1401 (1961) (in Engl) & CA **56**, 7567(1962) (Separation of DPT by thin-layer chromatography)

N) M. Witanowski et al, JACS **86**, 2570(1964) (N^{14} nuclear magnetic resonance spectrum of DPT)

O) T.C. Castorina & J.R. Autera, "Amino-Nitrogen-15 Tracer Studies of the Nitrolysis of Hexamethylenetetramine", Picatinny Arsenal, Dover, NJ, AD **601786**(1964), 49pp & CA **62**, 563(1965) (DPT as intermediate in nitrolysis of hexamine)

P) J.A. Bell & I. Dustan, JChemSoc, C, Org **1966**(9), 867 & CA **64**, 19615(1966) (Isolation of DPT from reaction of 1-acetoxymethyl 3,5-dinitro-1,3,5-triazacyclohexane by treating with Na Azide in dimethylformamide)

1,5-Endomethylene-3,7-dinitro-1,3,5,7-tetraaza-2,4,6,8-cyclooöctane (DPT) as impurity in crude RDX. Its detection and estimation by chromatographic method is described in Ref
Ref: E.W. Malmberg et al, AnalChem **25**, 901–07(1953)

Endosmosis. The diffusion which proceeds through a semi-permeable membrane, separating two miscible solutions, and tends to equalize their concentrations. The chief movement of solvent toward the denser solution *(endosmosis)* usually masks the slower diffusion *(exosmosis)* in the opposite direction.
Refs: 1) Hackh's Dict (1944), 305-R
2) Webster's Collegiate Dictionary (1962)

"Energa" Rifle Grenades were manufactured as early as 1950 by the Société Anonyme Belge de Mécanique et d'Armement, a private Belgian corp commonly known as Mecar. The US Government purchased substantial quantities of Energa grenades & launchers for use during the Korean war

The Energa grenade was capable of piercing 200mm of armor of a hardness close to 240 Brinell. It is a shaped-charge weapon weighing approx 1½ lbs, and has proved effective at ranges from 75–100 meters. It has a nose fuze with built-in safety. A hard rubber cap is provided as an addnl safety device. If the firer forgets to remove this cap before firing, the grenade will still function. There is a paraffin coated cork in the orifice in the tail assembly. The cork contains the propelling cartridge. Procedure on firing is to remove the cork, place the grenade over the launcher, and the soldier then has the propelling cartridge ready to load into rifle
Ref: G.F. Elliot, Ordn **36**(187), p 91 (July–August 1951)

"Energa" and "Stream" Rifle Launched HEAT Grenade. Title is unclassified, but the grenade cannot be described because it is classified
Ref: Dept of the Army, Senior Standardization Representative, Canada, User Trial Directive, "Rifle-Launched" HEAT Grenade (Energa and Stream) (U), 30 April 1959 [US STDZN/Inf 1372(S)]

Énergie. Fr for Energy

Energie. Ger for Energy

Energiegehalt. Ger for Energy Content

Energies, Activation. See Activation Energies in Vol 1 of Encycl, p A101-L

Energit and Tri-Westfalit, known also as **Nitroglycerin Pulvern,** were German mining expls prepd from surplus double-base propellants of WWI

Accdg to Naoúm (Ref, p449), the plants of the Nobel Co ground the different kinds of NG proplnt in Excelsior Mills betw steel discs, with an abundant flow of water, to a particle size of 0.5 to 2mm, dried it, packed it in cartridges 25 to 30mm diam and sold it under the name of **Energit.** WASAG prepd a similar expl, under the name of **Tri-Westfalit SN,** using in 1919 the following method: the NG proplnt was first softened by a 50% aqueous acetone soln, and then kneaded in a mixer with the gradual addn of 5 to 10 times its wt of water, which broke the material to a granular paste. However, it was found later to be more economical and simpler to crush the NG proplnt in rolls or edge runners to thin plates and then reduce this brittle form further. The product obtd gave a lead block expansion of 330 to 350cc. Potash mining has consumed the largest quantities of these expls. When unconfined Energit and Tri-Westfalit SN did not propagate detonation, but when confined they attained velocities of 3000 to 5000m/sec. The Prussian mining authorities prescribed the use of the strongest blasting cap to ensure deton, although fuse alone could cause expln when charges were well confined. On the other hand, the so-called "combined shooting" was found advantageous. This consisted in placing one or two cartridges of BkPdr upon the chge of Energit and igniting by a fuse which caused expln of Energit

In the 1st Mining List of Feb 1, 1923 (Reichsanzeiger, 1923, No 41, Appendix 2) both of the above expls were shown among expls for use in rock as **No 33 Nitroglycerin Pulver** (Nitroglycerin Powder), with the following composition: NG 30–40, NC 60–70 Nitroderivatives of toluene and/or naphthaline 0–5, paraffin and/or urethane/or carbamid and/or dicyandiamide 0–10%

In the later Mining Regulation, the No 33 NG Powder was stricken from the Mining List and replaced by **No 35 NG Powder 1**, which consisted of No 33 Pdr 94–96 & 50% Ca nitrate soln 6–4% and by **No 36 NG Powder 2**, which consisted of No 33 Pdr 97–99 & substituted urethane 3–1%. In the Regulations of 1924, No 35 appeared in place of Energit and No 36 in place of Tri-Westfalit SN

The above expls were also known as "Mining List Explosives Nos 33, 35 and 36"
Refs: 1) Naoúm, NG(1928), 448–50 2) Clift & Fedoroff **2**(1943), p E1

Energit A. One of the Ger expls studied by Lingens from the point of view of its thermal decompn. Its compn is not given in CA of the Ref, but probably is listed in the Belgian journal
Ref: P. Lingens, IndChimBelge **1967**, 32 (Spec No, Pt 3), 515–18(Ger) & CA **70**, 79647t(1969)

ENERGY. Energy may be defined as capacity or power to perform work and overcome resistance (Ref 1, p 305)

Energy may appear in various forms, such as:
Kinetic Energy. It is the force possessed by a body due to its motion and its numerical value is $mv^2/2$, where m is mass in grams and v is velocity in cm/sec (Ref 1, p 470)
Potential Energy, also known as *static* or *latent* is stored-up energy capable of performing work (Ref 1, p 681). It arises from the position of a body with respect to another body and is equal to the work required to achieve this difference in potential. Chemical, atomic (nuclear) and radiant energies are special forms of potential energy (Ref 3, p 441) and so is *Explosive Energy* described in Vol **4**, p D264 as "Energy of Detonation (and Explosion)"
Chemical Energy is energy produced during chemical reaction. Its intensity is expressed by chemical affinity or chemical potential, while the capacity is expressed by the equivalent weight or the active mass of the substance (Ref 1, p 187)
Atomic (or Nuclear) Energy is defined in Vol **1** of Encycl, p A500. See also "Nuclear Detonation (and Explosion) in Vol **4**, p D461
Radiant Energy is a dynamic disturbance of ether that diverges from common center and manifests itself as *heat, light* and *electricity. Infrared, ultraviolet, radio, radar* and *TV* belong to radiant energy (Ref 1, p 713 & Ref 2, p 107)
Mechanical Energy manifests itself in mechanical work which may be defined as the work done by the force which produces either a change in velocity of a body or change in its shape and configuration, or both. It can be defined as force multiplied by distance or as one-half mass multiplied by the square of velocity. The *unit of energy* is the *erg*, which is a force of one *dyne* acting thru one centimeter. The dyne is the force which when acting for one second, will give to a mass of one gram a velocity of one centimeter per second. Erg is also known as the *CGS* unit of work or energy (Ref 1, p 312 & Ref 3, p 441)
Specific Energy may be defined as the power of a body due to its relative position (potential) and motion (kinetic). Table of "Units of Specific Energy" is given in Ref 1, p 306
(Energy Content of Explosives and Propellants is discussed as a separate item)
Refs: 1) Hackh'sDict (1944), pp 187, 305–07, 312, 470, 681 & 713 2) Glossary of Ord (1959), 107 3) CondChemDict (1961), 441

Energy, Available in Detonation (and Explosion). See under Detonation (and Explosion) Power, Available Energy and Strength in, on p D476 of Vol **4**

Energy Content of Explosives. Since the consequence of detonation is the formation of gases: CO_2, CO, H_2O, H_2, N_2 and oxides of N_2, the energy release can be increased by choosing the ideal OB to CO_2 (oxygen balance to CO_2) and by storing up initial energy in the explosive in the form of molecular stress represented by negative heat of formation. In calculating the energy produced by the combstn of an org compd, one must subtract the heat of formation of the compd from the heat of formation of H_2O and CO_2. Hence, if the heat of formation of compd is negative, it is added to the total energy. A few expls have negative heats of formation. RDX is an example and is notable for its good expl performance, with the heat of expln 1302cal/g. In considering a mixture of fuel and a source of oxygen, it is interesting to note that TeNMe (Tetranitromethane) (first introduced in expl mixts in 1871 by H. Sprengel) has a negative heat of formation amounting to -64.8cal/g (-12.7kcal/mole). A number of fuel compds also have large negative heats of formation, as for example: acetylene -2110cal/g, carbon subnitride -1805, dipropargyl(hexadiyne-1,5) -1428, cyanogen -1336, hydrazoic acid -1267, hydrocyanic acid -918, Lead Azide -343, Mercuric Cyanide -246, Mercuric Fulminate -227, Silver Acetylide -350, etc. Some of these fuels have by themselves (without mixing with an oxidizer) an energy content greater than the best useful military expl. This energy may not be utilized, however, to its full extent unless the compd is oxidized, but it is potentially available. Dipropargyl(hexadiyne-1,5), for example, exceeds the heat of expln 1397cal/g reported for PETN, and carbon subnitride has an energy content in excess of that of any military expl. Yet these subjstances are not considered as expls unless they contain an oxidizer, such as TeNMe. When dipropargyl-(hexadiyne-1,5) is brought to zero OB by adding TeNMe, its energy (heat of expln) gives a calcd value of 1969cal/g, the highest of any mixts known. The carbon subnitride mixt with TeNMe with zero OB g ves a value of 1905cal/g. Mixture of toluene with TeNMe of zero OB gives 1784cal/g

Several other fuels were examined by Gilliland in zero OB mixts with TeNMe, but it was found that some fuels are incompatible with TeNMe. Most notably, acetylene is only very sl sol in it and metallic salts, such as Mercuric Cyanide and Mercuric Fulminate, are insoluble

Ref: ADL PureExplCompds, Part 2(April 1947) & Part 3(May 1950)

Energy Content of Explosives and Propellants. Accdg to Naoúm (Ref 2, pp 151-54), various ways have been proposed to express the relative value of the mechanical work performed (energy content) of an explosive. M. Berthelot expressed it in 1871 as the product of the specific gas volume and heat of explosion, V_o x Q_e (See "Berthelot's Characteristic Product" in Vol 2 of Encycl, p B105). W.H. Rinkenbach (Ref 3) using recent data for Q_e & V_o, found that there is a good relationship betw "characteristic product" and Ballistic Test values. Moreover, the theoretical work performed by an expl has often been expressed by multiplying the heat of expln by 425, the mechanical equivalent of heat, and thus giving it in meter-kilograms. Marshall (Ref 1, p 469) designated the expression 425 x Q as W and called it *total energy*. This is the "maximum quantity of work that can possibly be done by a unit wt of an explosive"

Note: Heat of Explosion, designated by us as Q_e, can be detd as described in Vol 4, p D377

Expressions Q_eV_o and $425Q_e$ take no consideration of the velocity of detonation, a very important factor in the kind of expl action, and one which is very considerably affected by the physical condition of the expl, its aggregate state, density, crystal form and granulation, as well as by extreme conditions of detonation, such as the initiating impulse, confinement and tamping (Ref 2, p 153)

Hess of Austria pointed out in 1873 the defect of both expressions and introduced the concept of "brisance" as the work done by a unit of wt of expl in unit time. He assumed a numerical value for this as the quotient of the Berthelot Characteristic Product and the time consumed by the expln. This period of time was to be calcd from the experimentally detd vel of deton. However, this method of detg the work done by an expl was not satisfactory. Still less satisfactory was the formula of Bichel published in Glückauf **41**, 465(1905) and mentioned in Ref 2, p 153

More satisfactory proved to be the formula of Kast, B = f x d x V, where B = brisance, f =

specific energy, d=density and V=deton vel. A detailed discussion of Kast's calcn is given in Vol 2 of Encycl, p B265, ynder BRISANCE OR SHATTERING EFFECT. Kast's formula (which he himself called, accdg to Naoúm, p 153, approximate) permitted the determination of specific energy f, if B, d and V were known. Brisance (B) can be detd experimentally by Kast's or Hess' Crusher Tests, described under "Compression (or Crusher) Tests for Determination of Brisance", in Vol 3, pp C492 & C493 or by Quinan Method described on p C493-R & C494-L

Brisance may also be detd by Sand Crushing Test, described in Vol 1, pp XXI & XXII and by Plate Tests, described in Vol 1, pp XIX & XX and in Vol 3, p D85 (Dent Tests)

Density (d) can be detd as described in Vol 3, pp D64-R to D85-L

Velocity of Detonation (V) can be detd experimentally as described in Vol 4, pp D632ff or it can be calcd as explained on p D630

It is mentioned above that Marshall (Ref 1, p 469) defined the *total energy* as $W=425Q_e$, but he also defined the *kinetic energy of the gaseous products of combustion* as 1.5pv, if pressure p is expressed in kg per sq meter for 1 kg exploding in vol v equal to one cubic meter. The Table on p 469 lists values of total energy and kinetic energy for 12 explosives

Marshall also stated that Trauzl's Lead Block Test affords a ready means of ascertaining the approximate relative strength of expls (pp 469–73); so does the Concrete Test (p 43) and Ballistic Pendulum Test (pp473ff) These tests, as well as other tests for detg strength (or power) are described in Vol 1 of Encycl, pp VII, VIII & XXV

Accdg to Naoúm (Ref 2, pp 154 & 156) the tests like Trauzl- or Crush Test measure the "work done in actual practice" or the "explosive effect of an explosive"

The following items related to energy of detonation (and explosion) are discussed in Vol 4 of Encycl: Critical Energy of Impact (or Shock) in Detonation (or Explosion), p D198; Energy of Detonation (and Explosion), p D264; Energy Relationships in Detonation (and Explosion), p D266; Energy (Relative) Release During Susan Test for Ignition of Explosives by Impact, p D266; Power, Available Energy and Strength in Detonation, p D476; Work Capacity in Detonation (and Explosion), p D730

Refs: 1) Marshall **2**(1917), 469-73
2) Naoúm, NG(1928), 151-58 (In Table 6, p 152 are listed for several explosives, the following properties related to energy: Berthelot's Characteristic Product, Work Done in meter-kg, and Relative Value, assigning to Blasting Gelatin the value of 100)
3) W.H. Rinkenbach, private communication, Allentown, Pa (1960)
Addnl Ref: F. Zwicky, Experientia **10**, 326-28(1954)(In Engl) & CA **48**, 14209 (1954)(Energy Content of Propellants and Explosives)

Energy Content of Propellant Charge. Accdg to Stickland et al (Refs) the energy content was considered in German calcns as equal to the charge weight of a propellant multiplied by its calorific value. For a given projectile and a given initial (muzzle) velocity, the energy content was considered as constant and independent of the type of proplnt used. For example, if for a certain initial velocity of a projectile, the charge weight of a proplnt with a calorific value of 820cal/g were 4.3kg, a propellant with a value of 570cal/g (such as "cool" Nitroguanidine proplnt, known as Gudolpulver) required a charge of 6.2kg
(See also Effective Calculated Calorific Values of Propellants)
Refs: 1) O.W. Stickland et al, PB Rept **925** (1945), p 82 2) PATR **2510**(1958), p Ger 42-R

Energy Content and Work Performed. Under the title "Energiegehalt und Arbeitsleistung", Stettbacher (Ref) gives the following equation: $A=Q \times 426.9$ meterkilograms, where A (Arbeitsleistung means performance of work, efficiency or output), Q temp of expln and 426.9 – mechanical equivalent of heat. This equation differs slightly from $W=425Q_e$ given here under "Energy Content of Explosives and Propellants"
Ref: A. Stettbacher, "Schiess- und Sprengstoffe", Barth, Leipzig (1933), 94

Energy, Critical, of Impact (or Shock) in Detonation (or Explosion). See Vol 4 of Encycl, p D198

Energy Equation. The equation of energy is a statement of how energy released by the combustion of a propellant is distributed during the operation of a gun. The general equation of interior ballistics can be written as:

$$\frac{cF}{\gamma - 1} = \frac{P(Ug - c\eta)}{\gamma - 1} + K$$

where: c=mass of proplnt burned, equal to mass of gas
γ=ratio of specific heats of the gas (assumed value 1.30)
P=space average pressure in lb/sq inch.
Ug=vol of proplnt gas in cubic inches
η=specific covolume of gas in cubic inches/lb
K=energy expended by gas in doing work and in heat conducted to the gun
F=energy per unit wt of proplnt(specific force)

Refs: 1) Corner (1950), pp 175 & 184
2) Anon, "Interior Ballistics of Guns", AMCP **706–150**(1965), pp 2–3ff

"Energy Exchange in Shock and Detonation Wave". Title of the paper by D.F. Homing, InstInternConseilChim **12**, 311–33(1962); Discussion in English, 334–39(30 refs); CA **61**, 8905(1964). It is listed as Ref 80 on p D728-R in Vol 4 of Encycl

Energy (Kinetic) of the Gaseous Products of Explosion. If in the equation of "force f of an explosive" (force spécifique, in Fr): f=pv, the pressure p is expressed in kg per sq meter for 1 kg charge exploding in 1 cu meter of closed space, then the value 1.5pv, accdg to the kinetic theory of gases, is equal to the kinetic energy of the explosion

Marshall (Ref) gives in Table on p 469 comparative values of 1.5pv (expressed in kg-meters per 1 g of expl) with W=425Q (where W is total work produced by explosion, Q is the heat of explosion and 425 mechanical equivalent of heat). Some examples are given in Table E12

Table E12

	W	1.5pv
Blasting Gelatin	604	158
Dynamit No 1	497	108
Carbonit I	255	73
Black Powder	244	42

Ref: Marshall **2**(1917), 469

Energy Measurements in the Explosion of Primers. Accdg to Ref, pressures produced by the detonation of various primer explosives in a lead-lined, nitrogen-filled closed bomb were measured and recorded with a piezoelectric sensing element (built into the bomb), an amplifier, and a cathode-ray tube. The explosives used were Silver Azide, Lead Azide, Mercury Azide, and Mercury Fulminate. The pressure diagrams obtained differed depending on the kind and quantity of explosive, the packing and whether the material was lumpy or finely crystallized

Ref: W. Noddack & E. Grosch, Zeitschrift für Elektrochemie, **57**, 632–36(1953), Picatinny Arsenal, Translation No 72. Translated by US Joint Publications Service

Energy of Recoil. In the book of Meyer (Ref, p 115) is the following statement:

"A point worthy of notice is that the force of explosion of the propelling powder is exerted equally upon the projectile and upon the gun in terms of Newton's Law of Action and Reaction. The backward acceleration of the gun, proportionally to its weight, is nearly the same as that of the projectile, and results in the phenomenon of recoil. Energy of recoil is made use of in many features of gun design including automatic firing"

In free recoil of gun & carriage, the kinetic energy of recoiling parts is (Ref 2):

$$\frac{(W+C/2)^2\ V^2}{2gR}$$

where
W = wt of projectile
C = wt of proplnt chge
V = vel of projectile in inch/sec

g = gravitational acceleration, inch/sec^2
R = wt of recoiling parts in lb
Ref: 1) M. Meyer, "Explosives," Th.Y. Crowell Co, NY (1943), p 115   2) Anon, "Interior Ballistics of Guns", AMCP **706–150**(1965), p 2-4

Energy Relationships in Detonation (and Explosion). See Vol 4 of Encycl, p D266

Energy (Relative) Release During Susan Test fof Ignition of Explosives by Impact. See Vol **4**, p D266

Energy (Relative) of Shock Wave Produced by Charges Fired Under Water is discussed by Donna Price in conf NAVORD Rept 6829, Energy Partition in Underwater Explosions of Organic Explosives; Application to Waxed and Aluminized Explosives" (U) (March 1960)

Energy Sources. In explosive chemistry the energy released by rapid chemical reaction is used to provide heat, expand gas, create blast shock, provide fragmentation, and to create forces for propulsion

Another source of energy is that which holds together the particles of atomic nuclei. The magnitude of energy available from nuclear reactions is vastly greater than that from chemical reaction. For example, TNT can release 84000 ft–lbs of work per pound of expl, whereas plutonium in fission reaction may deliver 3.0×10 ft-lbs/lb. Also by changing the number of particles in the nucleus and converting one element into another, millions of times more energy can be produced than by any electrical or chemical processes known
Ref: Anon, "Elements of Armament Engineering", Part 1, "Sources of Energy", Ordnance Corps Pamphlet, ORDP **20–106** (1960) & AMCP **706–106** (1964)

Energy vs Velocity of Deflagration of Colloidal Propellants. Accd to Muraour & Aunis (Ref)' the energy Q, in kcals liberated at constant volume by decompn of 1kg of proplnt (or explosive) is shown graphically to be linearly related to the temperature, in degrees K, developed on deflagration (or explosion) of proplnts between 2000 and 4000°K. The values of the velocity of deflagration can be obtd from the equation:

$$\log V = 1.47 + 0.846\ (Q/1000)$$

They agree with those calcd from equation

$$V = 1.37 + 0.27\ (T/1000)$$

The proplnts investigated by Muraour & Aunis contd, besides NC, varying amts of DEGDN, PETN, NG (Nitroguanidine) and EtCentr
Ref: H.Muraour & G. Aunis, CR **229**, 173-75 (1949) & CA **43**, 8681 (1941)
Addnl Refs: A) H. Muraour, Chimie et Industrie **50**, 105-08; & 168-72 (1943); CA **39**, 3161 (1945) & **40**, 2986 (1946) (Relation between the temperature of explosion of a propellant and its velocity of deflagration) B) H.Muraour, CR **228**, 818-20 (1949) & CA **43**, 5189 (1949) (Study of the laws of combustion of colloidal propellants)

Enfield Rifle. British rifle invented before WWI and used successfully not only by the British but also by Americans. It was known in US as Rifle Caliber.,30, Model 1917. It was a bolt-type, breach-loading magazine-type weapon, holding 5 cartridges. Its length was 3' 10.3", the weight 2 lbs 15 ozs, and the range of up to 2000 yds (Refs)
Refs: 1) Maj E.G.B. Reynolds, "The Lee-Enfield Rifle", Herbert Jenkins, London (1960), pp 123-28   2) H.E.MacFarland, "Introduction to Modern Gunsmithing", Stackpole, Harrisburg, Pa (1965), pp88-90

