Copies of the “Encyclopedia of Explosives And Related Items” can be obtained by requesting CD ROM from the:

National Technical Information Service
(formerly Clearinghouse)
US Department of Commerce
Springfield, Virginia 22151

1-800-553-6847 USA only
703-605-6000
www.ntis.gov/FCPC

The contents of these volumes are UNCLASSIFIED

The distribution of these volumes is UNLIMITED

Neither the US Government nor any person acting on behalf of the US Government assumes any liability resulting from the use or publication of the information contained in this document or warrants that such use or publication will be free from privately owned rights.

All rights reserved. This document, or parts thereof, may not be reproduced in any form without written permission of the Energetics and Warhead Division, WECAC, TACOM, ARDEC, Picatinny Arsenal

Library of Congress Catalogue Card Number: 61-61759
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 (Pb₃O₄) 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 pdtr 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) Naoum, 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 w/ 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". D₂; gas, mw 4.03; fp 13.95° K, bp 20.57° K, d of liq 1.69 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 1). It has found use in military weapons as a constituent of thermonuclear bombs.

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

Deuterium Oxide or "Heavy Water", D₂O; mw 20.03; fp 3.79°, bp 101.41°, d 1.10452 at 25°, max d at temp 11.23° is 1.10602; Qₙₐ₉ₐᵥₗᵈₑᵣᵢₐₙ = 70414 cal/mol (liq), Q₊ₐₚ₉ₚₑᵣᵣᵢₐₙ = 10846 cal/mol; G.N. Lewis of Univ of Calif concd a large quantity of water to a small amt of nearly pure D₂O by electrolysis in 1931. D₂O 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) Partridge (1950), 176 2) Kirk & Othmer 6, 2nd edit (1965), 898-909

Deuterium Peroxide, D$_2$O$_2$; mw 36.03;
"heavy" hydrogen peroxide; is formed by passing "heavy steam" (D$_2$O vapor) thru a mixt of deuterousulfuric acid (D$_2$SO$_4$) & potassium persulfate (K$_2$S$_2$O$_8$) and condensing the vapor. The soln of D$_2$O$_2$ in D$_2$O thus obtained is fractionated, and pure D$_2$O results
Refs: 1) Partridge (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) Hackb’s (1944), 262 2) Cond-ChemDict (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 water 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

"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


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
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 predetermined 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 charges, 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 knife 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
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 to 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 charge. 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 charge thru the piston. This feature was important in the design of the pin since it was desired to main-
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).

c) **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).

d) 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 Bombler", 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** (Gomlinele, Starch Gum, British Gum, Vegetable Gum or Amylin), \( \left( C_6 H_{10} O_5 \right)_x \)\( mw (162.14)_x \); mp dec, d 1.0384 at 20°. White, or sl yellow amorphous pd; sol in w & dilute alc; insol in absolute alc or in ether. Can be prep by the partial hydrolysis of starch, corn, potato, wheat, sago or cas-
sava. For its prep, any of the above products is moistened with dil nitric or hydrochloric acid and then heated to 140-170\(^\circ\). After the acid & w are evaporated, the dried product is powdered and sieved. It can also be prep by careful heating of dry starch. (Refs 6, 7, 8 & 10). If starch is hydrolyzed with Bacillus macerans, some crystalline products, such as \(\text{\textalpha}-\text{dextrin}, (\text{C}_6\text{H}_{10}\text{O}_5)_6\) are obtd (Ref 7). Crystalline products may also be prep 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 comps (Ref 11). Davis (Ref 5, pp 62ff) gives numerous examples. As a binder & a desensitizer it has been used in primer comps, as for example in a friction primer used for an airplane flare (Ref 5, p 454), or in a compon 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 "Nobellite" & "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 water 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 amnt 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 phpt indicator and titrate with 0.1N NaOH soln until a pink color is produced. Run a blank.

\[
\text{% Acidity} = \frac{(0.60N(A-B))}{C}
\]

where:
\[
N = \text{normality of NaOH soln}
\]
\[
A = \text{ml of NaOH soln used in the test}
\]
\[
B = \text{ml of NaOH soln used in the blank}
\]
\[
C = \text{wt of dextrin in 100 ml filtrate}
\]

6) Reducing sugars, as dextrose. Prepare Gooch crucibles with a mat of prepared asbestos ca 0.25-inch thick. For prep 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 obt.
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, prep as above.
Prepare copper sulfate soln by dissolving 34.63 g of CuSO₄.5H₂O 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 25 ml 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 the 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

5) Davis (1943), 64 & other pp listed on p 200; also 425 & 454 6) Pérez Ara (1945), 355

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 propmts. 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 crystals.
It has been used in Belgian expls Casteau, described in Ref 2, p 117 and this Encycl, Vol 2, p CB3-R.