Engels patented at the end of the 19th century the following expl compn: NG 60-70, "pyropapier" (See Note) 15-18, NH_4 salts (nitrate, sulfate or chloride) 10-30, saltpeter 8-10, Pyroxyline 5-10, Nitromannite 1.5, Nitrostarch 0.5, Nitrobenzene 0.5 & waterglass 0.5%
Ref: Daniel (1902), 268
Note: "Nitropapier" or Papier fulminant" was prepd by plunging for 2 mins sheets of paper not contg glue in a 50:50 mixture of nitric-sulfuric acids, followed by washing and drying. It served as a component of some

expls mixts, such as *Spiralite* and *Unionite*
Ref: Daniel (1902), 665

Engin. Fr for device, engine, appliance or missile

Engin autopropulsé. Self-propelled device. This term corresponds to both reaction motor and jet-propèlled motor. It is also used for a rocket launcher, such as "Bazooka"

Engin criminel. French term for any device, such as a bomb, mine, etc used by revolutionaries or terrorists in attempts to assassinate Govt officials or for the purpose of sabotage, etc. These devices were called in Russia "Adskiye Mashiny" (Hell's or Infernal Machines"). Stettbacher called them "Anarchistenbomben, Nihilistenbomben" or Höllenmashinen"
Ref: 1) Daniel,Dict (1902), 268-83 (Engins criminels) (Interesting, detailed description) 2) Stettbacher (1933), p 417-19 3) Stettbacher (1948), p 131

Engin à fumée chaude de Berger (Fr). A smoke-producing pyrotechnic device invented by Berger and used during WW I, It was operated by the combustion of mixtures such as CCl_4 40, ZnO 20, powdered Zn 15, Ca silicide 15 & Na perchlorate 10%. Several models existed
Ref: Pepin Lehalleur (1935), p 470

Engin à fumée froide de Verdier (Fr). A smoke-producing device invented by Verdier and used during WWI. It operated by ejecting a mixture of stannic chloride and ammonia into a humid atmosphere. Several models existed
Ref: Pepin Lehalleur (1935), p 471

Engin sol-sol. Fr for Surface-to-Surface Missile

Engine. Any mechanism or machine designed to convert energy into mechanical work. Also a machine or instrument used in warfare, as a battering ram, catapult, piece of artillery. Engines converting heat energy into mechanical work include: steam engines, internal combustion engines (such as Diesel and gasoline engine), turbines (such as steam and gas turbines), pulse-jet engine, ramjet engine, rocket engine (or motor) and turbojet engine
Refs: *1)* J.R. Allen & J. B.Bursley, "Heat Engines", McGraw-Hill, NY (1941) 2) D.A. Wranghan, "The Theory and Practice of Heat Engines", Cambridge Univ Press, Cambridge (1942) 3) C.L. Barhart, Ed, "The American College Dictionary" Random House, NY (1952),p 398-R 4) Glossary of Ord (1959), p 107 (Engines) and p 246 (Rocket engine) 5)Collier's Encycl **9** (1965), p 139-47

Engineering. The art or science of making practical application of the knowledge of pure sciences such as chemistry or physics
Refs: 1) J.C.L.Fish, "The Engineering Method", Stanford Univ Press, Stanford, Calif (1950) 2) J.H. Perry, Ed, "Chemical Engineers' Handbook", McGraw-Hill, NY (1950) 3) O.W. Eshbach, "Handbook of Engineering Fundamentals", Wiley, NY (1952) 4) J.E. Goldman, Ed, "The Science of Engineering Material", Wiley, NY (1957) 5)Encycl Britannica **8**(1967), p 391ff

Engineer's Special Blasting Caps which were covered by US Army Spec No 49-20A seem to be identical with *US Army Special Blasting Caps*, described in Vol **2** of Encycl, p B188-R

Engineer's Special Detonator. Its casing is bronze(90/10-Cu/Zn), 3 inches long OD 9/32", wall thickness 0.006-0.008" and bottom 0.020-0.030". Lead Wires No 22 Cu 12" long with a resistance of 0.015 ohms, insulated from the casing by an asphaltic material. The bridge is of Pt-Ir, 0.0013" in diam

with a DC resistance of 1 ohm. The initiating chge is 0.3 g Diazodinitrophenol & 0.9 g PETN
Ref: E. Bonner et al, BRL Tech Note No **1470** (1962)

English Military Ammonals. See Vol **1**, p A290, table

English Service Explosive. An *Ammonite* of compn: AN 65, TNT 15, Al 17 & Charcoal 3%, which was used during WWI (Ref 1,p 117). A slightly different compn, namely: AN64,TNT 15, Al 18 & charcoal 3% is given in Ref 1, p 152 and also in Ref 2, p A290, Table 1. Its deton velocity was 4580 m/sec. It was used for demolition purposes.
Ref: 1) Marshall **3** (1932), pp 117 & 152
2) Fedoroff & Sheffield, PATR **2700**, Vol I (1960)

Engraving (in Ordnance). Process by which the rotating band of a projectile (or jacket of a bullet) is cut and formed by the rifling of the gun bore, thus producing the lands and grooves on the band or jacket (Ref, p 107)

This must not be confused with *body engraving* which is scoring of the body of a projectile made by the gun bore. Such scoring when it appears is usually on one side of the projectile only, and indicates that the projectile is bearing with excessive force against the same land or lands throughout its travel down the bore. It may signify eccentric conditions in the ammunition or other defects
Ref: Glossary of Ord (1959), p 107 (Engraving); p 40 (Body engraving); p 40 (Body); p 167 (Land); p 245 (Rifling); p 31 (Band, rotating)

Enheptin. See 2-Amino-5-nitrothiazole in Vol **I**, p A263-R

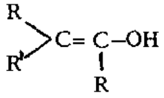
ENIAC (Electronic Numerical Integrator and Computer). The first electronic digital computer which was developed for the US Army Ordnance Corps, to compute WW II ballistic firing tables. An early model is now obsolete
Refs: 1) Glossary of Ordn (1959), 107
2) M.H. Weik, Ordn **45**, 571-75 (1961)
3) See also COMPUTERS in Vol 4, p D176

Ennayaku. A Japanese expl of light-yel color developed before WW II: K chlorate 80, MNT 15 & castor oil 5%. Used during WWII as a bursting chge, probably for grenades and mortor shells. (Compare with Entoyaku)
Refs: 1) OpNav **30-3M** (1945), p 28
2) R. P. Antonneli "Encyclopedia of Explosives", Ordn Liaison Group-Durham NC (1960), p 54

Enneanitrocellulose or Enneacellulose Nitrate, $C_{24}H_{31}O_{11}(ONO_2)_9$. See Enneanitrate in Table under CELLULOSE NITRATES OR NITROCELLULOSES on p C100-R of Encycl, Vol **2**

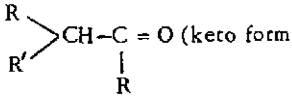
Enneaheptite, Anhydro-Pentanitrate, called in CA **2,2,4,4-Tetrakis (hydroxymethylnitrate)-1-pyranol.** See under Anhydroenneaheptilol and Derivatives in Vol **1** of Encycl, p A404

Enols are weakly acid compds of the general formula:



$$\begin{matrix}R \\ R'\end{matrix}\!\!>C=\underset{\displaystyle R}{\underset{|}{C}}-OH$$

which are tautomeric forms of a substance of the general type



$$\begin{matrix}R \\ R'\end{matrix}\!\!>CH-\underset{\displaystyle R}{\underset{|}{C}}=O$$ (keto form)

Enols form by tautomerism from the keto form by migration of hydrogen atom from the adjacent carbon atom to the carbonyl group (Ref 1)

The quantitative determination of **Enols** can be made either using an esterification procedure with acetic anhydride (Ref 2, pp 455-59), or by direct titration in nonaqueous media (Ref 2, pp 461-62)

Refs: 1) "The Van Nostrand Chemical Dictionery", NY (1953), 265-L 2) Std Methods Chem Analysis **2A** (1963), pp 455-59 & 461-62

Entac (Engin téléguidé anti-char) (Fr for Teleguided Antitank Device). An infantry guided missile and its launcher developed in France prior to 1959 and then used by the US Army
Ref: Anon, Ordn **50**, No 276, 630(1966)

Entac. (Engin Tactique Anti-char). A French surface-to-surface missile. Length 2.72' wt 26.46 lbs, range about 1 mile. Solid propellant powered, wire guided. Can be fired from launchers anchored to the ground, from triple gun carriages or from jeeps
Ref: F. I. Ordway & R.C. Wakeford, International Missile and Spacecraft Guide, McGraw-Hill, N. Y. (1960)

Entflammungsprobe or **Entflammungstemperature Probe.** Ger for Flash- or Kindling-Temperature Test, described in the book of H. Brunswig, "Das rauchlose Pulver", W. de Gruyter, Berlin (1926), 304

Enthalpy. The sum of the internal and pressure energies of a substance or system. Formerly called *Total Heat* or *Heat Content.* Change in enthalpy is the amount of heat added to, or subtracted from, a substance or system in going from one state to another under constant pressure (Ref 1)

Velicky et al (Ref 2) detd at PicArsn "Enthalpy Change, Heat of Fusion and Specific Heat of Basic Explosives"
Refs: 1) Glossary of Ordn (1959), p 107-R
2) R. Velicky et al, PATR **2504**, Jan 1959(U); Project TA3-5000B, Item C. Project 5A04-10-006

Enthalpy Changes, Heat of Fusion and Specific Heat of Basic Explosives are discussed by C. Lenchitz, W. Beach & R. Velicky in PATR **2504**, January 1959

Enthalpy Excess in Detonation (and Explosion). See Vol **4** of Encycl, p D267-L

Entkupferungsmittel. Ger for Decoppering Agent, such as described in Vol **3** of Encycl, p D36-R

Entlastungszünder. Ger *Antilifting Igniter,* **EZ44**, of WWII. It consisted of a flat cylindrical upper casing, base plate, clockwork and strike mechanism, and an explosive filling. After winding the clockwork mechanism, the EZ44 was placed under a landmine or other expl device and the arming bar (See Fig E30) was pulled out by means of a cord or wire attached to the ring. When released, the clockwork, which ran only for 35–40 seconds, gradually forced the safety pin ring outwards, thus withdrawing the pin. The striker was now retarded by means of a catch (release sear), which in turn was held in place by the compressed spring of the release button. Removal of the weight from the release button of the igniter allowed the striker spring to force up the sear by means of the beveled stop, thus releasing the striker to hit the percussion cap of the mine
Refs: 1) TM **9-1985-2**(1953), 318–19
2) PATR **2510**(1958), p Ger 99-L

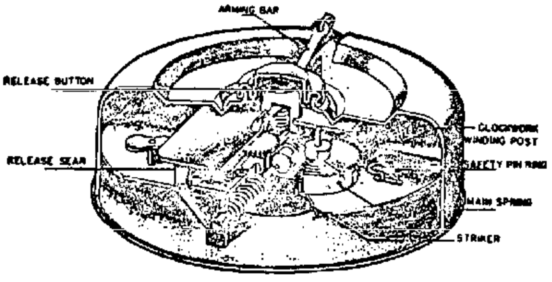


FIG E30 ENTLASTUNGSZÜNDER

Entoyaku. A Japanese explosive of lt brown color used during WWII as a substitute demolition charge or as a bursting charge in hand grenades and mortar shells. Its compn was: $KClO_3$ 80, MNT 16 & castor oil 4% (Compare with Ennayaku)
Refs: 1) OpNav **30–3M**(1945), p 28
2) R.P. Antonneli, "Encyclopedia of Explosives", OrdnLiaisonGroup-Durham, NC(1960) p 55

Entropy. The unavailable energy of a substance which is due to the internal (irregular and compensating) motion of the molecules. It is used internally and rarely enters directly into calcns, but rather in the form of its increments or changes. Entropy is arrived at in thermodynamics in the form of the conception of a change in the entropy of the system, which is equal to the heat taken up during each infinitesimal change of a reversible or *isothermal* process, divided by the temperature at which it is absorbed. For the entire change in the system, the change in entropy is equal to the summation of the infinitesimal terms as denoted by the equation:

$$\Delta S = \Sigma\, q/T$$

where ΔS is the change in entropy and $\Sigma\, q/T$ is the sum of the terms obtd by dividing the infinitesimal amts of heat by the temperatures at which they are gained or lost

At absolute zero the entropy of the system vanishes. It tends to the maximum when free (or available) energy tends to a minimum; at equilibrium of a reaction, the entropy is at maximum

Gibbs defined entropy as "the state of molecular chaos of a system", while Tolman defined it as "the extent to which an energy system has run-down"
Refs: 1) Hackh'sDict(1944), 308-R
2) VanNostrandChemist'sDictionary, NY (1953), 205-R

Entwässerung (Trocknung). Ger for Drying

Entzündlichkeit. Ger for Inflammability or Flammability

Entzündungsgemisch. Ger for Ignition (or Igniter) Mixture

Entzündungsprobe. Ger for Ignition Test

Entzündungspunkt or **Verpuffungstemperatur.** Ger for Ignition Point or Deflagration Temperature

Entzündungstemperatur. See Entzündungspunkt

Envelope of Explosive Charge. Same as Sheath. See under Enveloped (Blanketed, or Covered) Explosives

Enveloped (Blanketed, or Covered) Explosives, known in GtBritain and the USA as **Sheathed Explosives.** In order to render explosives absolutely safe for use in gaseous and dusty coal mines, E. Lemaire of Belgium proposed in 1914 to enclose cartridges of Dynamites (already accepted as safe on the basis of gallerly tests), by a mantle of an inert material, such as Ca fluoride (See Ref 1). This envelope is known in Belgium and France as **gaine** and expls enclosed in it **explosifs gainés** to distinguish them from the *explosifs non gainés* (Vol **3** of Encycl, p C450-L)

A list of Belgian "explosifs gainés" of ca 1960, as communicated to us by Dr L. Deffet and the late Dr F. Lebrun, is given in Vol **2** of Encycl, p B29-R

After the invention of Lemaire, the action of safety sheaths was widely investigated not only in Belgium but also in France, England, the USA, Italy, Russia and Spain. It seems probable that the finely dispersed material of the sheath produced on expln of enclosed charge of Dynamite (or of other explosive) functions as a flame trap in firedamp-coal dust-air mixture, thus preventing propagation of any flame or ignition (Ref 2, p 22)

Sheathed Explosives were introduced in GtBritain in 1934, after many sheathing materials were tried and rejected. Finally it was decided to use Na bicarbonate (or in some cases Na chloride) sheaths (Ref 5, pp 30–1). Typical Brit "permitted" expls which are sheathed are listed in Ref 2, pp 20–1, Ref 5, pp 103–05 and in the Tables given in Vol 3 of Encycl, pp C452 & C453. Examples: *Douglas Powder, Polar Ajax, Polar Dynobel No 2, Polar Viking, Al Rounkol, Polar Thames, Denaby Powder No 2* and *Gathurst Powder*

Under the term *esplosivi con guaina* or *esplosivi inguainati* are known as Italian enveloped or sheathed explosives (Refs 3 & 7). Giorgio (Ref 8, p 176) calls them "esplosivi quelli avvolti in un mantello di sali refrigeranti" (Explosives which are wrapped up in a mantel of cooling salts). Accdg to Belgrano (Ref 3, p 167) and Vettori (Ref 7), the materials of Ital sheaths have been Ca fluoride and Ca sulfate or Na bicarbonate. These expls found wide use in sulfur mines of Sicily

Under the term *Vzryvchatyiye veshchestva v predokhranitel'noy obolochke* (Explosive substances in protective envelope), sheathed expls have been used in Russia. There are two types of sheaths: "inert sheath" which consists of Na bicarbonate with or without Na chloride and "active sheath" which consists of Na bicarbonate with ca 10% NG incorporated. The active sheath has the advantage of being pulverized completely on expln of enclosed cartridge, thus filling the mine atmosphere uniformly with noncombustible dust (Ref 6, p 514)

Under the term *cartuchos con corteza de seguridad* (cartridges with safety crust) are known Spanish sheathed expls. They consist of a cartridge of permissible expl, 25mm in diam, surrounded by a 3.5mm thick envelope of mixt of Na bicarbonate with 15–18% NG (Ref 4, p 119)

United States sheathed explosives are not listed in the DuPont's Blasters' Handbook (1966), but are in other books listed in Vol 3 of Encycl, pp C455 to C459

It should be noted that a sheathed expl is safe against firedamp and/or coal dust as long as the sheath is not damaged, as could happen if the sheathed cartridge is driven into a borehole which is too tight. If the sheath is damaged, the cartridge must be discarded. In order to have expls which are safe under any circumstances, it was decided to incorporate cooling agents (AN, $NaNO_3$, NaCl, Ca sulfate) in expl itself. Such expls are known as **EqS** (qv)

Refs: 1) E. Lemaire, AnnMinesBelg **23**, 649 (1922) 2) Taylor (1952), 17–22 3) Belgrano (1952), 167 4) A. Stettbacher, "Pólvoras y Explosivos", BsAires (1952), 119 5) Taylor & Gay (1958), 30–1 & 103–05 6) Andreev & Belyaev (1960), 514 7) Dr Omero Vettori, Aulla (MassaCarrara), Italy; private communication (1964) 8) Giorgio (1964), 176 (Esplosivi in un mantello di sali refrigeranti)

Enzian E-4. A German anti-aircraft rocket which appeared toward the end of the war but quantity production was never realized. Length 1195', dia 35.4", range 18 mi, alt 9, vel 600 mph. Solid propellant.
Ref: F.I. Ordway & R.C. Wakeford. "International Missile and Spacecraft Guide", McGraw-Hill, N.Y. 1960, Surface-to-Air Missiles/91

Enzian Rakete. Ger Guided Rocket of WWII. See under Guided Missiles

EODT (US Army). Abbrn for "Explosive Ordnance Disposal Teams", formerly known as BDS-Bomb Disposal Squads

EPF (Emery Paper Figure). Brit test for detn of friction sensitiveness of explosives. See under Friction Sensitiveness Determinations in Vol **6** of Encycl

epi. An abbreviation for epichlorohydrin; also a prefix denoting a bridge of intermolecular connection
Ref: CondChemDict (1961), 443-R

Epichlorohydrin and Derivatives

α-Epichlorohydrin (epi) Chloropropylene Oxide, or **1-Chloro-2,3-epoxypropane,**
$Cl.CH_2.\underbrace{CH.CH_2.O}$; mw 92.51; liquid, less volatile than alc, eth, or acet, with chlf-like odor; fr p −58.1°, bp 115.2°, sp gr 1.1761 at 20/20°, vapor pressure 12.5mm at 20°,

viscosity 1.12 cps at 20°, n_D 1.4358 at 25°, miscible with alc, ether and most other org solvents; immiscible with water and petroleum hydrocarbons. It can be prepd by reacting 1,3-dichloro-2-propanol with NaOH at 12–15° or with $Ca(OH)_2$ & water at 20° (Ref 1)

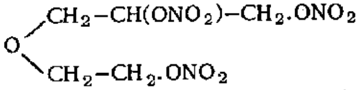
Accdg to Daniel (Ref 2), it was proposed by Dr Fleming as a solvent-plasticizer for NC's used in commercial expls. It has also been used in manuf of "Epon" & "Epoxy" Resins (qv) and as a solvent for cellulose esters & ethers, as well as for gums, resins, etc (Ref 4)

Nichols in a letter to the Editor of Chemical Industries (Ref 3) advised that epichlorohydrin must be handled very carefully because the incident described below showed how toxic and dangerous the substance is. The incident took place when 10% by volume of epichlorohydrin was added to bromoform, the former serving both as an inhibitor and a thinner for the mixture. As a result of overheating, the two men who conducted this operation suffered severe burns to their hands, although they wore light surgeon's rubber gloves. They also suffered from irritation to their eyes and throat, caused by toxic fumes evolved during mixing

Nichols advised to provide good ventilation in the laboratory, to wear something better than rubber gloves, and to remove promptly all clothing wet with epichlorohydrin; also to wash the hands with soap and water immediately after handling epichlorohydrin

Refs: 1) Beil **17**, 6, (4) & [13] 2) Daniels Dict (1902), 301 (Under Fleming) 3) J.E. Nichols, Reynolds Metals Co, Richmond, Va, Chemical Industries, **67**(No 2), p 158, Aug 1950 4) CondChemDict (1961), pp 443-R & 444-L

Epichlorohydrin, Nitrated Derivative. Desseigne (Ref 3) prepd *Hydroxyethyl-3-oxy-1,2-propanediol Trinitrate,*



$$O\begin{cases}CH_2-CH(ONO_2)-CH_2.ONO_2\\CH_2-CH_2.ONO_2\end{cases}$$

mw 271.14, N 15.50%, by reacting epichlorohydrin & glycol and nitrating the product with mixed acid (HNO_3 48.8, H_2SO_4 48.6 & H_2O 2.6%) at 20° for 20 mins. The Trinitrate has d 1.4839 at 15°, n_D 1.4690 at 20°, heat of combstn 2405cal/g at const vol & corrected to 18° (Ref 2), and its volatility at 80° is betwn Ethylenenitramine Nitrate & Ethylene Dinitramine, being considerably less volatile than NG

This new expl forms a homogeneous, stable emulsion with NC. Desseigne patented this compd as an expl & gelatinizing agent (Ref 3)

Refs: 1) Beil, not found 2) L. Médard & M. Thomas, MP **37**, 135(1955) & CA **51**, 716 (1957) 3) G. Desseigne, FrP 1127647(1956) & CA **53**, 16970(1959) 4) G. Desseigne, MP **39**, 181–90(1957) & CA **52**, 21107(1958)

Epichlorohydrin Perchlorate (called α-Überchlorsäureester des-γ-chlor-propylenglykol in Ger), $Cl.CH_2.CH(OH).CH_2ClO_4$; mw 192.99; viscous sl fragrant oil, deflgr when heated in a flame and explodes violently on impact. Its expl effect is greater than that of NG. The perchlorate is readily hydrolyzed in the presence of moisture at RT. It was prepd by reaction of α-eipchlorohydrin in ether with 60% perchloric acid at 0–5° (Refs)

Refs: 1) Beil **1**, 474 2) K.A. Hofmann et al, Ber **42**, 4390(1909) & CA **4**, 584(1910) 3) Urbański **2**(1965), 448

Epichlorohydrin Resins. See under "Epon" Resins and under "Epoxy" Resins

EPOMA. Abbr for The Explosive and Pyrotechnic Ordnance Manufacturers Association, 612 Oakmont Ave, Erie, Pa, 16505

"Eponite" 100. Shell Chemical Co trademark for a water-dispersible liquid epoxy resin used in textile industry to impart crease resistance, shrinkage, etc

Ref: CondChemDict(1961), 444-R

"Epon" Resins. Trademark of Shell Chemical Co, New York 20, NY for condensation products of epichlorohydrin and *bisphenol A* [which is para, para'-isopropylidinediphenol, $(CH_3)_2C(C_6H_4OH)_2$, described on p 156-R of Ref 1], having excellent adhesion, strength, and electrical properties when formulated into protective coatings, adhesives and structural plastics

The following "Epon" Resins have been proposed for use in US propellants, as indicated in conf Ref 3:

"**Epon 562**" – for use as a binder-fuel in rocket propellant, Unit No 544, or as a curing agent in propellant Unit No 603

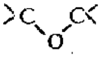
"**Epon 812**" – for use as curing agent in propellants Unit Numbers 611, 630 & 632

"**Epon 828**" – is a low molecular wt resin widely used in adhesives, castings, laminates, potting compds & solvent free coatings. Lenchitz et al (Ref 2) determined the heat of polymerization of Epon 828 and of its mixts with ethylenediamine (EDA), diethylenetriamine (DETA) & triethylenetetramine (TETA)

Refs: 1) CondChemDict (1961), 156-R & 444 2) C. Lenchitz, R. Brown & S. Stivala, PATR **2505**(June 1958) 3) PropIntManual **SPIA/M2** (1962)

Époussetage (French for Dust Removal). The removal of dusty material from the grains of BkPdr or smokeless propellants was effected in France by shaking the grains on a fine screen

Ref: Pepin Lehalleur (1935), 284

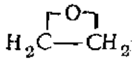
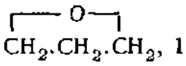
Epoxides are compds incorporating a three-membered ring contg one oxygen atom and two carbon atoms,  $>C\underset{O}{\diagdown\diagup}C<$

Epoxides have a great affinity for labile hydrogen, a fact which permits them to enter into a remarkable variety of useful reactions. They are susceptible to attack by almost all known nucleophilic reagents (Ref 2). One of the epoxide resins was used as a fuel in rocket propellant, Unit No 512, described in conf SPIA/M2 (1962)

Pujo & Boileau (Ref 1) studied the action of nitrogen peroxide on epoxides and found that the reaction did not yield nitro derivs, as expected, but nitrate-nitrite esters; methanolysis converted the esters to alcohol nitrates. Nitration with sulfuric-nitric acids (50/50) yielded dinitrate derivs and with mixed acid (62.24% nitric acid) the dialcohol dinitrate deriv

Refs: 1) Anne-Marie Pujo & J. Boileau, CR **237**, 1422–24(1953) & CA **49**, 853(1955); MP **37**, 35–48(1955) 2) Kirk & Othmer, 2nd edit **8**(1965), 263–93 (M. Lapkin, Epoxides)

Epoxies. A name sometimes given to Epoxy Resins (qv)

Epoxy. A prefix in organic nomenclature denoting an oxygen atom joined to each of two atoms which are already united in some other way, as  $H_2\overset{\ulcorner O \urcorner}{C—C}H_2$ epoxyethane and  $\overset{\ulcorner\!—\!—\, O \,—\!\urcorner}{CH_2.CH_2.CH_2}$, 1,3-epoxypropane

Refs: 1) Hackh'sDict(1944), 310-L 2) CondChemDict(1961), 445-L

2,3-Epoxy-2-ethylhexanol,

$C_3H_7\overline{CHOC}(C_2H_5)CH_2OH$, mw 144.21, liquid, sp gr 0.9517 at 20/20°, fr p -65°, bp – decomposes; sl sol in w. Its method of prepn was not found in the literature

Used as a stabilizer and as intermediate compd.

Refs: 1) Beil, not found 2) CondChemDict (1961), 445-L

2,3-Epoxy-1-propanol or Glycidol,

$\overline{CH_2OC}HCH_2OH$, mw 74.08, colorless liq, bp 162°; sol in w, alc or eth. It can be obtd by treatment of monochlorohydrins with bases and by other methods (Ref 1)

Used as stabilizer for natural oils and vinyl polymers and also as demulsifier (Ref 2)

Refs: 1) Beil **17**, 104, (50) & [104] 2) CondChemDict (1961), 542-R (Glycidol)

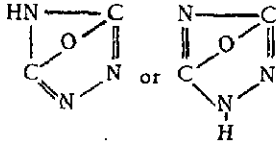
Epoxy Resins or Epoxies. The commercially available materials of this class are usually derived from epichlorohydrin and *bisphenol A* (See under Epon Resins). Before curing they are viscous liquids or clear, brittle solids of mw 400 to 8000, melting up to 155°. Chemical nature is indicated by the formula, $H_2\underbrace{COC}HCH_2O(RCHOHCH_2O)_nROCH_2\underbrace{CHOC}H_2$, where $R = C_6H_4C(CH_3)_2C_6H_4$. Variations from this basic compn are produced by the ratio of the reactants, the conditions of reaction and by the catalyst or agent and conditions used for their curing

Bisphenol A may be replaced in prepn of epoxies by other diphenols, glycols or glycerine, but the resulting resins are of limited utility. The term "epoxy plastic" is also sometimes used for other types of high mol wt compds that contain the epoxy group $-\underbrace{C-O-C}-$, or are derived from such compds. Thus "*Carbowax*" (which is trademark of Union Carbide Corp, New York 10017, NY for polyethylene glycols or methoxypolyethylene glycols, described in Ref 3, p 221-R) is sometimes designated as an epoxy compound

Epoxies show superior adhesion to metals and glass, and have limited shrinkage during cure. They are used for surface coatings, as adhesives and or laminating to produce plastic tanks, pipe, and aircraft parts, etc (Ref 3, p 445-R). They also have been used in manuf of plastics (Ref 1), construction of rockets & missiles (Ref 2) and in manuf of explosive compositions of improved mechanical and thermal stability (Ref 4)

Refs: 1) Irving Skeist & G.R. Somerville, "Epoxy Resins", Reinhold, NY (1958) 2) A.J. Zaehringer & R.M. Nolan, Missiles & Rockets, March 1958, p 69 3) CondChemDict (1961), 221-R (Carbowax); 445 (Epoxy Resins) 4) V. Jamieson, **RARDE** (RoyalArmamentResearch & DevelopmentEstablishment) **Memo(X)20/63**, "Explosive Compositions of Improved Mechanical and Thermal Stability", Part 1, "Polyurethane and Epoxy Systems", GreatBritain, Apr 1963 (Conf) (Not used as a source of information) (PicArsn C104439) 5) Kirk & Othmer, 2nd edit **8** (1965) 294–312 (J.R. Weschler, "Epoxy Resins") 6) H. Lee & K. Neville, "Handbook of Epoxy Resins", McGraw-Hill, NY (1966), 918pp

Epoxytriazole (called 3.5-Endoxy-1.2.4-triazol in Ger),



; mw 84.05, N 50.60%, crysts (from w), mp 250°; insol in w & alkalies. It was obtd with other substances when urea reacted with 4-phenylthiosemicarbazide, 4-p-tolylthiosemicarbazide or 4-(2,4-dimethylphenyl)-thiosemicarbazide at 125-40° (Refs 1, 2, 3 & 4)

No expl props are reported

Refs: 1) Beil **27**, [926] 2) P.C. Guha & P.C. Sen, QuartJIndianChemSoc **4**, 44–50 (1927) & CA **21**, 2900 (1927) 3) S.M. Mistry & P.C. Guha, JIndianChemSoc **7**, 793 (1930) & CA **25**, 1504 (1931) 4) S.L. Janniah & P.C. Guha, JIndianInstSci **16A**, 11 (1933) & CA **27**, 3711 (1933) 5) Not found in later refs thru 1966

Épreuve ou Essai. French for Test, Experiment, Assaying or Essay

Note: For reasons unknown to us, many tests, known generally as Epreuves are also called Essais in some books. These tests we marked as Épreuve (ou Essai) and those which are not called Essais, we marked just by the word Épreuve. The tests known as Essais are placed as such further down and they are not listed under Épreuve (See also under "Essais d'Explosifs" which are French Official Tests)

Épreuve d'Abel ou Épreuve anglaise. Abel Test or English Test. See Vol **1** of Encycl, p A2-L

Épreuve allemande. See 132° German Test in Vol **1**, p XV, known in Ger as "Erhitzungsprüfung bei 132°". It has also been conducted at 134.5°

Épreuve (ou Essai) au bloc de plomb de Trauzl, also known as **Cavité au bloc de Trauzl.** The international method of conducting this test is described in Vol **1**, p XXV, under Trauzl Test or Lead Block Expansion Test (Trauzlsche

Probe oder Bleiblockausbachung Methode in Ger). The French method of conducting the test is slightly different and is described as *Coefficient d'utillisation pratique* (abbr *CUP*) *ou Épreuve de travail spécifique* in Vol 1, pp IX & X and in Vol 3, p C390. Official test of the French Army is given as "Travail spécifique relatif, test 13", under Essais d'Explosifs

Épreuve à la bombe ou Essai de poudre en vase clos. (Test in the Bomb or Test of Propellant in a Closed Vessel) (Similar methods are called in English "Closed Bomb Tests", and they are described in Vol 3, pp C330 to C345 of Encycl. The French method is described in detail in the book of instructions issued in about 1950 by "La Commission des Substances Explosives", 4ème partie, articles 189–210, which could be obtd on request from the Services des Poudres, thru the État-Major de l'Armée, 2ème Bureau, Paris, France

The purpose of this test is to determine the burning characteristics of a propellant in a closed vessel. The test gives two ballistic characteristics: relative force and relative quickness, designated by the French as la force and la vivacité de poudre. The apparatus consists of a thick, vertical, steel cylindrical vessel of either 22 or 150cc capacity, closed at the bottom by a threaded plug and provided at the top with a steel cover thru which two electrical wires pass, serving to ignite the charge inside the vessel. After inserting a pressure gauge inside the cylinder, a charge of propellant is added, the cover is closed and the charge is ignited electrically

Épreuve (ou Essai) de chauffage de la gouttière ou Épreuve de propagation dans une gouttière. See Combustion en gouttière de 20mm, described as Test 5 under Essais d'Explosifs

Épreuve (ou Essai) au choc des balles. See Bullet Impact Sensitiveness Test in Vol 1, p IX giving four French references describing French Test. A detailed description of test such as developed after WWII at the Laboratoire des Substances Explosives is given in Vol 2 of Encycl, pp B333-R and B334-L. This test is considered more rigid than three US tests A, B and C described on pp B332 & B333 of Vol 2

Épreuve (ou Essai) de combustion en tas conique ou Épreuve de la sensibilité a l'inflammation. See Test 6 under Essais d'Explosifs

Épreuve a la dynamite de pilotis (Fr for the Test of Dynamite by Means of Piles). The test, first conducted at Budapest in 1881 by Lt Colonel Prodonóvic, consisted in placing on top of several piles (stakes) iron plates with charges of various Dynamites of the same weights. One of the charges served as a standard and the ensemble was covered by sand. The charges were detonated simultaneously by means of electric blasting caps and the depth of penetration of each pile was measured and compared with that produced by the standard. The work in kilogram-meters developed by the standard Dynamite was previously evaluated by other methods. It was claimed that this method gave fairly accurate results if the number of tests was sufficiently large

Ref: Daniel, Dict (1902), 282 & 632–33

Épreuve de finesse des coton-poudres (Fineness Test for Nitrocelluloses). The test as conducted accdg to the instructions of La Commission des Substances Explosives, Chapter V, Article 20 is as follows:

Procedure:

a) Weigh accurately 10g of pulped NC, previously dried to constant weight at 100°

b) Transfer the sample to a mortar and add distilled water, mixing to a semi-liquid paste. Transfer the paste to a 250ml graduate, 35 to 40mm in diameter and without a lip. Rinse the mortar and pestle with several portions of water from a wash bottle and use the rinsings to fill the graduate to about 125ml. Close with a rubber stopper and shake

c) Rinse the walls of the graduate and fill it to slightly below the 250ml mark. Shake again and rinse the upper part of the graduate with a sufficient amount of water to fill to the 250ml mark

d) Place the graduate on a surface not subject to vibrations and note the volume occupied by the NC after exactly one hour of standing. The finer the pulping, the smaller is the volume

Épreuve des petits plombs ou Épreuve aux blocs de Hess (Small Lead Blocks Test or Blocks of Hess Test). This test, known in the USA as Lead Block Compression Test (LBCT) or Hess' Crusher Test (Stauchprobe nach Hess, in Ger) was invented in Austria by Hess. It is one of the Compression (or Crusher)Tests for Determination of Brisance, described in Vol 3 of Encycl, pp C492 to C494. Its French modification, designed by La Commission des Substances Explosives, is listed on p C493-L, but not described. The test has been conducted accdg to Vennin, Burlot and Lécorché (1932), pp 190–93 as follows:

Procedure:

a) Compress 45g of explosive in a cylindrical sheet-iron can (40mm diameter, 0.5mm wall thickness) to the prescribed density

b) Attach the bottom of the can to a steel disc 40mm diameter and 5.8mm thick and place the ensemble on top of a lead cylindrical block, 40mm diameter and 50mm high, resting on a steel base. The sides of the lead cylinder are marked by circles parallel to the base and 5mm distant from each other. (Compare with Fig on p C492 of Vol 3 of Encycl)

c) Provide the upper part of the expl with a No 7 blasting cap (1.5g MF) and fire

d) As result of the crushing action of the explosive, the upper part of the lead block will be compressed, while the lower part will remain practically intact. This will give the block a mushroom appearance

e) Measure the crushed block, as prescribed by the Commission des Substances Explosives and compare the resulting diminution of height in mm (considered as brisance) with that of a standard explosive such as PA. (Compare the test with Esop's Test described in Vol 1, p XI)

The so-called *Quinan Test* permits one to determine simultaneously the brisance and the work performed on detonation. This value is known in France as "potentiel" and may be considered as being approximately proportional to power. A brief discussion of the test is given in Vol 1, p XXI, while in Vol 3, p C494-L is reproduced the Fig of apparatus

Following is the description of test as given by Vennin, Burlot & Lécorché (1932), p 192 and by Pepin Lehalleur (1935), p 63

Procedure:

a) Arrange the apparatus as shown in Fig on p C494-L of Vol 3 of Encycl and have 1 or 2g sample of explosive, provided with 0.25g priming charge, placed in the cavity, F, of solid steel cylinder, C, 100mm in diam and 128mm high

b) Cylinder C is resting on top of solid lead cylinder, A, 25mm in diam and 25mm high, which, in turn is placed on steel plate, B, 25mm resting on a cast-iron base, E, embedded in cement

c) On top of C is placed a solid steel cylinder, D, 100mm in diam, and 254mm high. A small hole, H, is drilled thru the center of D to allow a fuse or electric wires to pass to the priming chge of explosive, placed in F

d) When the charge in F is exploded, the resulting gases push cylinder, C, downwards, while cylinder, D, is pushed upwards. Both cylinders slide on vertical rods, G, especially provided for this purpose. The upper part of G is provided with an arrangement to arrest D in the highest position reached on expln

e) The downward movement of C compresses (crushes) the lead cylinder, A, diminishing its height, and the extent of this shortening of cylinder is taken as the value of brisance, in mm

f) The simultaneous pressure of explosive gases on cylinder, D, causes its movement upwards and the arrest by a special catch at the maximum height reached. This height, expressed in mm, serves as a measure of work performed by the explosive

There is also the so-called *Copper Cylinder Compression Test (CCCT)*, known also as *Kast's Crusher Test* (Stauchprobe nach Kast, in Ger) which is described under Compression Tests in Vol 3, p C491, but it does not seem that this test is used in France

Épreuve (ou Essai) au chock du mouton (Fr for Shock Test by Ram). See Vol **1**, p XVII, under Impact Sensitivity

Épreuve du premier rouge (Test to the First Red). See under Resistance to Heat Test (Épreuve de la résistance à la chaleur) in Vol **1** of Encycl, p XXI

Épreuve de Quinan. See under Épreuve de petits plombs

Épreuve (ou Essai) de résistance a la chaleur. See Resistance to Heat Test in Vol **1** of Encycl, p XXI and also in Daniel, Dict(1902), pp 672–81

Épreuve (ou Essai) de résistance à l'exsudation (Fr for Resistance to Exudation Test). One of the Fr methods is described in Vol **1**, of Encycl, under "Exudation (or Sweating) Test, p XI. Another test is in Daniel, Dict(1902), pp 293, under Exsudation and the 3rd test in Pepin Lehalleur (1935), 61

Épreuve (ou Essai) à la résistance totalissée (Test for Total Resistance). See under Resistance to Heat Test, Vol **1**, p XXI

Épreuves de sécurité. Under this title, M. Dutour lists in MP **31**, 74(1949) the following tests:

a) *Sensibilité au choc* (Sensitivity to Shock or Impact). See Tests 7 and 8, under Essais d'Explosifs

b) *Coefficient de self-excitation* (Coefficient of Self-excitation), described as Transmission de la détonation à distance (Transmission of Detonation at a Distance), Test 12, under Essais d'Explosifs. This test is known in the USA as Gap Test. See Vol 4, p D393-R

c) *Combustion en gouttière de 20mm* (Combustion in a 20mm Trough). See Test 5 under Essais d'Explosifs

d) *Combustion en tas conique* (Combustion in a Conical Pile). See Test 6, under Essais d'Explosifs

Épreuve de sensibilité à l'amorce (Sensitivity to Priming Test). See Test 11, under Essais d'Explosifs

Épreuve de sensibilité à l'inflammation (Sensitivity to Ignition Test). See Combustion en tas conique, Test 6 under Essais d'Explosifs

Épreuves de stabilité des explosifs et des poudres (Stability Tests for Explosives and Propellants). A list of Thermal Stability Tests is given under Heat Tests in Vol **1** of Encycl, p XV. As example of tests used in France may be cited:

a) *Épreuve d'Abel* described in Ref 1, p 391 and Ref 4, p 107

b) *Épreuve Bergmann-Yunck* described in Ref 1, pp 395–97. This, originally German Test, is described as Bergmann-Junk Test in Vol **2** of Encycl, pp B102-R & B103-L

c) *Épreuve Desmaroux* described in Ref 5, p 90

d) *Épreuve Grotannelli* described in Ref 3 and in Ref 4, p 108

e) *Épreuve Leclercq* described in Ref 7

f) *Épreuve Marqueyrol* described in Ref 5, p 90

g) *Épreuve Rouvillois* described in Ref 6

h) *Épreuve Taliani* described in Vol **1** of Encycl, pp XXIV & XXV and

i) *Épreuve Taliani modifiée* described in Ref 1, pp 397–98 and Ref 2, p 109

j) *Épreuve Vieille ou Epreuve dite a 110°* described in Ref 1, pp 392–93, in Ref 4, p 107 and Ref 5, p 78

k) *Épreuve Vieille dite du coefficient d'échange* described in Ref 1, pp 399–400 and also as Coefficient d'échange in Vol **3** of Encycl, p C389-L

Refs: 1) Vennin, Burlot & Lécorché(1932), pp 391-400 2) P. Vieille, MP **26**, 7–14 (1934) 3) F. Grotannelli, MAF **14**, 2ème fascicule (1935) 4) Pepin Lehalleur (1935), pp 107–08 5) Reilly (1938), pp 78 & 90 6) L. Rouvillois, MAF **17**, 773–97(1938) (Evaluation of stability of propellants by pH determinations) 7) R. Leclercq, MAF **25**, 635–50(1951) (Evaluation of stability of propellants by determination of the amounts of nitrogen oxides evolved on heating)

EqS Explosives (Equivalent in Safety to Sheathed Explosives) (British). As was noted under the description of Enveloped or Sheathed Explosives, the EqS were developed in order to have expls which are safe under any circumstances. They were developed by J. Taylor and proved such a success that they replaced Sheathed Explosives in many cases. As was already mentioned they were prepd by incorporating a cooling agent in expl itself, instead of using a shield. Compositions of the following EqS Explosives: Unirend, Unigex (Initial), Unigex (Modified), Unigel, Unibel, Unifrax and Unikol are given in Refs

Refs: 1) J. Taylor, TransMiningEngrs(London), **109**, 358(1949–50) 2) W. Shepherd & H. Grimshaw, Ibid, **109**, 889(1949–50)
3) Taylor & Gay(1958), 31 & 86–106
4) Fedoroff & Sheffield, Encycl of Expls, Vol **3**(1966), Table on pp C452 & C453

Equations of State in Detonation (and Explosion). See Vol **4**, pp D268 to D298. They are listed on pp VIII & IX of Vol **4**

Addnl Refs on Equations of State:
A) A.D. Crow & W.E. Grimshaw, PhilTransRoySoc **230A**, 39–72(1930) (Equation of state of propellant gases)
B) P. Caldirola, JChemPhys **14**, 738–41(1946) & CA **41**, 2245(1947) (Equation of state for gases at extremely high pressures)
C) S.R. Brinkley, Jr, JChemPhys **15**, 113–14 (1947) & CA **41**, 2245(1947) (It is stated that Caldirola's equation is not of sufficient general form for application to the products of detonation of solid expls, since it implies that the gaseous products are thermodynamically ideal)
D) M.A. Cook, JChemPhys **15**, 518–24(1947) & CA **41**, 6047(1947) (Equation of state for gases at extremely high temperatures from the hydrodynamic theory of detonation) (Data calcd by various equations is presented for RDX, PETN, TNT, Tetryl, PA, AmmP, LA & MF and correlated with similar data obtd by other investigators)
E) S. Paterson & J. Davidson, JChemPhys **22**, 150(1954) & CA **48**, 4911(1954) (Equations of state and detonation theory)
F) N.M. Kuznetsov, ZhurFizKhim **35**, 1430–34(1961) & CA **55**, 24011–12(1961) (Equation of state of the products of RDX detonation)
G) J.W. Enig & F.J. Petrone, PhysFluids **9**(2), 398–408(1966) & CA **64**, 12453(1966) (Equation of State and Derived Shock Initiation Criticality Conditions for Liquid Explosives)
H) R.G. McQueen et al, High-Velocity Impact Phenomena **1970**, 293–417 & CA **76**, 17869 (1972) (A review of equation of state of solids from shock wave studies, 52 refs)

Equivalent in Safety to Sheathed Explosives. See EqS Explosives

Equivalent Weight. The equivalent wt of an element is the wt that will combine with or react with or can replace one atomic wt of hydrogen or one half atomic wt of oxygen. A gram-equivalent of a substance is obtd by dividing its formula wt by its valency. With acids it is the number of replaceable H-atoms, with bases the number of OH-groups

Refs: 1) Hackh'sDict(1944), 311-R 2) CondChemDict (1961), 445-R

Equivalent Weight and Volume and Their Precision Indexes for Comparison of Explosives in Air. Data for mean peak pressures and positive impulses determine figures of merit which express performances of expls fired in air. Equivalent Weight (EW) & Equivalent Volume (EV) are easily interpretable by ordnance designers. The EW of a new expl is the ratio of wt of a known expl to the wt of a new expl which gives equiv power as measured by peak pressure or positive impulse. The EV is similarly defined

Two independent means, graphical & relative pressure methods, are used to obtn EW & EV from peak pressure measurements. Two comparable methods obtn these figures of merit from positive impulse data. There is also described a method for detg EW & EV from an analytical study of positive impulse data

It was concluded that within its limitations the relative pressure method is simpler and easier to apply than the graphical method, is less subject to errors in interpretation of graphical data, and extracts the max info from data points

It was also concluded that in detg EW &

EV from positive impulse data, the graphical method should be used in cases where the impulse vs distance curves for the expls being compared have the same slope

The main advantage of the analytical method appeared when the av value of EW or EV was reqd from expls whose impulse-distance slopes were not the same
Ref: J. Maserjian & E.M. Fisher, NAVORD Rept **2264** (Nov 1951)

Ercole Dynamine consisted of NG, K chlorate and a small amt of organic material
Ref: R. Molina, "Esplodenti", Hoepli, Milano (1930), p 285

Erdstuka (Ger). A rocket-assisted, 1800-kg armor-piercing bomb used by Stuka bombers during WWII against land targets. This bomb was listed but not described in TM E9-1983, File No 2324.92, formerly available at Aberdeen Proving Ground Museum, in charge of Col G.B. Jarrett and Mr K.F. Kempf

Erg. The CGS (Centimeter-Gram-Second) unit of work or energy representing force necessary to overcome the resistance of one dyne acting thru one centimeter. One dyne is equal to 1 gram/gravity acceleration in cm per sec per sec, which gives 0.00102g. One joule is equal to 10^7 ergs
Ref: Hackh'sDict(1944), 291-R (Dyne); 312-L (Erg)

Erhardt (Poudres). Three formulations are known: 1) Artillery – $KClO_3$ 33.33, KNO_3 33.33 & tannin or catechu 33.34%
2) Bombs – KNO_3 50 & tannin or catechu 50% 3) Mining – $KClO_3$ 12.5, KNO_3 12.5, charcoal 50.0 & tannin or catechu 25.0%
Ref: L. Gody, "Matières Explosives", Wesmael-Charlier, Namur (Belgium), (1907), p 263

Eriksen's Military Dynamite, developed at Picatinny Arsenal, is described in this Vol, p D1609, under DYNAMITE, Class X and also in PATR **1611** (1946)

ERL Polar PE. An American plastic explosive developed by the Explosives Research Laboratory, Bruceton, Pa. It consisted of RDX (50-70% thru 30 & retained on 200 US Std Sieve and 30-50% thru a 200 US Std Sieve) 88% and Gulf Crown Oil E or Gulf 300 Process Oil [(95%) + Lecithin (5%)] 12%. Its properties resembled the *British PE No 2*, but possessed improved low temperature behavior
Refs: 1) W.E. Campbell Jr & L.H. Brown, "On Literature Survey on Smokeless Propellants", AerojetEngrgCorp Rept No **194** (May 1946); p 20, 2) E.H. Eyster, "The Preparation and Properties of Plastic High Explosives", OSRD Rept **5631** (Jan 1946)

Erlenmeyer, R.A.K.E. (1825–1909). A German chemist noted for organic research. Designer of a conical glass flask with flat bottom, used extensively in chem laboratories
Ref: Hackh'sDict (1944), 313-L

EROSION OF GUN BARRELS [Erosion (ou Usure) des bouches à feu, in Fr; Erosion der Schusswaffen; Bohrabnutzung, oder Bohrausbrennung der Gewehrläufe, in Ger; Erosiya i Razgar Stvola Orudiya, in Rus; Erosiones producidas en las ánimas de cañones, in Span; Erosione (Usura) dei cannoni, in Ital]

Erosion is the breaking down and wearing away of metal in the bore of a weapon (cannon or rifle) either by *mechanical action, erosive burning of propellants* or by *corrosion* (which is briefly discussed in Vol **3** of Encycl, pp C544 & C545-L). Mechanical action is caused by friction of the surface of *rifling lands* and the surface of the *driving(or rotating) band* of a shell or the *jacket of a bullet. Erosive burning* is caused by gas produced on burning of a proplnt, and the higher the temperature of the gases the more burning with formation of brittle FeO is produced. *Corrosion* is due to chemical action of solid and liquid substances present in propellant gases. On being deposited on the surface of the bore, they attack the metal, unless they are removed immediately after each firing. As result of mechanical, burning and corrosive action, the surface of the bore becomes rough and pitted, causing high friction between lands and driving band

[See also definition of erosion given by Kirk & Othmer (Ref 33)]

Historical. Ever since the replacement of smoke-producing BkPdr (Black Powder) (described in Vol **2**, pp B173ff) with smokeless proplnts (which took place in the middle of 1860's) more rapid wear away of gun barrels became noticeable. The damage was the smallest with proplnts based on NC (See Single Base Cannon Propellant in Vol **2**, pp C31-R to C32-L), than with NG-contg proplnts (See Double-Base Cannon Propellants in Vol **2**, pp C33 to C36). The greatest damage was caused by *Ballistite*, invented by A. Nobel and described in Vol **2**, p B8. Less damaging was *Cordite* (developed in England and described in Vol **3**, pp C531ff). It had vaseline acting as a cooling agent incorporated in formulation similar to Ballistite

The first serious experimental investigation of phenomenon of erosion was done ca 1884 by Dr Paul Vieille of France (1854–1934), the inventor of Poudre B, described in Vol **2** of Encycl, p B11 [See also Davis (Ref 26, p 294) and Pérez Ara (Ref 31, p 430)]. Vieille also discovered (Ref 1, p 195 & Ref 26, p 387) that the gases from the explns of NGu (Nitroguanidine) were much less erosive than those from other expls of comparable force, and considered this fact to be in harmony with his general conclusion that the hotter expls are the more erosive. In his experiments the explns were made to take place in a steel bomb, 17.8cc capacity, equipped with a crusher gauge and with removable perforated, steel plug thru the perforation in which the hot gases from the expln were allowed to escape. They swept away, or eroded off, a certain amt of the metal. The plug was weighed before and after the experiment, its density was detd, and the number of cubic millimeters of metal lost was reported as a measure of the erosion. Some of Vieille's results are listed by Davis in the Table on p 388, in which it is shown that erosion (per gram), caused by expln of NGu was 2.3mm, Poudre BF gave 6.4, BkPdr (military) 2.2, BkPdr (sporting) 4.5, Cordite 18.1, Ballistite VF 24.3, NMnt (Nitromannite) 23.6, and BG (Blasting Gelatin) 31.4. Following Vieille's experiments, causes of erosion were investigated by Siwy of Germany (Refs 2 & 4). Bravetta of Italy (Ref 3), Noble of England (quoted from Marshall, Ref 5), Létang and Paquelier of France (Refs 6 & 7), Greaves et al of the USA (Ref 11), McNair of the USA (Ref 12), Krylov of Russia (Ref 8), Piantanida of Italy (Refs 9 & 19), Tchekov (Chekhov) of Russia (Ref 13), Gabeaud and Muraour of France (Refs 15 & 16), Lintès of Rumania (Ref 18), Schwinning of Germany (Ref 20), Poddubnyi of Russia (Ref 21), Hayes of the USA (Ref 22), Costier and Sutterlin of France (Refs 24 & 25), Comer of the USA (Refs 27 & 35), Alvaro-Alberto of Brazil (Ref 28), Gen Gallwitz of Germany (Refs 29 & 30), Daubrée of France and R. Austen of the USA (quoted from Pérez Ara, Ref 31), Ficheroulle & Kovache of France (Ref 34), Green of the USA (Ref 36), Abram et al of GtBritain (Ref 37), Tavernier et al of France (Ref 39), Chernov of Russia (as quoted from Gorst, Ref 44, p 153) and Andreev & Belyaev of Russia (Ref 45). Much work was done on erosion of guns accdg to the late G.D. Clift, in the USA by the Bethlehem Steel Corp, but the most important work was done in Germany, as described in Refs 29 & 30 and discussed here following the "Description of Burning, Corrosion, and Erosion of Gun Barrels"

Description of Burning, Corrosion and Erosion of Gun Barrels. Accdg to Gorst (Ref 43), the first indications of erosive burning are dull spots on the bright, polished surface of the bore, especially in and near the proplnt chamber. These spots consist of an accumulation of thin, shallow cracks in the metal. After further firings these cracks (especially those located in the direction of gun's axis) become longer, deeper and wider, taking shape of canals. The resulting space between the bottom of the canals and of the surface of a projectile driving band allows the escape of propelling gases directly into atmosphere without exerting any pressure on the base of projectile. At the same time, some of the unburnt particles of proplnt escape thru the canals. The formation of cracks is accompanied by wearing away of rifling (lands) of the gun, thus causing enlargement of the diameter of the bore. As the distance betw the surface of the driving band and lands of the bore becomes greater and greater, more gases escape thru the muzzle without exerting any pressure on the projectile. This causes reduction of pressure in the chamber, as well as of muzzle velocity of the projectile. With very excessive wearing away, the gun might lose its ability to rotate the projectile which always results in diminished velocity, shorter range, smaller penetration and loss of precision in firing. It has been customary in Russia to consider a gun as unfit for further service if initial velocity of projectile is diminished 10%

The above described phenomenon, known in the USA as "erosive burning", is called "razgar" in Russia and it is caused, accdg to D.K. Chernov (as quoted from Gorst, Ref 43, p 153), by excessive temperatures developed in the gun during firing. At first, a thin surface layer in the bore is heated and it starts to expand against a colder layer located beneath the surface. The resulting resistance causes a stress evidenced by damage to the surface in the form of cracks, mentioned above. The cracks form easily because the surface of the bore consists of hardened (tempered) steel formed on account of quick cooling of bore by air entering the bore on opening of breech-block after each firing. Propellants contg NG, in addition to NC, cause more excessive damage than those based only on NC, even if the calorific values of proplnts are the same. Proplnts contg colloided NC cause less damage than those contg the same amt of non-colloided NC (Ref 43, p 153)

Experimental procedure for detn of erosive burning of propellants used by Vieille is described in Ref 1, p 195 and briefly by Davis in Ref 26, p 294 and also in this item under "Historical". More recent determination was done by Andreev & Belyaev as described in Ref 45
(See also Refs: 16, 27, 28, 29, 30, 35, 36, 37, 39, 41, 42, 43 and 44)

Erosion ("eroziya", in Russian), or mechanical wearing away of bore of a gun, can also be caused, accdg to Gorst (Ref 43, p 154), by friction of rapidly moving (about 600m/sec) hot propelling gases against the surface of the bore. This results in sweeping away part of the metal on the surface, thus causing the formation of cracks, chipping and the enlargement of the diameter of the bore. The enlargement begins at the proplnt chamber and gradually spreads towards the muzzle of the gun. Erosion by mechanical action of hot gases can be diminished by a device, such as "cardboard obturator", preventing the escape of gases between the rifling of the bore and lands of projectile (Ref 43, p 154). One of such devices was the so-called *prossal'nik*, which can be defined as "greaser" or "lubricator", called Dichtungsdeckel, in German. A better name would be a "lubricating gasket" or "bore lubricator". Accdg to description given in PATR **2145** (Ref 38b), which is an abbrd translation from the books of Blinov (Ref 31b), prossal'nik was used occasionally in separate-loading rounds for 107mm and 122mm cannons with a full chge of proplnt. The device consisted of a cardboard obturator, cardboard covers, a thick ring (consisting of a mixture of red lead, ceresin & vaseline) and a gasket. The device was mounted at the base of projectile just before its loading. It was claimed that on firing, the propelling gases caused the lubricating material to disintegrate and to be spattered inside the bore, thus depositing a protective insulating coating on the bore surface. It was claimed by the Russians that prossal'nik increased the life of a gun (and this may be true), but it decreased muzzle velocity (Ref 38b)

Accdg to experiments conducted in the USA (Refs 38 and 38a), it was not proved that the idea of using a bore-lubricant is unsound. When oil was used in the gasket the muzzle velocity of the first round was practically the same as in the gun previously cleaned and dried, but the velocities of the 2nd, 3rd, etc rounds (using no gaskets) were appreciably lower due to the presence of carbonaceous products of decompn of oil deposited on the rifling of the bore. After firing many more rounds (the number not stated), the velocity started to climb back to that obtd when firing was done without lubricator. It would be interesting to repeat experiments using a heat resistant oil or grease, such as silicone compd (Ref 38b)

A simpler method consisted of wrapping up a complete charge of propellant in a sheet of paper impregnated with a phlegmatizing compn. It was believed that on firing such a round, the hot gases of combustion of proplnt would cause the phlegmatizer to spread on the inner surface of the gun barrel, thus forming a protective coating against the erosive action of hot gases [PATR **2145**(1955), p Rus 6]

Still simpler was to cover the driving band of a projectile with a special heavy lubricant (Ref 43, p 54)

Erosion may also be caused by occlusion (absorption) of propelling gases by metal of the bore, thus forming a solid solution, sometimes reacting chemically. The rate of occlusion and the depth of penetration into metal increase with an increase of temperature and pressure. The occluded gases remain, after each firing, sealed in the pores of metal and when they are using pressure to release themselves, considerable damage to the surface of the bore results. If the gases of occlusion are not released immediately after firings, and the weapon is cleaned, lubricated and stored, some rusting might take place due to the reaction between slowly released gases and metal. Besides rusting, the slow release of occluded gases causes roughing and softening of the surface of the bore. Occlusion may be diminished by employing "progressive burning" propellants (Ref 43, pp 154–55)

One of the reasons for increased erosion is the formation of soft steel on the surface of the bore. This softening is caused by increased percentage of carbon, which comes from C of propelling gases when they attack the surface of the bore while it is at temp of 300–500°(Ref 43, p 155)

Erosion is increased and life of a gun is decreased with increase of propelling charge and increase in caliber of gun

For example, barrels of machine guns of caliber less than 10mm are good for 10000 or more firings; cannons of cal smaller than 100mm can stand thousand or more firings; cal 120mm – 200 firings; 152mm – 166; 208mm – 125; 254mm – 100 and 305mm – 83 firings. With the same caliber, shorter range weapons (such as howitzers) have longer life than long range cannons (Ref 43, p 155)

Development of "Cool" Propellants. Due to the fact that the armor of tanks and ships after WWI period was made thicker and thicker and the speed of the planes greater and greater, it became necessary to increase the penetrating power of existing guns, which could be done by increasing their muzzle velocity to as much as 3300ft/sec. In order to achieve such velocities it was necessary to use proplnts of high ballistic potential, such as those contg NG. As these proplnts were "hot" (calorific value about 950kcal/kg) they caused excessive erosion, thus lowering considerably the life of the gun. For example, the life of AA guns using a 950kcal/kg proplnt was only 1700 firings, while for a 820kcal/kg proplnt about 3500 firings were obtd. Even before the number 1700 firings was reached, the gun became less effective because of the escape of gases between the lands of the bore's rifling and the driving band of projectile. This escape of gases not only reduced the chamber pressure (thus causing reduction in muzzle velocity of the projectile, with consequent reduction of range, penetration and precision of firing). It also caused excessive muzzle flash. As the decrease in efficiency of an older gun is usually compensated for by increasing the proplnt charge, this leads to a still brighter flash. In order to reduce the flash, more and more "flash reducing agent" (such as K_2SO_4) had to be incorporated and, as these agents are inert materials, they diminished the ballistic potential of proplnts. As erosion of a gun wears away its rifling, the result is always unsatisfactory rotation of the projectile (spin) with associated fuze failure. Particularly bad erosion was observed with high muzzle flash guns

The problem of erosion was particularly serious during WWII for Germans because they suffered considerable shortage of steel-hardening metals (such as Cr, Ni, Mn, Mo, etc) required for making modern gun barrels, and due to the shortage of labor and, in many cases, shortage of ordinary steel, the replacement of eroded guns was quite a serious problem

Anticipating these difficulties, the team of General Uto Gallwitz and also the Laboratories of Dynamit AG started, as early as 1934, to investigate the problem of development of proplnts of low calorific value but of high ballistic potential comparable to NG proplnts. As result of their work, a series of low calorific value proplnts (known as "cool" proplnts) were developed, such as **"G"** (Gallwitz) **Pulver** by Gen Gallwitz. It contd DEGDN, NC, EtCentralite, K_2SO_4, MgO and graphite. It was called **"K" Pulver** by the Ger Air Force. This proplnt was very suitable for high caliber guns, especially howitzers and mortars, but unfortunately, when fired from the same weapons as NG proplnts, it produced more flash due to the fact that larger charges were required with "G" Pulver

Note: Accdg to ARE Rept No 8/54 (Ref 37, pp 2 & 8), investigation at Woolwich Arsenal showed the German claim that DEGDN proplnts produce less erosion than NG proplnts of the same ballistic potential was not valid

"G" Pulver was, however, unsuitable for medium howitzers and mortars due to the low speed of combstn as compared to NG and straight NC proplnts. In order to eliminate this drawback, the Dynamit AG suggested incorporation of NGu (Nitroguanidine). The resulting proplnt, known as **Gudolpulver**, was very satisfactory in field howitzers from the point of view of ballistics, as well as because it produced only low muzzle flash without addn of K_2SO_4 or other flash-suppressing agent and also low erosion and corrosion (Refs 29, 30 & 44)

Cool proplnts developed in the USA during and after WWII contd large amts of NG in addn to NC and "explosive oils" (NG, DEGDN, TEGDN, or Metriol Trinitrate). A brief description of these proplnts known as **Triple-Base Cannon Propellants** is given in Vol **2** of Encycl, p C37-R, while compns of proplnts M15, M17, T20, T29, T34 and T39 are given in Table V on p C34

Several cool proplnts were developed after WWII in Great Britain. They contd NGu *(Picrite)* in addn to NG (or DEGDN) and NC. Their compns are given in ARE Rept of 1954 (Ref 37). Erosive action of these proplnts was tested in 17-pounder gun barrels made of Ni-Cr-Mo steel and results reported in Ref 37

Several cool proplnts were developed in Belgium after WWII. They were patented by the Recherches Chimiques SA, BelgP 566271(1958), listed in CA**53**, 10766(1959)

Note: Definition of terms driving (or rotating band) bore of a gun barrel, grooves, lands, muzzle and rifling are given in Vol **5**, under Driving (or Rotating) Band, p D1547-Rff

Refs: 1) P. Vieille, MP **11**, 156–219(1901) (Étude sur des phenomènes d'érosion) 2) P. Siwy, SS **3**, 42(1908) & CA **2**, 2862(1908) (Causes of erosion in guns) 3) E. Bravetta, SS **4**, 341–65(1909) & CA **4**, 1674(1910) (Erosion of guns and methods for prevention) 4) P. Siwy, SS **5**, 331–33(1910) (Die Abnutzung der Geschütze und deren Ursachen) 5) Marshall **1**(1917), 315–18 (Erosion and Noble's erosion experiments) 6) G. Létang, MAF **1**, 1013–35(1922) (Sur l'usure des bouches à feu) 7) P. Paquelier, MAF **1**, 1037–40(1922) (L'usure, l'encuivrage, l'aciérage et le baguage) 8) I.A. Krylov, "Izucheniye Razgoraniya Kanalov Ognestrel'nago Oruzhiya" (Study of Barrel Erosion in Firearms), Gosizdat, Moscow (1922) 9) E. Piantanida, Rivista Marittima (October 1922) (Sulle alterazioni subite dall'acciaio di tubi animma dei cannoni usurati), translated in MAF **2**, 102(1923) under the title: "Sur les altérations subies par l'acier de l'ame des canons usés" 10) E.B. Di Seconco, MAF **8**, 3^e fascicule (1929) (Usure des bouches à feu) 11) R.H. Greaves, H.H. Abram, S.H. Rees, MAF **2**, 2^e fascicule(1930), translated from the article "Erosion of Guns", published by Iron andSteel Institute (May 1922) 12) D. McNair, SS **25**, 198(1930) (Die Abnutzung von Geschützrohren) 13) G. Tchekoff (G. Chekhov), MAF **10**, 3^e fascicule(1931); **11**, 2^e fasc(1932) and **12**, 1^{er} fasc(1933) (Usure des bouches à feu) 14) Marshall **3** (1932), 93 (Erosion) 14a) Vennin, Burlot & Lécorché(1932), 274 15) L. Gabeaud, MAF **12**, 285–334(1933) (Essai d'une théorie de l'usure des bouches à feu) 16) H. Muraour, MAF **12**, 441–47(1933) (Note sur le pouvoir érosif des poudres B additionées des divers produits destinés à abaisser la temperature de combustion) 17) A. Rege, Rivista Marittima (1935) (Sull'usura dei cannoni) 18) J. Lintès, MAF **14**, 175–223 (1935) (Étude critique sur l'usure des bouches à feu) [Translated from Rumanian Revista Artillerei **1933**, p 327(May) and 394(June)] 18a) Pepin Lehalleur (1935), 102 19) E.

Piantanida, Rivista Marittima, **1935** (Oct, p 45) [Translated in MAF **17**, 137–51(1938) under the title "Des altérations de l'acier des canons usés par le tir] 20) W. Schwinning, MAF **16**, 441–94(1937) (Recherches sur l'usure des bouches à feu) 21) V.N. Poddubnyi, Tekhnika i Voöruzheniye **6**(9), 52–63(1937) & CA **33**, 9267(1939) (Erosion of arms) 22) T.J. Hayes, "Elements of Ordnance", Wiley, NY(1938), 81–2 23) T. Felsztyn & S. Spiewak, MAF **17**, 283–321 (1938) (Usure des canons, d'armes portatives et automatiques et leur analyse metallographique) 24) J. Costier, MAF **18**, 961–84 (1939) (L'usure des bouches à feu) 25) R. Sutterlin, MAF **19**, 300–13(1940) (Application d'une théorie des résistances passives a l'étude de l'usure des bouches à feu) 26) Davis (1943), 294–96, 387–88 and 457–58 (Erosion) 27) J. Corner, TrFaradSoc **43**, 635(1943) (Erosion of proplnts at gun pressures) 28) Alvaro-Alberto, Anais AcadBrasileira de Ciencias **15**, 39–61, 187–89, 239–42(1943) & CA **40**, 2629(1946) (Erosive power of various propellants, such as Ballistite, Cordite, Solenite, etc is discussed) 29) General Uto Gallwitz, "Die Geschützladung" (The Cannoncharge), Heerenwaffenamt, Berlin (1944) (Development in Germany of propellants of reduced erosion) 30) O.W. Stickland, "General Summary of Explosive Plants", PB Rept **925**(1945) (Development in Germany of proplnts of reduced erosion) 31) A. Pérez Ara, "Tratado de Explosivos", Edit "Cultural SA", La Habana(Cuba) (1945), 429–32 (Erosiones producidos en las animas de los canones) 31a) H. Muraour, "Poudres et Explosifs", Presses Universitaires, Paris (1947), 115–16 31b) A.D. Blinov, "Kurs Artilleriye", Voyen-Izdat, Moscow, Vol **4**(1947), p 67 and Vol **5** (1949), p 163 31c) Stettbacher (1948), 42 & 48 (Erosion der Schusswaffen) 32) "Ordnance Proof Manual" **16–17**(1948), "Erosion and Service Life Tests" (The purpose of these tests is to determine: a) the useful life of a given cannon tube and the type of ballistic failure which limits its life b) the rates of bore erosion and muzzle velocity loss and c) the means of minimizing erosion) 33) Kirk & Othmer **4**(1949), p 529 (Erosion is defined as a combination of corrosion and mechanical wear which varies all the way from a corrosion action to mechanical action analogous to fatigue. In any particular case both aspects are involved and it is not possible to separate one from the other) 34) H. Ficheroulle & A. Kovache, MP **32**, 377–81(1950) (Erosion of sporting firearms caused by priming mixtures was smaller when using Lead Styphnate-Tetracene mixtures than compositions contg Mercuric Fulminate) 35) J. Corner, "Theory of the Interior Ballistics of Guns", Wiley, NY (1950), 73–84 (Erosion of propellants) 36) L. Green, JetPropulsion **24**, 9–15(1954) & CA **48**, 11061(1954) (Erosive burning of some composite solid propellants) 37) H.H. Abram, T. Williams & K.F. Allen, "Examination of Six QF 17-pr Gun Barrels Used in Ballistic and Erosion Trials of Propellants of Various Compositions and Flame Temperatures", Armament Research Establishment Metallurgy Report **No 8/54**, Woolwich, SE 18(1954)

Six 17-pounder gun barrels were used for comparative wear trials with proplnts of different compns. The barrels were all made of Ni-Cr-Mo steel to the same specification, and were sufficiently similar in compn, mechanical props and microstructure to ensure that differences in the final conditions of their bores were due solely to the different propellants used to obtain approx similar internal ballistics

Comparative examination of the six barrels has shown the large reduction in erosion which resulted from the use of the three "cool" proplnts of adiabatic FT (flame temperature) = 1950°K as compared with the erosive effect of *Cordite N* (FT = 2450°K) and still more so when compared with the amt of erosion produced by proplnts of FT = 2800°K. The results also showed that Picrite proplnts (Picrite is Brit designation of Nitroguanidine, abbr by us as NGu) are less erosive than their non-picrite equivalents of similar FT but accelerate the incidence and increase the severity of "craze-cracking" of the bore surface. This is due to the diluent effect caused by high nitrogen content (order of 30% by vol) of the gases formed by Picrite proplnts

Note: Craze-cracking means the developing of small cracks or surface markings on the surface of a case-hardened metal

Bore enlargement to rear of C of R (Commencement of Rifling) was due to the erosive action (thermal and chemical) of the proplnt gases and not to mechanical wear by the driving band (which was of copper). This bore enlargement in the three barrels which fired "cool" proplnts (FT = 1950°K) was sufficiently small to enable very little forward movement of the projectile to produce gas-sealing by the driving band. In the three barrels which fired hotter proplnts (particularly those of FT = 2800°K) bore enlargement became sufficiently great to necessitate considerably greater travel of the proj before gas-sealing by the band was effected. During this longer travel proplnt gases would escape past the driving band, thereby causing it to become heated and softened. This softening of the Cu band by escaping gases presumably provided the reason why during the wear trials the forward portions of the three barrels firing the hotter proplnts showed no measurable increase in wear, whereas a small but definite amt of bore enlargement occurred in the three barrels which fired the 1950°K proplnts

In worn guns which have fired std Service proplnts and Cu-banded projs, it was usually found that bore enlargement forward C of R decreased relatively rapidly to very small values and thence – forward the bore exhibited only slight wear. This relative absence of erosion and wear in the forward portion of the bore was due to cooling of the proplnt gases by expansion, and shorter duration of gas heating of the bore surface, as well as the fact that the Cu band suffered frictional heating and softening, which caused a progressive decrease in band pressure and frictional drag as the proj passed along the bore. When bore enlargement in C of R region reached and exceeded the maximum diam of the unengraved driving band, then escape of proplnt gases past the band accelerated the heating and softening of the band, and thus helped to preserve bore dimensions in the forward part of the barrel, although in some instances the softened band could fail to center the proj with consequent groove-marking and muzzle wear. Driving bands made of steel, soft iron or sintered iron did not suffer the same degree of softening as Cu bands, and by their abrasive action progressively extended land wear towards the muzzle

In many guns firing Cu-banded projs, the major factor responsible for wear of C of R region was the erosive action of the proplnt gases, although the frictional drag of the band did tend to play an early part which decreased in importance as wear increased. The part played by a Cu-driving band in the early stages of wear development depended on the type of gun and the design of the band. A heavy band produced more wear than a light one, but under conditions of rapid erosion the action of the proplnt gases overshadowed any wearing effect of the Cu band. Erosion by proplnt gases is mainly due to exothermic oxidation of the steel to form FeO as in the flame cutting of steel by oxygen torch. Where erosive wear was rapid, the FeO formed was swept away by the proplnt gases, whereas the rate of erosion was slow, a thin film of FeO remained to be subjected to the scraping action of the driving band of the succeeding round. This surface film of FeO has been found and identified (by X-ray diffraction) on the blackened bore surface immediately forward of the "clean swept" region of rapid erosion. The black surface film was strongly adherent and, judging by wear curves, appeared to be very resistant to the scraping action of Cu bands, although it commenced on the land tops a little farther forward than on the surface of the grooves

As wear in a gun barrel progressively increased, a certain amt of "free run" (unrotated travel of the proj) was introduced and a stage could eventually be reached when a "free-running" Cu-banded proj fails to engrave correctly on meeting the rifling. Then the band slipped relative to the rifling and could become machined by the rifling to a smooth condition instead of being engraved. Smooth bands would obviously be produced more readily if the Cu band had already been softened by proplnt gases escaping past it during free-run travel

As *plastic bands* would not suffer such drastic softening by escaping proplnt gases

Table E13

Constituents	Composition, %					
	F488/312	F551/66	Cordite N	F487/68	F428/180	F487/46
Picrite (NGu)	Nil	55.0	54.7	Nil	Nil	60.0
NC	56.5	21.0	19.0	70.0	65.0	20.0
NG	33.0	21.0	18.7	Nil	15.4	Nil
DEGDN	Nil	Nil	Nil	14.3	Nil	11.16
Carbamite (Ethylcentralite)	3.5	3.0	7.3	2.0	2.0	2.64
DBuPh	7.0	Nil	Nil	13.7	17.6	6.2
Na Cryolite	Nil	Nil	0.3	Nil	Nil	Nil
Adiabatic Flame Temperature, °K	2800	2800	2450	1950	1950	1950
Gas Composition (% by Volume) at T_0 °K						
CO_2	7.6	5.2	3.5	3.4	3.1	2.5
CO	45.4	27.7	31.3	51.4	52.3	32.3
H_2	16.8	16.6	22.6	28.6	28.8	29.2
H_2O	19.2	21.5	15.1	8.3	7.5	10.0
N_2	11.0	29.0	27.5	8.3	8.3	26.0

as Cu bands, it was reasonable to surmise that plastic-banded projs would function better than Cu-banded ones in worn gun barrels firing fixed ammo. Some supporting evidence for this surmise was provided by the results of comparative ballistic trials carried out at ARE with ebonite-banded and Cu-banded projs in worn 6-pr, 6-cwt and QF 5.25 inch guns

Craze-cracking was more prolific and persisted further along the bore in the three barrels which had fired Picrite proplnts than in the other three barrels. The depth of altered microstructure below the bore surface decreased towards the muzzle more gradually in the Picrite firing barrels than in the other barrels, a fact which would appear to be related to heating of the bore surface by proplnt gases escaping past the driving band via the bore cracking

Table 13 gives compositions of propellants and calculated compositions (volume per cent) of gases formed therefrom at adiabatic flame temperature

It must be noted that the two non-picrite proplnts of FT = 1950°K were different in that one (F487/68) contd 14.3% DEGDN, while the other (F428/180) contd 15.4% NG. The results of comparative trials, as was pointed out in ARE Memo No 11/50, did not support the German claim that DEGDN was a less erosive ingredient than NG

Table 14 lists propellants used in respective barrels, and charge weights employed in main series of firings for wear determinations

38) Anon, Combat Forces Journal(USA), Vol **4**, No 8, p 40(1954) 38a) J.E. Capell, PATR **2033**(1954) and private communication (1954) 38b) B.T. Fedoroff et al, PATR **2145**(1955), p Rus 19 (Lubricating gasket for reduction of erosion in guns, known as "prossal'nik) 39) P. Tavernier, P. Prache & J. Berger, MP **37**, 207-15(1955) (Contribution à l'étude de l'érosion des poudres colloidales) [Comparision of erosion produced by Poudre SD (temp of expln at const vol 2561°K), Amer proplnt JPN (temp of expln 3886°K) and Ger froide (cool) proplnt (temp of expln 1880°K) showed that poudre SD is particularly sensitive to the phenomenon of erosion. Unfortunately no compn of any of the above proplnts is given)] 40) Anon, "Cannon Tube Erosion and Service Life Tests", OP Manual **70-50**(1956) (See Ref 32) 41) J. Berger, MP **38**, Annexe(1956), 1-108 (Thesis, entitled: "Contribution à l'Étude de l'injection Pariétale") 42) J. Boisson, MP **39**, 381-96(1957) & CA **53**,

Table E14

7-pr Barrel No	Propellants						Rounds Fired After Proof	
	Designation	Type	Flame Temperature	Charge Weight lb	oz	dr	Prior to Wear Trial	Total
38130	F488/312/S	Non-picrite	2800°K	5	14	0	77	477
16113	F551/66/S	Picrite	2800	5	15	4	79	579
28013	Cordite N/S	Picrite	2450	6	5	0	75	575
10147	F487/68/T	Non-picrite (+DEGDN)	1950	7	4	12	78	578
16600	F428/180/T	Non-picrite (+NG)	1950	7	0	8	78	578
26809	F487/46/S	Picrite (+DEGDN)	1950	8	4	0	65	565

Note 1: Charge weights were detd to produce a max chamber pressure of 20psi at 80°F
Note 2: Firings were continued until production of considerable wear (Total Rounds Fired)

3696(1959) (Erosive burning of colloidal propellants) 43) A.G. Gorst, "Porokha i Vzryvchatyiye Veshchestva" (Propellants and Explosive Substances), GosIzdatOboronProm, Moscow (1957), 152–55 (Razgar stvola orudiya i eroziya) (Burning and erosion of gun barrel) 44) B.T. Fedoroff et al, PATR **2510**(1958), p Ger 43: "Erosion of the Bore"; pp Ger 70 to Ger 71 ("G" Pulver), p Ger 81 (Gudolpulver) 45) K.K. Andreev & A.F. Belyaev, "Teoriya Vzryvchatykh Veshchestv" (Theory of Explosive Substances), Oboronghiz, Moscow (1960), 430–32 (Erosion of gun barrels, which includes on p 431 description of test in the bomb presented in Fig 8.4) 46) R.F. Mocharnuyk, VoprPoroshkovoiMet i ProchnostiMaterialovAkadNaukUkrSSR **1960**, No 8, 81–89 & CA **57**, 8283(1962) (A review with 31 refs) 47) A.I. Maksimov et al, Zavodskaya Lab **27**, 1311–12(1961) & CA **56**, 6242(1962) (App for corrosion-erosion studies of thermally stable materials in combstn products of liq fuels) 48) S.P. Gary, ProdFinishing (Cincinnati) **28**(1), 36–46(1964) & CA **62**, 6219(1965) (Evaluating the props of plated finishes) 48a) The mechanism whereby a titanium oxide–wax additive reduces erosion in a gun barrel is described and discussed in a recent report by C. Lenchitz, R.W. Velicky, L.A. Bottei, and G. Silvestro of Picatinny. This additive was found to work in the following ways: (a) the wax acts directly to reduce erosivity; (b) the wax interacts with propellant gases to reduce the temperature next to the barrel wall, and to reduce the calorific value of the propellant; and (c) the titanium oxide strengthens the protective coating in the gun barrel and helps to disperse the wax evenly over the surface of the barrel wall, (PATM **1768**, November 1965) 49) M.G. Sirotyuk, AkustZh **12**(1), 87–92(1966) & CA **65**, 417(1966) (Effect of temp & gas content of a liq on cavitation processes) 59)Gorst(1972), 169-73

Erosionless Priming (Erosionsfreie Zündung). Any priming composition contg MF (Mercuric Fulminate) with or without a chlorate (usually KClO$_3$) has been known to cause considerable erosion of gun barrels. In order to reduce the erosion, H. Ziegler of Switzerland proposed in 1904 to replace chlorates with nitrates, such as Ba(NO$_3$)$_2$. These compns were named "rostfreie Zündungen" (rust-free primers). As these substances were not entirely satisfactory, further search resulted about 1930 in the invention of compositions based entirely on organic ingredients such as Tetracene (Tetrazen). These substances, known as "erosionsfreien Sinoxydsätzen" were manufd before and during WWII by the Rheinisch-Westfälische Sprengstoffe AG, Nürnberg (Refs 1, 2, 3 & 5). Ficheroulle & Kovache (Ref 4) reported that erosion of sporting firearms caused by priming mixtures was smaller when using LSt (Lead Styphnate) Tetracene mixtures, than compositions contg MF

Refs: 1) P. Wolf, SS **27**, 397–99(1932), "Die korosionsfreie Zündung" 2) E. von Herz, DRP 518855 and SS **28**, 37–42 (1933), "Die erosionsfreie Zündung" 3) A. Stettbacher, Spreng- und Schiesstoffe", Rascher, Zürich (1948), 106–07 (Erosionsfreien Sinoxydsätzen) 4) H. Ficheroulle & A. Kovache, MP **32**, 377–81(1950) (Erosion of sporting firearms) 5) B.T. Fedoroff et al, PATR **2510**(1958), pp Ger 43-L & Ber 43-L (Erosionless Priming)

Erosion of Ground; Control by Explosives. See item h, p A113-R in Vol **1** of Encycl, under "Agriculture and Forestry Uses of Explosives"

Erosive Burning (Action or Power) of Propellants. This subject is briefly discussed under "EROSION OF GUN BARRELS" and more fully in the following references listed there: 1, 16, 27, 28, 29, 30, 35, 36, 37, 39, 41, 42, 43, 44 & 45

Ersatzdynamit (Ger for *Substitute Dynamite*). Any Dynamite in which a large proportion or all NG is substituted by some other HE (such as TNT, PA, PETN, RDX, etc) in such a manner that the resulting compn is equal in strength to the original Dynamite
Refs: 1) Naoúm, Expls (1927), 99 2) PATR **2510**(1958), p Ger 43-R

Ersatzgeschoss (Ger for *Substitute Projectile*). Due to the shortage of steel and other metals, the Germans, during WWII, developed among many other substitute ammunition items, an HE-shrapnel shell which was made of a combination of concrete and steel scrap. Some of these shells were used toward the end of the war
Refs: 1) L.E. Simon, "German Research in WWII", Wiley, NY(1947), p 190 2) B.T. Fedoroff et al, PATR **2510**(1958), p Ger 43-R

Ersatzsprengstoffe (Substitute Explosives). Due to the acute shortage in Germany of TNT and other aromatic nitrocompounds, several substitute explosive mixtures were developed and used during WWII. Many of the "Ersatz" explosives were developed at the Krümmel Plant of Dynamit AG, others at Christianstadt and other plants

In the preparation of various melt-loading compositions, the following trends were noticeable:

a) Substitution of RDX for part of the TNT in Amatols
b) Substitution of DNB for TNT in Amatols
c) Substitution for TNT, by nitroaromatics such as Dinitrodiphenylamine, Hexanitrodiphenylamine, Trinitroxylene, Dinitronaphthalene, etc
d) The use of lowmelting hydrous inorganic nitrate compounds, such as Ca, K and Na nitrates, to pemit the reduction or replacement of TNT
e) The use of Al powder as an ingredient
f) The use of miscellaneous organic ingredients such as urea, PE (Pentaerythritol), Guanidine Nitrate, Ethylenediaminedinitrate, Methylaminenitrate, etc
g) The use of sodium chloride (up to 60%) or of so-called "Scheidemehl" (powder consisting of a mixture of Ca and Mg silicates) in order to reduce the amount of TNT

Most of the explosives containing these substances were much less powerful and brisant than TNT alone.

Note: From German documents, it appears that the critical period with regard to the supply of explosives and ammunition was reached in August 1944. From that date, serious shortages occurred. It was in September 1944 that, on account of the shortage of NH_4NO_3, the High Command ordered the use of mixtures of 50/50 TNT/NaCl, or even 40/60 TNT/NaCl, for loading shells. However, previous to this, mixtures of 50/50 TNT/$NaNO_3$ (Sodatol) and 45/40/15 TNT/$NaNO_3$/Al had been used to a considerable extent.

Table E15 lists the principal "substitute explosives" used by the Germans during WWII

Table E15

	Designation and % Composition																			
	Amatol			Ammonit							HEXO		HEXA			KMA			NaCl	TNX
Components	39	39a	40	H-1	H-2	H-5	H-8	43c	S-6	S-6 modif	S-19	S-22	S-22 (see note)	S-26	E-4	Block	S-16	S-19	Explosive	Explosive
TNT	50	-	-	-	-	-	-	30	40	30	-	-	-	-	-	40	-	-	40-50	80
Amm Nitrate	40-45	35	40	50	50	50	50	45	-	-	55	45	45	55	44	-	32	73.3	-	-
Na Nitrate	-	-	-	-	-	5	-	-	-	-	9	9	9	9	10	-	6-8	17.4	-	-
K Nitrate	-	-	-	-	-	-	25	-	-	-	4.2	3	3	4.2	-	-	0-2	-	-	-
$Ca(NO_3)_2.4H_2O$	-	-	-	15	15	15	-	10	-	-	-	-	-	-	-	-	-	-	-	-
RDX	5-10	15	10	25	25	20	15	-	-	-	15	14	-	-	-	-	10	-	-	-
PH - Salz	-	-	-	-	-	10	-	-	-	-	-	14	14	-	-	-	10	-	-	-
Urea	-	-	-	-	-	-	-	-	-	-	1.8	-	-	1.8	2	-	-	9.3	-	-
Al Powder	-	-	-	-	-	-	-	-	10	15-25	15	15	15	15	30	10	40	-	-	-
HNDPhA	-	-	-	-	-	-	-	-	30	35-30	-	-	14	15	14	30	-	-	-	-
DNN	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	20	-	-	-	-
DNB	-	50	50	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
PETN	-	-	-	10	-	-	10	-	-	-	-	-	-	-	-	-	-	-	-	-
GuN	-	-	-	-	10	-	-	15	-	-	-	-	-	-	-	-	-	-	-	-
DNDPhA	-	-	-	-	-	-	-	-	20	15-20	-	-	-	-	-	-	-	-	-	-
Na Chloride	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	60-50	-
TNX	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	20

Note: Composition S-22 sometimes exploded during the loading of projectiles

Abbreviations: **Amm** Ammonium; **DNB** Dinitrobenzene; **DNN** Dinitronaphthalene; **DNDPhA** Dinitrodiphenylamine; **GuN** *Guanidine nitrate;* **HNDPhA** *Hexanitrodiphenylamine;* **PETN** *Pentaerythritol Tetranitrate;* **PH-Salz** Ethylenediamine Dinitrate; **RDX** Cyclonite, or Hexogen; **TNT** Trinitrotoluene; **TNX** Trinitroxylene.

To this table may be added the following:

a) An explosive composition prepd by I.G. Farbenindustrie by nitrating a mixture of MNX, methylaniline and MNT. The nitrated product considted of TNX 45, Tetryl 50 and TNT 5%

b) An explosive mixture of the Krümmel plant of D A-G contained TNT 45, Am Nitrate 40 and Al powder 15%. It was suitable for cast-loading bombs, grenades and land mines.

c) An explosive mixture of the Christianstadt plant of D A–G was a slurry of 70% $Ca(NO_3)_2 4H_2O$ and 30% TNT

The following explosives also belong to Ersatzsprengstoffe: Amatol, Ammonal, Ammonit, DI-Salz, Fillers Nos 13, 13a, 13-113, 19, 20, 52, 56, 57 (or Abonachit), 60, 61, 64, 70, 84, and 88, Formit, HDD, Man-Salz, Myrol, PH-Salz, Tetansprengstoffe (TeNMe explosives), Tetramethylnitraminotetramethylmethane, TETRA-Salz, Trinitroethanol Perchlorate

In addition to the explosives mentioned above, before and during WWII, the Germans developed and used several new explosives and explosive mixtures which cannot be called "substitutes" (Ersatzsprengstoffe) because they were more powerful than the previously used military explosives, such as TNT and PA. These new powerful explosives included PETN and RDX, as well as various mixtures containing these substances

Refs: 1) O.W.Stickland et al, "Survey of German Practice and Experience in Filling High Explosive Items", U.S. Office of Technical Service, PB Rept No 1820 (1945), pp 11,15,16,24,29 2) O.W. Stickland et al, "General Summary of Explosive Plants", PB Rept No 925 (1947), Appendix 7

Erupterite. A chlorate based explosive manufd by the Northern Explosives Company, Ltd, Glasgow, Scotland and authorized in 1899 by British Govt for use in some mines. Its compn is not given
Ref: Daniel, Dict (1902), 283

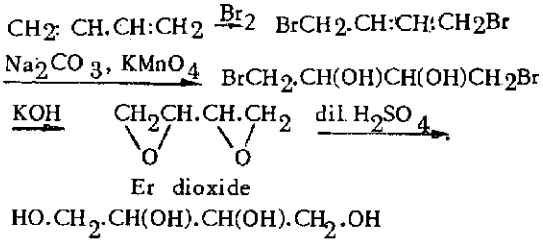
Erythranediol Dinitrate,
$O_2NO.CH.CH_2O-CH_2CH.ONO_2$, mw 194,10, N14.43%. Its props & methods of propn were not found in the literature

It is an explosive more powerful (119% PA by Trauzl Test) than PA and comparable with it in impact sensitivity (FI 103%PA)
Refs: 1)Beil (not found) 2)AC-956/Org EX 74(1941) 3) Blatt,OSRD **2014** (1944) (under Nitrates) 4) Not found in CA

Erythrene or Vinyl⁻thylene. See Butadiene in Vol **2** of Encyc ⁻365-R

Erythrite. A natural hydrated cobalt arsenate which is found in USA and in Canada. Not to be confused with Erythritol which is also called Erythrite
Ref: CondChemDict (1961), 448-L

Erythritol (Er); i-Erythrite; 1,2,3,4-Butanetetrol or Tetrahydroxybutane (also known, accdg to Daniel, as *physite*),
$HO.CH_2.CH(OH).CH(OH).CH_2.(OH)$; mw 122.12, wh., sweet-tasting tetragonal prisms, sp gr 1.451 at 20/4°, mp 120°, bp 329-31°, sol in w(61% at RT), sl sol in pyridine & in cold alc; insol in ether bz & chlf. Had been prepd until recently by extraction from lichens (Rocella Montagnei) which contains in dry state about 3.5% of Er. At present accdg to info obtd by us from Dr Jean P. Picard *(Ref 3)* Er can be prepd from 1.4 butadiene, $CH_2:CH.CH:CH_2$ which, in turn, can be obtd in 70% yield, by modified Russian Process from alcohol and acetaldehyde:
$C_2H_5OH + CH_3COH \rightarrow CH_2:CH.CH:CH_2 + H_2O$
This process is used now by Carbide and Carbon Chemical Corp
Prepn of Er from 1,4-butadiene is as follows:



$$CH_2:CH.CH:CH_2 \xrightarrow{Br_2} BrCH_2.CH:CH:CH_2Br \xrightarrow{Na_2CO_3,\ KMnO_4} BrCH_2.CH(OH)CH(OH)CH_2Br$$

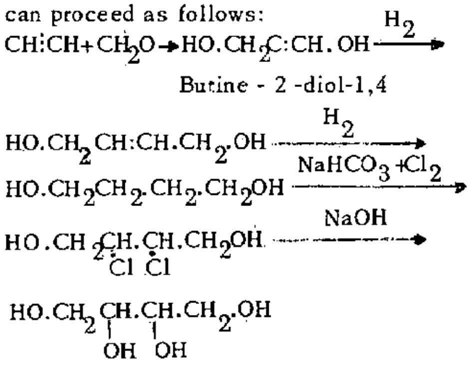
$$\xrightarrow{KOH} CH_2CH.CH.CH_2 \xrightarrow{dil.\ H_2SO_4}$$

Er dioxide

$$HO.CH_2.CH(OH).CH(OH).CH_2.OH$$

Racemic d,l- erythritol

In the German process for prepn of erythritol (by Reppe), starting materials are acetylene and formaldehyde and reactions can proceed as follows:



$$CH:CH + CH_2O \rightarrow HO.CH_2C:CH.OH \xrightarrow{H_2}$$

Butine - 2 -diol-1,4

$$HO.CH_2CH:CH.CH_2.OH \xrightarrow{H_2}$$

$$HO.CH_2CH_2.CH_2.CH_2OH \xrightarrow{NaHCO_3 + Cl_2}$$

$$HO.CH_2\underset{Cl}{CH}.\underset{Cl}{CH}.CH_2OH \xrightarrow{NaOH}$$

$$HO.CH_2\underset{OH}{\underset{|}{CH}}.\underset{OH}{\underset{|}{CH}}.CH_2.OH$$

Mesoerythritol

Note: It has been claimed by the GerP 734025 of IG Farben-Industrie, CA **38**, 981 (1944) that a soln of butene-2-diol-1,4 in aq alkali carbonate gives, if acted upon by chlorine, the chlorhydrin derivative which will give upon saponification the Meso-erythritol
Refs: 1) Beil **1**, 525,(279) & {2356} 2) Daniel (1902), 480 (under Nitrérythrite) 3) Dr Jean P. Picard of PicArsn; private communication (about 1950) 4) N.R. Trenner & F.A. Bacher, USP 2571967(1951) & CA **46**, 8672 (1952) (Prepn) 5) M.G. Lambon, AnalChem **28**, 1216 (1956) & CA **50**, 15324 (1956) (Detection of erythritol & other polyols, by multiple spray technique, chromatographically)

Erythritol Dinitrate (ErDN) or 1,2,3,4 - Butanetetrol Dinitrate,
$(O_2NO)CH_2.CH(OH).CH(OH)CH_2(ONO_2)$; mw 212.12, N 13:21%, OB to CO_2 -30.2%, crysts, mp 84.5-85°; bp-explodes

Was prepd by Fichter (Ref 2) on stirring for 20 mins, 10g of erythritol with 50g of mixed mitric-sulfuric acid (1:2), previously cooled to 0°. It is a mild explosive

Refs: 1)Beil- not found 2) F. Fichter, HelvChimActa **25**, 229-40 (1942) & CA **36**, 5713(1942)

Erythritol Tetranitrate (ErTeN), Nitro-i-erythrite or 1,2,3,4-Butanetetrol Tetranitrate (called by Stenhouse Salpetersauerer Erythromannit) $(O_2NO)\,CH_2(CH.ONO_2)_2CH_2\,(ONO_2)$; mw 302.12,N18.55%, OB to CO +26.5%, OB to CO_2+ 5.3%; col plates (from alc), sp gr 1.59, mp 61°,bp-detonates violently; insol in cold w; sl sol in cold alc, more in hot; sol in ether, acetone & glycerol; reduces Fehlings soln on prolonged heating, while PETN does not(Ref 9, p 280)

Was first prepd in 1849 by Stenhouse (Refs 1&2) by dissolving with cooling, erithritol in fuming nitric acid and then precipitating the resulting ErTeN by the addn of concd sulphic acid. Bergeim (Ref7) patented a method of prepn with better yield in which erythritol (1 part) was dissolved (at 40-50°) in 5.5 parts sulfuric acid and the resulting soln was added to 7ps concd nitric acid or to a mixed acid contg a large proportion of nitric acid. The resulting ppt of ErTeN was separated, dissolved in alc or acetone, the soln neutralized by a weak alkali (NH_4 or Na carbonate) and diluted with a large amt of cold water to reprecipitate ErTeN

Accdg to Naoúm (Ref 6, p 238), explosion of ErTeN proceeds as follows:

$$C_4H_6\,(ONO_2)_4 = 4CO_2 + 3H_2O + 2N_2 + 0.5O_2$$

Properties of ErTeN, as listed in Refs 6,10,11, 13,14,15,16 and 17, are as follows:

Brisance - not known

Detonation velocity - not known

Explosion Temperature 220° (205°) vs 222° for NG in 5 sec

Friction sensitivity - detonates

Gas volume at NTP 705 l/kg vs 715 for NG

Heat of combustion 1546 cal/g at Cv, vs 1616 for NG

Heat of detonation 1414 cal/g at C_v, vs 1486 for NG

Heat of formation 376 cal/g at C_v, vs 400 for NG

Impact sensitivity, BurMines App, 2 kg wt - 20cm vs 15cm for NG

Loading density 1.50 vs 1.60 for NG

Power (Strength) - sl lower than for NG, 10g of which gives expansion of 550cc in Trauzl Block Test, vs 285 for TNT

Specific heat - 0.0337 cal/g /°C vs 0.356 for liq NG

Stability, Thermal - not known

Temperature of explosion 4565°C vs 4645° for NG

Thermochemical, pressure 8.15 atm x 10^4 vs 9.79 for NG

Uses of ErTeN, as listed in Refs 3,4,5,8,10,13 & 15:

The first reported use of ErTeN was in medicine against chest angina (l'angine de pointrine, in Fr). For this purpose finely pulverized ErTeN was triturated in a mortor with powdered lactose or sugar which was a dangerous operation resulting in some cases in violent explns. One of such explns, which killed in 1897 an English pharmacist, was described in Refs 4 & 5. The first use of ErTeN for expl purposes was described in two 1898 patents listed here as Ref 3. In both patents ErTeN was proposed to be used as an ingredient of smokeless proplnts in lieu of NG

Bergeim, who patented (Ref 7) a better method of prepn than the original Stenhouse method of 1849, proposed to use ErTeN as a chief sensitising expl constituent (Ref 8). Vest & Kuhn (Ref 15) of BRL prepd some solvent double-base propellents contg ErTeN in lieu of NG and observed slight "blooming" on the surface of such proplnts. It was suggested to try the prepn of "solventless" proplnts contg EtTeN in order to avoid blooming (Ref 15)

Accdg to the unpublished write-up obtd by us from Dr Picard (Ref 13), ErTeN could be considered as a possible substitute for NG, because it has a better oxygen balance, is less sensitive to impact, and, being a solid, is easier to handle. It seems however ,that proplnts contg ErTeN are not as stable as those contg NG and are more expensive. There was no hope until recently, to use ErTeN on a large scale on account of scarcity of material (lichens) used for prepn of erythritol. The problem of scarcity seems to be now eliminated after development of synthetic methods of prepn of erythritol from butadiene and acetylene outlined here under Erythritol. This, however, did not reduce the cost of ErTeN to such an extent that it could compete with that of NG

Refs for ErTeN: 1) Beil **1**, 527 & **9** [887] 2) J. Stenhouse, Ann **70**,225-28 (1849) (Prepn of ErTeN) 3) Ger P 110289(1898) and Brit P27397 (1898) (quoted from Davis, p 236, Footnote) (Uses of ErTeN in smokeless proplnts) 4) A. Ford, JSCI **18**, 415 (1899) (Reported an accident at Dartford in 1897 by which a pharmacist lost his life while engaged in triturating ErTeN with lactose powder in a mortor. Such mixt served as a medicine) 5) Daniel (1902) 480-81 (under Nitrérythrite) (Gave some props of ErTeN and described the accident at Dartford, England) 6) Naoúm,NG (1928), 237-38 (Prepn and some props of ErTeN) 7) F.H. Bergeim, USP 1691954 (1928) & CA **23**, 708 (1929) (Method of prepn of ErTeN) 8) F.H. Bergeim, USP 1751436 (1930) & CA **24**, 2606 (1930) (Use of ErTeN as a chief sensitizing expl constituent) 9) A. Schmidt, SS **29**, (1934) (The Thermochemical properties of ErTeN) 10) Davis (1943), 235-36 (Some props of ErTeN); 280 (ErTeN reduces Fehling's soln on boiling while PETN does not) 11) Blatt, OSRD, **2014** (1944) (Some props of ErTeN) 12) Pérez Ara (1945) (Some props of Tetranitroeritrita) 13) Dr Jean P. Picard of PicArsn; private communication (about 1950) (Some props and uses of ErTeN; synth methods of prepn of erythritol) 14) H. Henkin & R McGill, IEC **44**, 1391-95 (1952) (The lowest expl temp 205°, as observed for ErTeN in modified Marshall apparatus) 15) D.E. Vest & L. P. Kuhn, BRL (Ballistics Research Laboratory) Tech Note **757**, (Nov 1952) (Investigation of proplnts in which NG was replaced by ErTeN. They were both hard and tough at RT, but showed a slight "blooming" of ErTeN on the surface of dried grains. Virtually all the migration to the surface took place during the period of solvent removal by evaporation. It was expected that blooming will be eliminated in solventless proplnts using ErTeN in lieu of NG) 16) J. Taylor, "Detonation in Condensed Explosives", Clarendon Press, Oxford (1952), 62 (Comparison of some props of ErTeN with those of PETN, NG and NGc) 17) J. Büchi & R. Alther, PharmActaHelv **31** 121 (1956) & CA **50**, 13368 (1956) (Prepn) 18) J. Carol, JAssocAgricChemists **43**, 256-61 (1960) & Explosivst **1964**, 45 (Infrared detn for ErTeN) 19) CondChemDic (1961), 448-L (Some props of ErTeN)

E-Salz (Ger for E-Salt). One of the Hexogens (RDX's or Cyclonites), developed in Germany before WWII. It was prepd from formaldehyde, AN and acetic anhydride by the process known as E-Verfahren (See Vol **3** of Encycl, p C614-L). The earliest of the German Hexogens was *W-Salz* developed in 1935 (See W-Verfahren on p C613-L). Other Ger Hexogens were *SH-Salz*, developed in 1937-1938 (See SH-Vervahren on p C614-L), **K-Salz**, developed in 1938 (See K-Verfahren on p C614-L) and *KA-Salz*, manufd by combination of SH and K processes (See KA-Verfahren on p C614-R). Eventually KA-Salz replaced E-Salz

More detailed descriptions of German processes than given in Vol 3 of Encycl, are found in PATR **2510** (1958), pp Ger 88-R to Ger 90-L, under Hexogen

Escales, Richard, Dr (1863-1924). German scientist, specializing in explosives. Author of the book "Explosivstoffe" in several volumes. (See Vol **1**, of Encycl, p Abbr 69). Founded in 1906 the monthly journal "Zeitschrift für das gesamte Schiess-und Sprengstoffwesen" (abbreviated as SS). Holder of several patents on expls and author of numerous papers

Ref: A. Schrimpf, SS **19**, 145-46 (1924)

Note: The Zeitschrift für etc was suspended during WWII, but its publication was resumed in 1950's as "Sprengtechnik" changed to "Explosivstoffe" in 1952

Escales and Novak's Explosive, patented in 1909 was prepd as follows: glycerol (contg 6-20% H_2O) was heated with dil HCl for several hours at 100° and then mixed with about an equal part of glycerol After heating for a while at 110° to remove free HCl, the resulting mixture of glycerol, polyglycerols and their chlorohydrins was nitrated with mixed nitric-sulfuric acid to produce an explosive oil of low freezing point

Ref: R. Escales & M. Novak, USP 915165 (1909) & CA **3**, 1461 (1909)

Eschbachzünder oder Verzögerungszünder Eschbach (Eschbach Primer or Delayed Action Primer of Eschbach). Two models are described in Beiling-Drekopf (Ref 1), the "older construction" (Eschbachzünder älterer Bauart) is shown in Fig 55, p 232, while the so-called "Gasloser Eschbachzünder" is shown in Fig 56, p 235, These primers were called by Taylor et al (Ref 2): "Eschbach Gasless Delay Detonators"

Refs: 1) Beyling-Drekopf (1936), 232-35
2) W. Taylor et al, BIOS Final Rept **644** (1945), 3-16

Eschka Mixture. A mixt of 2 parts Mg oxide and 1 part dried Na carbonate, used as a reagent for determining sulfur in coal, coke or petroluem used as fuels. Such analysis is routine in the laboratories of plants manufg explosives and acids. We are not describing the test because it is described in the literature, such as in Ref 2

Refs: 1) Hackh's Dict (1944)' 314-L 2) Std-Methods Chem Analysis, Vol **2A**, 1963, pp 1155ff

Escho (Esplosivo) (Ital). See Echos (Esplosivo)

Esclangon, Ernest (1876-1954). French professor and "Membre de l'Institut" who published some important works in the field of Artillery

Ref: Garnier, MAF **28**, 1er fascicule (1954)

Eso. Prefix indicating that an element or radical is substituted for a hydrogen atom attached to a ring atom(Ref 2). The term "eso" was proposed by Baeyer (Ref 1)

Refs: 1) A. Baeyer, Ber **47**, 962 (1884) 2) Hackh's Dict (1944), 314-R

Esop's Test for Efficiency of Detonators. A very brief description of original procedure designed by Esop in 1889 (Ref 1) was given in Vol **1** of Encycl, p XI, but its modifications of 1926 & 1927 (Ref 2) was only mentioned but not described and so was modification of 1930 by Haid & Koenen (Ref 3) described by Marshall (Ref 4)

In the test described in Ref 2, the pellets were made of TNT instead of PA (Picric Acid) of original test (Ref 1), because TNT is less sensitive and more uniform. They were not phlegmatized with oil, as only a limited quantity can be incorporated with the expl, the excess being driven out again on pressing. Most of the reported experiments were carridd out with TNT incorporated with 4 to 14% paraffin wax pressed at 400 kg/cm^2 to pellets of 25mm diam weighing 27g each, but the conclusion was arrived that is better to use talc as a phlegmatizer. Accdg to the proportion of talc, 30 to 36g of the thoroughly incorporated mixture of finely pulverized materials were taken and pressed at 1250 kg/cm^2 to form a pellet 25mm in diam and 25mm high with a hole 7mm in diam and 10.5mm deep for the detonator to be tested. The detonation was judged by exploding the pellet on the brisance meter, such as Copper Crusher Tester described in Vol **3** of Encyl, p C493. The measure of effectiveness of the detonation was the amt of phlegmatizing agent required to reduce the compression of copper crusher from 4mm (caused by unphlegmatized TNT to 1mm (caused by phlegmatized TNT)

In modification of Haid & Koenen (Refs 3&4), a pellet 41 to 42mm high was used, and the hole for the detonator was 25mm deep. Completeness of deton was judged by the effect upon a lead plate, as described under Plate Tests in Vol **1**, of Encycl, p XX. Increasing percentage of talc was incorporated with TNT until the diam of the depression made in the lead plate was only 25mm, same as that of the pellet. It was considered then that the deton was incomplete, and the corresponding percentage of the talc was taken as a measure of the violence of the detonator. Completeness of deton can also be judged by firing the pellet in a lead block similar to that used in the Trauzl Test (Vol **1** of Encycl, p XXV). The enlargement of the hole gave a direct measure of the strength ot the detonator (See also Ref 5)

Refs: 1) K. Esop, Mittheilingen über Gegenstände des Artillerie- und Geniewesens, 1889 2) Jahresbericht Chemisch-technische Reischsanstalt, Berlin **V** (1926), p 112 & **VI** (1927), p 121 3) A. Haid & H. Koenen, SS **25**, 393,433 & 463 (1930) 4) Marshall **3** (1932), p 164 5) Reilly (1938), p 154

ESP. Abbr for End of Sustained Pressure (in Rocketry)

Ref: 1)

Espadon. An early (1945), French, experimental rocket airplane. It was Europe's first postwar rocket-powered airplane. Propellant — nitric acid and furaline

Ref: F.I. Ordway & R.C. Wakeford, "International Missile and Spacecraft Guide," McGraw-Hill, NY (1960), Rocket Airplanes, Manned Missiles and Spacecraft, 147

Esparto Grass, known also as ''Tall Grass''. Any of several grasses, esp *Stipatenacissima*, of S. Europe and N. Africa, used for making paper, cordage, etc. (Refs 1&2). Its use as a cellulose source is described by Ott (Ref 3)
Refs: 1) Hackh's Dict (1944), 314-R 2) ''The American College Dictionary'', Random House, NY (1952), 410-L 3) E.Ott, Edit, ''Cellulose and Cellulose Derivatives'' Interscience,NY, Vol 5, Part 2 (1954), 584-85

Esparto Wax. Hard vegetable wax extracted from esparto grass, having a mp of 75° C and the ability to blend well, emulsify easily, and impart smoothness to products using it
Ref: CondChemDict (1961), 449-L

Espir Powder. Brit proplnt patented in 1885: Na nitrate 60, sulphur 14 & sawdust 26%. It was hygroscopic due to presence of Na nitrate
Refs: 1) Daniel (1902),283 2) Fedoroff & Clift 4 (1946), p 31

Esplodenti: Same as Esplosivi

Esplosioni. Ital for Explosions. They can be lente (slow) o deflagrazioni (deflagrating) or veloci (quick) o detonazioni (detonating)

Esplosivi. Ital for Explosives (plural for Esplosivo)

ESPLOSIVI O ESPLODENTI ITALIANI (Italian Explosives. Summarizing information given in the books listed at the end of this item as Refs 1 to 9, explosives used in Italy can be classified as shown in Table 16
Note to Table 16: Giorgio (Ref 9, p 18 and others divided Esplosivi according to physical, state (*lo stato fisico*) as follows;
Esplosivi solidi: can be subdivided into: *esplosivi fusi o solidificati da fusione* (fused or cast explosives), such as Tritolo (TNT) or its blend with T4(RDX); *esplosivi compressi da polveri* (pressed explosives), such as TNT, Tetryl, PA, *flemmatizzato* (phlegmatized) T4; and *polverulenti scilti* (loose powders) such as Amatol with 80% AN
Esplosivi quasi solvidi (Nearly solid explosives) can be subdivided into: *trafilati o pressati a caldo da miscele colloidali* [wiredrawn (passing "screw press", such as ''sausage machine'', shown in Figs 32 & 33 of Naoum's NG, pp 305-06 and in Fig 45, p 182 of Giorgio (Ref 9) or warm-pressed colloidal mixtures], e.g. *Balistiti, Dinamiti e ottenuti per raffreddamento di miscele pastose* (obtained by cooling dough-like mixtures), such as Amatol contg 60% AN
Esplosivi liquidi: nitroglicerina (NG), nitroglicole (NGc) dinitrotoluene (DNT), esplosivi all'aria liquida o con ossigeno liquido
Esplosivi plastici, such as consisting of blends of solids with viscous liquids, e.g. RDX or PETN, plus oil
Esplosivi gelatinizzati, gelatinized explosives of various consistencies, e.g. Gelatin-dynamites, Gelignite
Esplosivi molazzati- powdered explosives prepd by blending in a ''wheel mill'', called *molazze or macine*, in Italian (see Figs 128 & 129, p 381 in Ref 8). This method of manuf has been used in manuf of BkPdr and some Dynamites

Esplosivi Italiani (arranged in alphabetical order):

Abelli - Vol 1 of Encycl, p A2

Afosite - Vol 1, p A109-L

Ager C, Ager D e Ager E - Vol 3, p C439-R

Albite - Vol 3, p C440-L

Alto esplosivo (High Explosive) - Vol 1, p A141-R

Aluminized Plastic Military Explosive - Vol 1, A148-L

Alvisi - Vol 1, p A157-L

Amatolo - Vol 1, p A162-R and Vol 3 p C440-L

Ammissibili esplosivi (Permissible Explosives) - Vol 1, p A286-R

TABLE E 16

Miscele di gas combustibili in aria	Mixtures of combustible gases (H_2, C_2H_2, CH_4, firedamp) in air				
Miscele di vapori combustili in aria	Mixtures of combustible vapors (C_6H_6, ether, etc) in air				
Miscele di polvere suspense in gas	Mixtures of powders suspended in gas (Al, Mg or coal dust in air)				
Esplosivi solidi	Composti chimici esplosivi(Explosive chemical compounds)	Esplosivi di scoppio o dirompenti (Bursting or brisant explosives): Tritolo (TNT) e esplosivi flemmatizzati (phlegmatized): Pentrite (PETN) e T4 (RDX)	Uso militare (Military use)	Carica granate (Artillery shell charge), Carica mortai (Mortar shell charge),Carica razzi (Rochet charge),Carica mine (Mine charge),Carica bombe aereo (Air bomb charge), Cariche subaque (Underwater charges), Cariche demolizione o distruzione	Esplosivo fuso, Esplosivo compresso, Esplosivo plastico, Esplosivo polverulento (See Note to Table 16)
Esplosivi solidi	Miscele esplosivi (Explosive mixtures)	Esplosivi di scoppio:RDX+TNT and RDX+TNT	Uso militare	Same as above and to add: Cariche illuminanti e Cariche pirotecniche	Esplosivo fuso (See Note to Table 16)
Esplosivi solidi	Miscele esplosivi (Balistiti)	Esplosivi di lancio o propellenti (Propellants	Uso militare	Polveri per cannoni, obici (howitzers) e mortai Polveri per armi portativi (rifles and pistols) Propellenti solidi per razzi (rockets)	See Note to Table 16
Esplosivi liquidi	Miscele esplosivi	Esplosivi di lancio	Uso militare	Propellenti liquidi per razzi	
Esplosivi solidi	Miscele esplosivi	Esplosivi di lancio	Uso civile	Armi personali, armi de caccia (hunting) e pirotecnica	
Esplosivi solidi	Composti chimici esplosivi	Inneschi (Initiators):MF, LA,LST	Uso militare	Cannelli (primers) e detonatori	Instantenei e tardati (delay)

(continued)

TABLE E16 (continuation)

Esplosivi solidi	Miscele esplosivi	Miscela ternaria (triple mixture:MF, $KClO_3$ & Sb_2S_3)	Uso civile	Capsule (caps), detonatori, accenditori (igniters) e chiodature (riveting)	
Esplosivi solidi	Miscele esplosivi e composti	Micce (fuses)	Uso civile e militare	Alenta (Slow) e rapida combustione	
Esplosivi liquidi	Composti chimici esplosivi	NG, NGc DNT DEGDN	Uso militare	In Esplosivi di lancio	See Note to Table 16
Esplosivi liquidi	Composti chimici esplosivi	NG e NGc	Uso civile	in Dinamiti	
Esplosivi solidi	Miscele esplosivi	Esplosivi da mina	Uso civile	per apetto (for open)[normali e subacquei (underwater)] per sottosuolo (for underground)[normali e antigrisou (permissible)]	See Note to Table 16
Esplosivi solidi	Esplosivi nucleari di fusione e di fissione				

Notes to this Table are given on page E127

Ammonal - Vol 1, p A291-R

Ammondinamite - Vol 3, p C440-L

Ammondite- Listed by Giorgio (Ref 9, p 172) as one of the "Esplosivi polverulenti normali" without giving its exact compn

Ammongelatina - Vol 3, p C451-L

Ammonite No 1 - Vol 3, p C440-L

ANS (Antisanzionate) - Vol 1, p A457

Antonite per cava (for quarry) - Vol 1, p A473-L

Antonite per galleria (for tunneling) - Vol 1, p A473-L

Antonite per galleria, extra - Vol 1, p A473-L

Ares. Listed by Giorgio (Ref 9, p 172) as one of the "Esplosivi polverulenti normali" without giving its exact compn

ASN - Vol 1, p A496-L

Astralite - Vol 1, p A499-L

Avigliana 3 o *Nitramite* - Vol 1, p A516-R

Balistite - Vol 2, pp B8-R & B9-L

Black Powder. See Polvere nera in this section

BM Esplosivi. Non-permissible expls manufd by the Mangiarotti Co, Codroipo (Udine). The following are listed in their catalog obtd for us by Dr Omero Vettori, Aulla (Massa Carrara), who also gave their approx compns

BM1 per galleria - TNT, AN & thermite; lt gray pdr [See also Giorgio(1964), p 172]
BM 2 per galleria - inorg nitrates, org nitrocompds and powdered Al; shiny gray pdr
BM a2- AN, TNT, and thermite; brown pdr
BMAC per uso a cielo aperto (for use in open): AN & TNT; brown-yel pdr
BMAS per uso a cielo aperto-(AN, org nitrocompds & metallic pdrs; reddish pdr)[See also Giorgio (1964), p 172]
BM 57 - per uso a cielo aperto: AN & other ingredients; gray-blk powder
Super BM per galleria: org nitrocompds, inorg nitrates and plasticizers; lt green, sl plastic pdr [See also Giorgio (1964), p 172]
Gomma BM Italiana, listed by Giorgio (1964), p 170, among "Esplosivi al 56-60% NGL con 4-5% nitrocellulosa, 30-35% nitrato, ammonico and other ingredients"
BM esplosivo da mina con nitroglicerina listed by Belgrano (1952) in Table of p 281 as formulation 22 contained: NG66, Collodion cotton 4, AN 20, wood flour 2.7 & Na nitrate 7.3%. Its Trauzl value 510cc, gap test value 27cm and detonation velocity 5000 m/sec
C-2 (Polvere) - Vol 2, p C1-R
Cadinite - Vol 3, p 440-L
Canopus - Vol 2, p C39-R
Carica cava direzionale or Carica a punta vuota. Shaped Charge in Engl, Charge creuse in Fr and Hohlladung, in Ger. See Giorgio (1964), p 180 and "Shaped Charges" in Vol 4, pp D442-D454
Cava M esplosivo. A mining expl manufd by the SGIMC (Società Generale per l'Industria Mineraria e Chimica), Milano (No compn is given in their catalog)
Chedditi- Vol 2, p B117-R (under Binary, etc) and Vol 3, p C159(Table)
Cloramite - Vol 3, p C329-R
Cordite: See Polvere C2
Cratiti: Vol 3, p C555-L
Cremonita - Vol 3, p C556-L
Cyclonite. See T4

DINAMITI ITALIANI(Esplosivi Italiani da Mina con Nitroglicerina). Italian Dynimites can accdg to Belgrano (Ref 6, p 85) and Giua (Ref 8, p 338), be subdivided into two classes: 1) *Dinamiti a base inerte* (Dynamites with a chemically inactive base) and 2) *Dinamite a base attiva* (Dynamites with active base)

To the 1st class belong compns consisting of NG absorbed on "farina fossile" (fossil flour) to which belong kieselguhr (guhr) and tripoli. The only known compn was *dinamit n 1*, which contained NG75 & guhr 25% (Ref 8, p 339)

To the 2nd class belong compns consisting of NG absorbed on chemically active substances which can be either "inorganiche" (nitrates, chlorates or perchlorates) or "organiche" (NC, carbon or powdered woody substances, called "materie legnose polverulente" in Ital, but usually both inorganic and organic substances are present. The resulting mixts are powdery *(polverulenti)* and correspond to Amer ''Straight Dynamites". Giua (Ref 8, p 340) gives three examples: *Dinamite Numero 0:* NG 70, materie legnose 5 & $NaNO_3$ 25%; *Dinamite Numero 1:* NG 70, materie legnose 8, $NaNO_3$ 18 & **carbon** 4%; *Dinamite Numero 3:* NG 25, materie legnose 19, $NaNO_3$ 54 & $NaCO_3$ with ocher 2%. Belgrano (Ref 6) lists in Table on pp 282-83 thirty formulations of *Esplosivi da mina con nitroglicerina, polverulenti,* with NG content ranging between 4 and 15%. Their properties: Trauzl value, *distanza colpo* (gap test value) and *velocita detonazione* (detonation velocity) are also given in Table

To the 2nd class also belong *dinamiti gelatinosi (gelatinizzati)* (Gelatinized Dynamites), which can be subdivided into *dinamite-gomma* (called by Giorgio in Ref 9, p 169, *gomma A italiana) e gelatine esplosive o gelatine dinamiti.* One of the dinamiti-gomme consists of NG 92 & CC (cotone collodio) 8% which corresponds to Blasting Gelatin while the 2nd formulation: NG 83.0, CC 5.0, Na nitrate 8.0, farina di legno (woodflour) 3.7 & Na carbonate with ocher 0.3% does not correspond to any Amer Dynamite (Ref 8, p 343). The formulation for *gelatina-dinamite* given in Ref 8, p 344 is: NG 74, CC 5, Na nitrate 15.5, wood flour 5 & Na carbonate 0.5%. It might be called Gelatin Dynamite. Belgrano (Ref 6) lists in Table on pp 280–81 under the title ''Esplosivi gelatinosi con nitroglicerina", twenty-nine formulations with NG content from 7 to 92% and CC content from 1 to 8%. Their properties Trauzl, gap test value and detonation velocity are also given in the Table

Non-freezing Dynamites, known as *dinamiti antigel o incongelabili* are discussed by Belgrano (Ref 6, pp 87-8). They contain in addition to NG the ''sostanze congelanti a basse temperatura" (substances freezing at low temperature), such as nitroglycol (NGc), iso-butylglycerin tetranitrate, metriol trinitrate and liq DNT

Permissible Dynamites *(Dinamiti antigrisoutosi)* consist, accdg to Giorgio (Ref 9, p 176) of *grisoutina* contg 13.2% and 10% of NG. Belgrano (Ref 6, p 165) lists *grisoutina* contg NG 10, CC 0.25 & AN 89.75%. Giua (Ref 8, p 375), lists *Grisoutina C*, manufd by Nobel di Avigliana, as contg NG 13.2, CC 0.25, AN 86.35, Na carbonate 0.1 and fossile flour 0.1%

Many Italian Dynamites which bear various individual names starting with A and ending with Z are listed here

Giorgio (Ref 9, pp 169-72 does not have a section entitled ''Dinamiti'', but places them under ''Esplosivi da mina per uso civili'' and subdivides them into ''Esplosivi gelatinosi'' and ''Esplosivi pulverulenti". He spells ''pulverulenti'', while Belgrano and Giua spell ''polverulenti''

Among *esplosivi gelatinosi* Giorgio lists:
1) *Esplosivi al 90-94% di NGL,* to which belongs *Gomma A italiana* which contains 6-8% CC (collodion cotton)
2) *Esplosivi al 56-60% di NGL,* With the rest being AN 30-35% and small amts of other additives. To these belongs *Gomma BM italiana*
3) *Esplosivi al 36-42% di NGL,* with 1-3% NC, 0-6% DNT with TNT, the rest being Amm nitrate, Na nitrate, wood flour and a dye. To these belong ''gelatine dinamiti italiani'' GDIMT and GDS
4) *Esplosivi al 20-26% di NGL,* with 1% NC, about 10% aromatic nitrocompounds or organic combustible materials, Amm nitrate (with or without Na nitrate) To these belongs the *gelignite SA*

Among *esplosivi pulverulenti,* Giorgio lists:
1) *Classe dei ''Super Pulverulenti'',* which contains some NG, about 70% of nitrates (mostly of Amm), tritolo (TNT), or pentrite (PETN) or T4 (Cyclonite). To this class belong Ital expls *Superdinamon, Super BM galleria, Titan C and Vulcanite*
2) *Esplosivi pulverulenti normali,* which contains 4-6% NG, with 80/20 ratio-nitrates/TNT and some other additives. To these belong Ital expls: Dinamon I and S', BMI, BMAS, Antonite, Vulcania, Ares, Titan, Ammondite and Martia α and γ

Explosive properties of the above expls **are** listed in Table on p 178 of Ref 9

Giorgio also lists on p 178 ''esplosivi geosismici'', (seismograph types), some of which are based on NG

The following expls listed by Giorgio: ''*esplosivi economici pulverulenti*'', (p 172), miscele nitroto-nafta (AN/fuel oil mixtures) (p 173), ''esplosivi a base di clorati" (p 174), esplosivi "Slurry" (p 174) and ''esplosivi nella lotta antigrandine'' (expls to fight against hail) (p 180) do not belong to ''dinamiti'' because they do not contain NG

As an exception may be mentioned *Gelatine Chedditi,* which contains 5-7% NG (p 174 of Ref 9)(Refs are listed at the end of the section entitled ''ESPLOSIVI ITALIANI.

Dinamon. See Vol **3**, p C440-L

Dinamoni I ed S! Powdery expls, manufd by the Consortio Fabricanti Dinamiti. They are listed by Isso (Ref 7, p 32) without giving their compns. Giorgio (Ref 9, p 172) lists them among ''esplosivi pulverulenti normali'', which are based on 80/20 ratio of nitrate/TNT and might contain 4-6% NG

Dinitronaftalina esplosivi. See MST or Nougat, Schneiderite and Siperite

Echos or Escho-Vol **2**, p C440-L

Esplosivi all'aria liquida (Liquid Air Explosives). See Belgrano (Ref 6) p 156

Esplosivi antigeli o Esplosivi incongelabili (Non-freezing Explosives). See here under DINAMITI ITALIANI in this section.

Esplosivi a base di clorati. See Giorgio (Ref 9, p 174) and Giua (Ref 8, p 390)

Esplosivi antigrisoutosi (Permissible Explosives). See Belgrano (Ref 6, pp 165-72) and Giorgio (Ref 9, pp 177-78)

Esplosivi a base di nitrato ammonico. See Belgrano (Ref 6, pp 162-65) and Giua (Ref 8, p 372)

Esplosivi a base di ossigeno liquido o Ossiliquite (Liquid Oxygen Explosives). See Giua (Ref 8, p 403) & Giorgio (Ref 9, p 165)

Esplosivi a base di perclorato di ammonio. See Giua (Ref 8, p 400)

Esplosivi a base di perclorato di potassio. See Giua (Ref 8, p 400)

Esplosivi flemmatizzati. Expls such as PETN or RDX phlegmatized with wax, etc

Esplosivi gelatinosi al binitrotoluolo. See Esplosivi da mina gelatinosi al binitrotolulo

Esplosivi gelatinosi con nitroglicerina. See Esplosivi da mina gelatinosi con nitroglicerina

Esplosivi geosismici o Esplosivi per prospezioni sismiche(Seismic Prospecting Explosives). See Giorgio (Ref 9, p 179) and Giua (Ref 8, p 405)

Esplosivi con guaina (Sheathed Explosives). Accdg to Dr Camillo Belgrano of Codroipo (Udine), as communicated to Dr Omero Vettori of Aulla (Massa Carrara), Italian sheaths consist of mixts of Ca fluoride and sulfate with Na bicarbonate. These expls have been used in Sicilian sulfur mines
Ref: Dr Omero Vettori, private communications July 1 and August 17, (1964)

Esplosivi da guerra: 1) Cyclonite 95 & wax dyed red 5%; 2) Cyclonite 22.0, AN 73.4 & wax 4.6% (Vol **2** of Encycl, p B117-R); 3) TNT 30-90, PETN 65-5 & Al powder 5-30%; part of TNT can be replaced by DNN (Dinitronaphthaline) [CA **45**, 1770 (1951)]; 4) Cyclonite 75-90 & Al powder 25-10% [CA **44**, 6130 (1950)]

Esplosivi da mina: 1) Cyclonite or PETN 75 & nitro-isobutylglycerintriacetate 25% [CA **45** 1709 (1951)]; 2) TNT (of which up to 50% can be substituted by DNN) 30-90, Cyclonite 65-5 & Al powder 5-30% [CA **45**, 3160 (1951)]

Esplosivi da mina gelatinosi al binitrotoluolo. Eleven formulations are listed in Table on p 286 of Belgrano (Ref 6)

Esplosivi da mina gelatinosi con nitroglicerina. Twenty nine formulations are listed in Table on pp 280-81 of Ref 6

Esplosivi da mina polverulenti senza nitroglicerina,molazzati. Thirtv formulations are listed in Table on pp 281-82 of Ref 6

Esplosivi da mina con residuati di polveri di lancio, Fourteen formulations are given Table on p 287 of Ref 6 (See also Vol **3**, p C460-L.)

Esplosivi da mina senza nitroglicerina. Fifty formulations are given in Table at end

of Belgrano's book (Ref 6)
Note: Several formulations for Esplosivi da mina taken from Belgrano are listed in Vol **3**, p C451

Esplosivo plastico: Cyclonite 85-89, petrolatum 10-12, thiocol 0.5-2 & gylcerophthalic resin 0.5-2% [CA **43**, 7688(1949)]

Esplosivo P - Vol **3**, p C440-L

Esplosivo S - Vol **3**, C440-L

Esplosivo S20 - Vol **3**, p C440-L

Esplosivo Sc, Same as Schneiderite.

FNP (Fillers for bombs). Two formulations are listed in Vol **2**, p B118-L

Evangelidi (Esplosivo) (1904): $KClO_3$ 62.5, K ferrocyanide 30.0, charcoal 4.5 & gylcerine 3.0% (Ref 8, p 398-L)

GDI. Gelatinous mining expl: NG 60, CC 3.5, wood flour 5.2, Na nitrate 30.5, Na carbonate 0.5 & red ocher 0.3% (Ref 6, p 280, formulation 9)

GDIM: NG 38, CC 2.3, AN 50,9, Na nitrate, 6, red ocher 0.5, oil 2 & Na carbonate 0.3% (Ref 6, p 281, form 17)

GDII: NG 43.2, CC 2.3, wood flour 7, Na nitrate, 45.5, Na carbonate 1 & yellow ocher 1% (Ref 6, p 280, form 3)

GD2: NG 48.3, CC 2.7, wood flour 5.8, Na nitrate 42.7 & Na carbonate 0.5% (Ref 6, p 280, form 4)

Gelatina 808 - Vol **3**, p C440-L

Gelatina dinamita NO - Vol **3**, p C440-L

Gelignite - Vol **3**, p.C440-L

Gelignite all'ammonio - Vol **3**, p C440-R

Gelignite SA - Vol **3**, p C439-L

Geo. Gelatinous mining expl: NG 77.5, CC 5, wood flour 5, Na nitrate 12 & Na carbonate 0.5% (Ref 6, p 281), formulation 27)

GEOM: NG 57, CC 3.5, AN 29, woodflour 3.5 & Na nitrate 7% (Ref 6, p 281, formulation 19)

Geosamer. A seismic prospecting expl listed by Giorgio (Ref 9, p 180) without giving its compn

Gomma A: NG 92 & CC 8% (Ref 6, p 281, form, 29)

Gomma B: NG 82.5, CC 5.5, wood flour 3, Na nitrate 8.5 & Na carbonate 0.5% (Ref 6, p 281, form 28)

Gomma BM. See under BM esplosivi

Gomma incongelabile: NG 65-72, CC 5-6, liquid TNT 7-8 & AN 15-22% (Vol **3**, p C440-R)

Grisoutina al 10% - Vol **3**, p C451- R

Grisoutina al 13.2% - Vol **3**, p C451-R

Grisoutina C - Vol **3**, p C451-R

Idrolita - Vol **3**, p C440-R

Imperialita - Vol **3**, p C440-R

MABT - Vol **2**, p B118-L

Macarite - Vol **3**, p C440-R

Mangiarotti's Mining Explosives - Vol **3**, p C440-R

Manlianite - Vol **3**, p C440-R

Martia **α e γ**. Mining expls manufd by Sorlini (Antonio), Ghedi-Brescia, N Italy. Giorgio (Ref 9), lists them on p 172 under "Esplosivi polverulenti normali", which are based on 80/20-nitrate /TNT and contain 4 to 6% NG

MAT - Vol **2**, p B118-L

MBT - Vol **2**, p B118-L

MNBT - Same as Siperite

MST - Same as Nougat

Nitramite - Same as Avigliana 3

Nitronaftita - Vol **2**, p B118-L

Nougat or MST - Vol **2**, p B118-L

Piombite - Vol **3**, p C440-R

Pentrite - Ital for PETN

Plastigels - Vol **2**, Table 4, p C159 (two formulations are listed)

Polvere "Cannel". See under Cannel Explosives in Vol **2**, p C25-R

Polvere nera (Black Powder). Belgrano (Ref 6, p 175) gives two formulations for powders used in mining: 1) KNO_3 75, S 10 & C 15% and 2) KNO_3 70, S 18 & C 12%. For polvere nera di caccia (BkPdr for sporting compn, given on p 292 of Ref 6 is: KNO_3 75 to 78, S 9 to 12 & C 12 to 15%

Polveri Italiani Senza Fumo (Italian Smokeless Propellants) are divided by Giorgio (Ref 9, pp 204-10) into the following groups:
1) Polveri alla sola nitrocellulosa (a una base) (Single Base NC Propellants), which include:
Polvere italiana per cartucce cal 8, per mitragliatrici (machine guns): NC (12.8%N) 97, Centralite 2 & DPhA (diphenylamine) 1%; comes in the form of small tubes 0.75-0.1 x 2.2 mm, which are graphited. Its heat of expln and temp of expln are probably the same as for Poudre B, namely 950kcal/kg & 2700° C
2) Polveri a doppia base (con NGL) (Double Base Nitroglycerin Propellants), which are subdivided into:
a) *Polveri senza solvente volatile* (Solventless Propellants), which include: *Balistite Normale* and *Balistite a basso titolo*. Their compns are already given in Vol **2** of Encyl, pp B8-R & B9-L. Their heats of explns are as given in Ref 9, p 205: 1350 kcal/kg & 1250, resp and temp of expln is 3500° C for the normale but not given for the other propellants
b) *Polveri con solvente volatile* (Propellants with Volatile Solvent), which includes:

Polvere C-2. Its compn was already given in Vol **2**, p C1-L under the title C-2 (Polvere). Its heat of expln 960 kcal/kg and temp of expln 2700 ° C are given in Ref 9, p 206

Solenite: NG 36, NC 61 & mineral oil 3%; heat heat of expln 1150 kcal/kg, but temp of expln not given. It used to be manufd by Fontana Liri Co for use in armi portatili (pistols and rifles) (Ref 9, p 206)

Giorgio also lists on p 207:
Balistite attenuata al binitrotolueno, which is listed on p B9-L of Vol **2**, as *Balistite attenuata o Polvere CG13*

Polvere alla Centralite: NG 24, NC 69 & Ethylcentralite 7%. Here Centr serves as a gelatinizer, cooler and stabilizer

Balistite al 36% di NGL: NG 36, NC 62 & Et Centr 2%; heat of expln 1100 kcal/kg

Polveri alla nitroacetilcellulosa (NAC): NG 27-32, NC 66-63 & Et Centr 7-4% . Heat of expln 860-960 kcal/kg

Polveri alla ftalide: NG 28-32, NC 64-62, phthalide 4-3, Et Centr 3-2 & vaseline 1-0.5%. Heat of expln 860-960 kcal/kg
Note: Ballistites in the form of cords are sometimes called Filiti

In addn to the above proplnts may be mentioned the following described in Vol **2** of Encycl:
Balistite attenuata all'acetilcellulosa, developed by Giua and described in Ref 5, p 157 and manufd by the Società Dinamite Nobel: NG25, NC60, acetylcellulose 10 & Et Centr 5% (p B9-L

Balistiti o Filiti: a) NC (12.25% N) 64.0, DEGDN 35.0 & Et Centr 1%; b) Nitrocellulose acetate (10%N) 63.5, DEGDN 27.0, cellulose acetate 5.0 & EtCentr 4.5%; and c) NC 55.0, Metriol Trinitrate 40.5, metriol triacetate 2.0 & EtCentr 2.5% (p 37-L)

Polveri utilizzate per razzi (Rocket Propellants) are mentioned by Giorgio (Ref 9, p 208), without giving their compns. It is only stated that they must be slow burning and that they contain NG (but in small amt), NC, EtCentr and some K perchlorate in order to improve oxygen balance

Polveri da caccia (Sporting Propellants). They can be, accdg to Giorgio (Ref 9, p 210), either: a) straight NC, completely gelatinized; b) NC mixed with oxidizers and gelatinizers; and c) NC-NG proplnts of Ballistite types

Romite cava and Romite galleria. Mining expls manufd by Polverificio Stacchini and listed by Isso (Ref 7, p 32) without giving their compns

Sabulite - Vol **3**, p C440-R
Schneiderite o Esplosivo Sc - Vol **2**, p B118-L
Sheathed Explosives. See Esplosivi con guaina
Siperite - Vol **2**, p B118-L

Sismite. A seismic prospective expl mentioned by Giorgio (Ref 9, p 180), without giving its compn. Dr Omero Vettori of Aulla (MC) gave its compn as An 48, TNT 32 & Tetryl 20% (Vol **3**, p C439-R)

Solenite. See under Polveri a doppia base con solvente volatile
Solfite - *Vol* **3**, p C440-L
Stabilite - Vol **2**, p B118-L
Super BM per galleria. See under BM esplosivi
T4. Ital for Cyclonite (RDX). See Vol **3**, p C614-R & C615-L
T4 Mixtures - Vol **3**, p C627-R
Tionite - Vol **3**, p C454-L
Toluolammonal - Vol **2**, p B118-L
Tritolita. Mixture of Tritolo (TNT) with T4
Tritolital - Vol **2**, p B118-L
Tritolo. Ital for TNT
Tutamite - Vol **3**, p C439-L
Umbrite A - Vol **3**, p C440-R
Umbrite B - Vol **3**, p C440-R
Vibrite - Vol **2**, p B118-L
Victorite - Vol **2**, p B118-L
Vulcan - Vol **3**, p C440-R
Vulcania DB - Vol **3**, p C439-R
Vulcania DBS- Vol **3**, p C439-R
Vulcanite P - Vol **3**, p C439-R

Refs on Esplosivi Italiani: 1) R. Molina, "Esplodenti", Hoepli, Milano (1930) 2) E. Piantanida, "Chimica degli Esplosivi e dei Gas di Guerra", Tipo-Litografia della R. Accademia Navale, Livorno (1940) 3) A. Mangini, "Quaderni di ChimicaIndustriale No 14, Esplosivi", Patron, Bologna (1947) (Lithographed) 4) C. Caprio, "Corso di Esplosivi", Scuola Salesiana), Roma, Vol **1** (1948) & Vol **2** (1949) 5) M. Giua & C. Giua-Lollini, "Dizionario di Chimica Generale e Industriale", UTET, Torino, Vols 1,2,& 3 (1948-1950) 6) C. Belgrano, "Gli Esplosivi", Hoepli, Milano (1952) 7) A. Izzo, "Manuale del Minatore Esplosivista", Hoepli, Milano (1954) 8) M. Giua, "Trattato di Chimica Industriale, "UTET, Torino, Vol **6**, Part **1** (1959) 9) C. Giorgio, "Tecnica degli Esplosivi" Del Bianco, Udine (1964)

Essai au chock du mouton. See Épreuve au chock du mouton

Essai au clou. See Nail Test in Vol **1** of Encycl, p XIX

Essai de déflagration (Deflagration Test). It may be conducted by the following two methods:

1) Place a 0.2g sample of an expl or proplnt. into a test tube the lower part of which is inserted in a heating bath (oil, glycerin, molten tin, etc) preheated to 100°. If the sample does not immediately decompose, continue to heat the bath in such a manner that the temperature of liquid rises at the rate of 5° per minute. Note the temp at which the sample deflagrates (or ignites, or detonates) and repeat the operations. Following are some values obtd by this method: BkPdr 280 to 285°; Dynamites No 1 (NG75 & guhr 25%) decomp with evolution of nitrogen oxides at 200 to 210°; LA(Lead Azide)> 235°; MF(Mercuric Fulminate) 175 to 180°; NC, poorly stabilized, 130° to 140°, and NC, well stabilized 180 to 185°

2) Place a 0.2g sample into a test tube and immerse the lower part of the tube into the liquid in a bath maintained at constant temp

of 100°. If the sample does not decompose, raise the temp of the bath about 10° and repeat the operation with a new sample and if no decompn takes place at 110°, test a new sample at 120°, etc. When the temp is reached at which the sample deflagrates instantaneously, lower the temp of the bath 5° or 10° and repeat the test with new sample. Several tests at various temps should be made for each expl or proplnt and the exact temp and time interval in seconds between the insertion of the tube in the bath and the deflagration should be observed. Then the time vs temperature data should be plotted on graph paper and the curve drawn. The assymptote to this curve will give the minimum temperature of deflagration (or ignition, or detonation). Following are some values obtd by this method: Dynamite No 1 192 to 197°; LA 320 to 330°, MF 137 to 142° and NC, well stabilized 174 to 179°

Note: If the original temp of 100°, of the bath will cause immediate decompn of the sample, lower the temp 10°, or 20°, or even lower and start with a new sample, etc. These tests will give values below 100° and such expls are considered unstable

Ref: Vennin, Burlot & Lécorché (1932), 211-12

Essai à la friction (Friction Test). Sensitivity to friction may be detd by one of the following simple tests:

a) Place a grain of expl in a porcelain mortar and see if a rubbing with a pestle will cause deflagration or detonation of the grain

b) Place a small sample on a tile or on an anvil and strike it with a glancing blow by a wooden hammer

Other Friction Sensitivity Tests are listed and briefly described in Vol 1 of Encycl, p XIII

Expls sensitive to any of the friction tests should be considered as dangerous to handle and if used in gaseous or dusty coal mines should not be tamped, even with a wooden tamper (bourroir de bois)

Refs: 1) Vennin, Burlot & Lécorché (1932), 212 2) Pepin Lehalleur (1935), 75-6

Essai au mortier éprouvette (Ballistic Mortar Test). The apparatus prescribed for this test by La Commission des Substances Explosives consists of a small stationary mortar mounted at a 45° angle and the test is conducted in the following manner: A fixed charge of an expl or proplnt is loaded thru a muzzle on top of a small amt of clay (argile) previously placed at the bottom of mortar (See Note 1, below). A steel cylindrical missile of fixed weight (such as 15kg) is inserted into the muzzle and the charge is fired by means of an electric detonator. The distance (range) the missile traverses is measured and compared with the range obtd when a standard expl or proplnt is fired in the same manner. It is considered that the work performed by an explosive (travail de l'explosif) is proportioned to the range.

Note 1: Accdg to Vennin et al (Ref), a calcd amt of clay is placed in the mortar to assure the charge of expl to be always at constant density

Note 2: This test as well as Essai au pendule balistique, described here and on pp 269-72 of Vennin et al is not recommended in France for brisant expls, but rather for proplnts and BkPdr

Ref: Vennin, Burlot & Lécorché (1932), 189

Essai au pendule balistique (Ballistic Pendulum Test). Under the title "Mesure de la puissance balistique au pendule", Vennin et al (Ref 2) describe a test which is not exactly the same as the tests used in USA and described in Vol 1, of Encycl, p VII. The French apparatus (Pendule balistique) reproduced here in Fig 31, consists of a weapon (mortar, rifle or cannon) A, attached to a pendulum suspended on a knife edge and a cast iron receptor "B", attached to another pendulum. Some lead is placed inside the receptor. A charge of propellant and a missile are placed inside the gun A and when fired the missile hits B causing deflection of its pendulum! At the same time the recoil of A caused the deflection of its pendulum in opposite direction. The greater the deflections, the greater are the velocities of projectiles. Historical background of such pendulum was outlined by Cranz (Ref 1) and also briefly in Vol 2 of Encycl, p B6-R, under Ballistic Pendulum Chronographs

A rather complicated method of calcula-

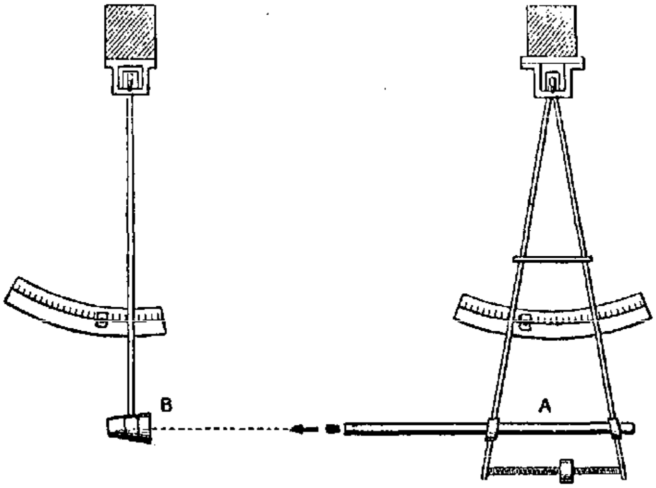


FIG E31

tion of various values is given in Ref 2, pp 270 & 271, but it is stated that the tables prepd in France give in most cases the required values without resorting to calculations

Refs: 1) Cranz 3 (1927), 39-51 2) Vennin, Burlot & Lécorché (1932), 269-72

Essai dans la terre (Test in the Ground). See under Cratering Effect Tests in Vol 3 of Encycl, p C554-R

ESSAIS d'EXPLOSIFS (French official Tests of Explosives).

Fourteen Tests which have been devised and used after WWII by the Laboratore de la Commission des Substances Explosives were described by Médard (Ref 8, pp 323-55)

These tests include:

1) **Densité de tassement** (Packing Density). This density was designed to determine at what density explosives should be packed into cartridges, boxes, etc so that the volume would remain the same after the items had been transported. This density is called by Pepin Lehalleur (Ref 3, p 62) ''la densité de chargement"

For its determination, weigh 50g of previously dried expl or proplnt to the nearest cg and transfer it to a Kraft paper cylindrical container, 30mm in diam. Pack the contents by allowing the cylinder to drop vertically on a wooden surface from a height of 50mm and repeat this operation in rapid succession for one minute (about 120 drops). Then measure the height of material in the cylinder to the nearest 0.5mm. Repeat the tamping until no change in height will take place. Determine the final volume in cc and calculate the density by dividing 50 by volume (Ref 8)

2) **Reprise d'humidité de l'explosif en vrac** (Hygroscopicity of an Explosive in Bulk) (The term ''en vrac'' means in bulk, not packed or arranged, but loose)

For its detn spread evenly on the bottom of tared crystallizing dish a previously dried 50.000g sample, which passed thru a sieve with openings of 1mm. Place the dish in a desiccator over aq sulfuric acid of 11° Bé at 15° (sp gr 1.083). Prepare a 2nd and a 3rd sample and place them over acids of 21° Bé at 15° (sp gr 1.171) and 31° Bé at 15° (sp gr 1.274), respectively. Reweigh the dishes with the samples after 24 and 48 hours of storage and express the results in gain per 100g of sample (Ref 8)

Note: The above dilute acids form atmospheres in the desiccators with relative humidities of 94%, 85% and 65%, which are considered as being ''very humid'', ''rather humid'' and ''slightly humid'', compared to the French climate

3) **Reprise d'humidité en atmosphère saturée**

d'eau des explosifs encartouchés (Hygroscopicity of Cartridged Explosives in an Atmosphere Saturated with Water)

This test designed for cartridges of mining explosives wrapped in plain or paraffined paper is conducted in the following manner:

Place an 8-cm layer of sawdust mixed with an equal amount of water in a zinc-lined wooden box and arrange about 2cm above the sawdust a perforated wooden shelf. Weigh the paper covered cartridges to the nearest cg and lay them horizontally on the shelf. Close the box and determine the gain in weight of cartridges after 12 and then after 21 days of storage (Ref 8)

4) **Exsudation par étuvage** (Exudation on Storing).

This test designed to determine whether expls, such as DNT, TNT or Dynamites would exude any liquid when stored in hot climates, is conducted in the following manner:

Weigh to the nearest mg a square piece (5 x 5 cm) of porous cardboard, 2mm thick, and place on top of it a tared Kraft paper container, 30mm in diameter and 0.2mm thick. Fill the container with a prescribed weight (not given) of an explosive to be tested and place the ensemble in an oven, thermostatically controlled at 40 ±1° or at 50 ±1°. Leave for 21 days and reweigh the container as well as the cardborad. The loss in weight of the container (P) corresponds to the weight of exudant plus the volatiles, while the gain in weight of the cardboard (p) corresponds to the amount of exudant. The difference, P-p, corresponds to the loss in volatiles (Ref 8)

5) **Combustion en gouttière de 20mm** (Combustion in a 20mm Trough). Also called **Épreuve de chauffage de la gouttière** or **Épreuve de propagation dans une gouttière de 20mm de diametre.** The purpose of this test is to determine the inflammability of a substance when it is spread in a thin layer. The test is one of the **Épreuves de combustion** (Combustion Tests), also called **Aptitudes à l'inflammation.** The data obtained from this test are useful from the point of view of safe handling of the explosive

In this test a sample of an explosive or propellant is spread evenly in a thin layer on the inside surface of a semi-cylindrical steel trough(gutter), 20mm diameter (or 10mm diam for substances that burn extremely fast), 2mm in thickness and 200cm long, provided with marks at 0, 50, 100, 150 and 200cm. By applying an open flame (such as a Bunsen Burner, a candle or a piece of cotton saturated with alcohol) to the end of the trough marked 0, it is possible to determine whether or not the substance is flammable. If it is very flammable, it will ignite immediately when touched by the flame (as NC or loose proplnt). If it is not very flammable (as TNT) a few seconds (or minutes) will elapse before the substance catches fire. This time interval is detd with the aid of a stop watch

As soon as the substance catches fire the source of heat is withdrawn and observation is made as to whether the combustion continues in the part of the trough which was not heated . If the combustion does continue, the times necessary to reach the 50, 100cm, etc marks are recorded and the velocity of propagation of the flame in cm per sec, is calculated. If the time is below 5 sec for the entire length of the trough, it is advisable to use a chronograph, such as of Le Boulangé described in Vol 3 of Encycl, p C307-R

During the progress of burning, it is also advisable to observe the appearance of the flame (color, smoke etc) and its dimensions , If there is any residue left, its color and the size should be recorded. The results are compared with those obtained by burning a standard explosive or propellant (Ref 8) (See also Ref 5)

6) **Combustion en tas conique** (Combustion in a Conical Pile), also called **Épreuve de sensibilité à l'inflammation** ou **Aptitude à l'inflammation** [Sensitivity to Ignition (or Inflammation) Test]. The purpose of this test is to ascertain the ease of ignition (inflammation) of an expl or a proplnt in air. This is important from the point of view of safety in handling

The test is conducted by placing about 1kg (or more) of a sample to be tested on a noninflammable surface (such as beaten ground or a cement floor) in such a manner that a conical heap is formed. After measuring the height and diameter of the cone, its base is touched with either an open flame (such as a Bunsen burner or cotton soaked with alcohol) or a hot object (such as a red-hot wire). Observation is made as to whether the substance catches fire instantaneously or whether some time elapses before ignition. This time interval, as well as the time required for the entire pile to be consumed, is recorded by means of a stop watch or a chronograph, such as a Le Boulangé. It is also necessary to observe the appearance and the size of the flame and whether or not there is any residue left. If so, its size and color are recorded. The results are compared with those obtained with standard explosives or propellants (Ref 8) (See also Ref 5)

7) **Sensibilité au choc du petit mouton** [Sensitivity to Shock with a Small Ram (Hammer)] which corresponds to Amer "Impact Sensitivity with a Small Weight"

The official test, as conducted in "l'appareil de chute réglamentaire du Services des Poudres", described by Muraour (Ref 1), consists essentially in allowing a steel hammer (mouton) of a definite weight (1,2 or 5kg) to drop from a predetermined height onto a small, thin-walled cap (capsule de Bourges) contg a small sample of the expl to be tested. If the impact of the hammer causes a complete or partial detonation, the test is repeated with a fresh sample, but with the hammer allowed to fall from a lower height. The tests are continued until a sufficient number of samples is tested to permit a curve to be drawn from which it is possible to determine the height at which the probability of an expl is 50%. By multiplying the weight of the hammer (in kg) by the height of its fall in meters, a value expressed in kg-m is obtd. This value is known as "insensibilité de l'esplosif" and the greater it is, the less sensitive is the explosive. For example, the values are for TNT 8, PA 6, Tetryl 3, RDX 2.4, LA 1.1 and MF 0.5 (Ref 8)

8) **Sensibilité au choc du gros mouton de 30kg** (Sensitivity to Shock with a Big Hammer of 30kg) (Impact Sensitivity Using a 30kg Hammer)

This test supplements the previous test especially in cases of fairly insensitive explosives which give many incomplete detonations. It has been claimed that when larger samples are used (such as 100g) and a larger hammer, it is possible to differentiate the "incompletes" by determining the amount of explosive which remains undecomposed. In some cases when "incompletes" were obtained with the small apparatus, complete detonations were observed when the larger apparatus was used

The test is conducted by placing a 100± 0.1g sample, which has been previously dried and sifted thru a screen with 1mm openings, on a steel rectangular anvil of apparatus and then allowing the hammer to drop from a predetermined height. More detailed description of the test is given in Ref 8

Note: The apparatus "gros mouton" was first described by Commission des Substances Explosives in MP **15**, 216 (1909)

9) **Courbe de compressibilité des explosifs solides** (Curve of Compressibility of Solid Explosives). The curve detd by the test was described below gives the relationship between the density and pressure applied to powdered or granulated expls; it is useful in cases in which loading of ammunition is done by pressing

The test consists essentially in weighing to the nearest 0.01g a 10g sample of expl, transferring it to a steel mold of 30mm diam and pressing to a predetermined value by means of a hydraulic press. Then the sample is removed, its density is detd and operations are repeated using fresh samples and different pressures, until sufficient data are obtd for drawing pressures-density curve (Ref 8)

10) **Transmission de la pression dans un explosif pulvérulent** (Transmission of Pressure in a Powdered Explosive). It is known that when a column of a powdered expl is subject to pressure during loading of a cartridge the region nearest the piston has a higher density than the rest of the charge. This nonuniformity of distribution of density varies with the explosive and the size of its particles (grains). The purpose of the test described below is to determine under which conditions the most uniform distribution of pressure and density are obtd with a particular explosive

The test consists essentially in transferring a 100g sample of powdered explosive, weighed to the nearest 0.1g, into a long steel cartridge. After setting the cartridge in a vertical position in a hydraulic press, the predetermined piston pressure is applied to the top of the column of expl. Then the entire column is removed and carefully divided into three sections of equal length by cutting with a non-ferrous metal knife. Determine the density of each section to the nearest thousandth and repeat the operation varying the pressure, temperature, speed of compressing and other conditions of test until a desirable distribution is obtd (Ref 8)

11) **Sensibilité à l'amorce** (Sensitivity to Priming). The test is designed to determine the minimum amount of MF and the smallest booster required to detonate a given explosive

The test is conducted by laying horizontally on a lead plate(resting on a steel plate), a Kraft paper cylinder, 30mm in diam, loaded with 50g sample of expl to test, and provided with a MF detonator and a booster. After firing the cartridge, the damage caused to the lead plate is observed. If the detonation is complete, the impression in the part of the plate farthest from the deton will be somewhat deeper. In this event it will be necessary to repeat the test with a smaller detonator (or perhaps with a smaller booster). If no damage to the lead plate is observed after firing the cartridge, the detonation is not complete which means that a larger detonator is required (Ref 8)

12) **Transmission de la détonation à distance** (Transmission of Detonation at a Distance), also known as **Coefficient de self-excitation** (Coefficient of Self-excitation) or **Aptitude à transmettre la détonation à distance** (Capability to Transmit the Detonation at a Distance). This test is known in the USA as **Gap Test**, **Sympathetic Detonation Test** or **Detonation by Influence Test.** The French modification of the test described by Médard (Ref 8, pp 342-44) was earlier described by Dutour (Ref 6). This test is described in Vol 3 of Encycl, p C390 as ''Coefficient de Self-excitation" (CSE) and gap values for several expls at different densities are given

13) **Travail spécifique relatif** (Specific Relative Work) or **Coefficient d'utilisation pratique (CUP or cup)** (Coefficient of Practical Utilization). This test corresponds to **Trauzl Lead Block Test**, proposed in 1903 for detn of power (or strength of expls (See Vol **1**, p XXV), but is conducted in a different manner as briefly described in Vol **1**, p IX & X and in Vol **3**, p C390, where CUP values for different expls are given. The French test was standarized in 1912 by the CSE (Commission des Substances Explosives) and a detailed description was given in the book of L. Vennin, E. Burlot & H. Lécorché, "Les Poudres et Explosifs", Ch. Béranger, Paris (1932), pp 171-74

It must be noted that the formula $\frac{50 \times 100}{C}$ for calcn of CUP given on p X of Vol **1** is wrong. It should be $\frac{15 \times 100}{C}$

14) **Vitesse de détonation mesurée par la méthode de Dautriche** (Velocity of Detonation Determined by the Method of Dautriche). This method was invented in 1906 by H. Dautriche (1872-1914) and has been used not only in France but in other countries, including USA. It is described as item H in Vol **3** of Encycl, pp C311-R & C312, under CHRONOGRAPHS

Refs for ESSAIS: 1) H. Muraour, MAF **12**, 560-61 (1933) 2) E. Burlot, MAF **13**, 113 (1934) 3) Pepin Lehalleur (1935), 62 4) L. Médard, MAF **22**, 597-605(1948) 5) E. Burlot & P. Tavernier, MP **31**, 38 (1949) 6) M. Du Tour, MP **31**, 74 (1949) 7) M. DuTour, MAF **24**, 577 (1950) 8) L. Médard, MP **33**, 323-55 (1951)

Essence C. Colorless liquid not miscible with water, but misc with alc, acet, or petr ether; density 0.690 at 20/20°, $n_D 1.3883$ at 20°, range of distn 70 to 90°. Can be used as gelatinizer for NC

Ref: P. Tavernier & M. Lamouroux, MP **38**, 81-2 (1956)

Essence de mirbane. Fr for Nitrobenzene or Oil of Mirbane, described in Vol **2**, of Encycl p B45-R

Essigäther. Ger for Ethyl Acetate

Essigsäure. Ger for Acetic Acid

Esso flame thrower. An apparatus that throws a spray of liquid fuel that ignites in the air

"Essolube". Trademark of Esso Standard Div of Humble Oil & Refining Co, New York, NY, for detergent motor oils for all types of gasoline and high-speed Diesel engines
Ref: CondChemDict (1961), 449-R

Essons Poudre. Gunpowder manufd in France during the revolution (end of the 18th century): AN 75, sulfur 12.5 & charcoal 12.5%
Ref: Van Gelder & Schlatter (1927), 782

Essons Potassium Chlorate Gunpowder. A BkPdr in which part or total of K nitrate was replaced by K chlorate, manufd under Berthollet's direction until a disastrous expln took place in 1788
Ref: Van Gelder & Schlatter (1927), 782

"Essowax". Trademark of Esso Standard Div of Humble Oil & Refining Co, New York, NY, for fully refined paraffin wax available in slabs and in liquid form in wide range of melting points and hardness
Ref: CondChemDict (1961), 450-L

Essorage (Wringing). The purpose of this operation is to remove the bulk of the water from NC, crystallized TNT, etc. It may be conducted either by centrifuging or by filtering thru a Nutsch
Refs: 1) Pascal (1930), 231 2) Vennin, Burlot & Lécorché (1932), 588-92

"Estane". Trademark of B.F. Goodrich Chemical Co, Cleveland, Ohio for a thermoplastic polyurethane material, suitable for wire and cable jacketing, fuel hose and tanks, belting etc
Ref: CondChemDict (1961), 450-R

Ester and Esterification, An organic salt formed by interaction betw an alcohol (base) and an org acid. The formation of an ester by dehydrating or catalytic agents is called *esterification*
Refs: 1) Hackh's Dict (1944), 315-L
2) E.E. Reid, IEC **40**, 1596-1601 (Sept 1948) "Esterification", with 168 refs; and in Sept numbers of IEC of the succeeding years.
3) Groggins (1952), 596-650 (Esterification)
4) Kirk & Othmer, 2nd edit **8**(1965), 313-56 (Esterification), 356-65 (Ester Interchange), 365-83 (Esters, Organic)

Esters, Aliphatic. Gelatinization power of various aliphatic esters is discussed by G. Desseigne & J. Tranchant in MP **42**, 197-203 (1960)

Esters, Nitric, Liquid. Impact sensitivities of several liquid nitric esters were detd by Médard (Ref 2) by conducting the tests with the apparatus described by Muraour (Ref 1) and with the use of the "capsules de Bourges". The compds tested were: NG, TMGDN (Trimethyleneglycoldinitrate); NGc (Nitroglycol); 1,3-BuDDN (1,3-Butanedioldinitrate); 1,4-BuDDN; DEGDN (Diethyleneglycoldinitrate) (pure); DEGDN (commercial), known in France as NEO and TEGDN (Triethyleneglycoldinitrate)
Refs: 1) H. Muraour, MAF **12**, (1933)
2) L. Médard, MP **31**, 131-43 (1949)

Esters, Nitric of Polyhydroxydicarboxylic Acids. Krauz & Majrich (Ref) prepd in the laboratory the dinitrates of the following acids: *trihydroglutaric*, $(CHOH)_3(CO_2H)_2$; *mucic*, $(CHOH)_4(CO_2H)_2$; and *saccharic*, $(CHOH)_4(CO_2H)_2$, using the following method: One part of org acid was dissolved in 2-2.5ps of nitric acid (sp gr 1.52) at 0° and to this was slowly added 2 ps of oleum (25% SO_3) while maintaining the temp at 0°. After holding the mixture for several hours at 0°, it was drowned in ice-water and the precipitated solids separated by filtration, washed with cold water and dried. It was found that the above acids and their nitrated compds are good stabilizers for NC, and in this respect surpass *tartaric acid*, $(CHOH)_2(CO_2H)_2$ or its dinitrate

Ref: C. Krauz & A. Majrich, Collect Czech ChemCommun **11**, 639-48 (1939) & CA **35**, 2721 (1941)

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