Dextrose, d-Glucose, Dextroglucose, Corn Sugar or Grape Sugar, HOCH₂(CHOH)₄CHO; mw 180.16; col crys. 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 penta-nitrate.


Dextrose- or d-Glucose Pentanitrate, known also as Nitroglycerine, (C₆H₇O(NO₂)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 prep'd, accdg to Daniel (Ref 2) by nitrating glucose with equal parts of concd nitric and sulfuric acid; its expl props are weak.

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

Refs: 1) Beil 31, 146  2) Daniel (1902), 523 (Nitroglycerine)  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 chgs 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, 10g dry powdered dextrose polymer to 175g of mixed acid (HNO₃ 35%, H₂SO₄ 58% & H₂O 7%). The temp of nitrating was 23°. After stirring for 60 min the slurry was poured into 1 liter of cold w and the resulting ppt transferred into a Büchner funnel. After applying suction the nitrates 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 12.6g. 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 propellant, developed by ADL, for rocket motors. Its compn and props are given as Unit No 598 in conf Propellant Manual SP14A/M2 (1962).

DGV or DDP70). A modified double-base propellant, developed by ADL, for rocket motors. Its compn and props are given as Unit No 634 in conf Propellant Manual SP14A/M2 (1962).

A Hercules cast rocket propellant by the same description is described in conf Propellant Manual SP14A/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 slight differences in meaning for particular compds, or accordg to customary usage. Ref: CondChemDict(1961), p346

Diacetoldehyde 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, \((\text{Pb}_2\text{ac}_2)[\text{BrO}_3]_2\); mw 788.35, O 20.29%; thin lfts, mp deflgr at 165°5 + 5°. Weinland & Paul (Ref 1) observed that during the prepn of lead bromate, \(\text{Pb}(\text{BrO}_3)_2\cdot\text{H}_2\text{O}\), 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, \((\text{Pb}_2\text{ac}_2)[\text{C}_6\text{H}_2(\text{NO}_2)_3\text{O}]_2\cdot\text{H}_2\text{O}\); mw 974.73, N 8.62%; lemon-yl prisms, deflgr on heating Diacetato-triplumbo-nitrate, \((\text{Pb}_2\text{ac}_2)[\text{NO}_3]_4\); mw 987.75, N 5.67%; granular cryst aggregates Oxalato-diplumbo-perchlorate, \((\text{Pb}_2\text{ox})(\text{ClO}_4)_2\cdot\text{H}_2\text{O}\); mw 755.40, O 31.77%; deflgr weakly (Ref 1) Oxalato-diplumbo-nitrate, \((\text{Pb}_2\text{ox})(\text{NO}_3)_2\cdot\text{H}_2\text{O}\); mw 662.50, N 4.23%; leaflets Trioxalato-tetraplumbo-perchlorate, \((\text{Pb}_4\text{ox}_3)(\text{ClO}_4)_2\cdot\text{H}_2\text{O}\); mw 1381.90, O 28.94%; six-sied prisms

NOTE: ac stands for CH₃COO- and ox stands for -OOC.CO.O-


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

Diacetone; Diacetone Alcohol; 4-Hydroxy-4-methyl-pentane-2; or 4-Hydroxy-2-keto-4-methylpentane (called Diacetonalkohol; Dimethyl-acetonyl-carbinol; 2-Methyl-pentanol-(2)-on-(4); or Methyl-(β-oxo-isobuty1)-keton in Ger), \(\text{CH}_3\text{COC}_2\text{H}_2\text{CH}_2\text{OH}\); mw 116.16, col, pleasant-odorod liq fr p -42.8°, bp 160° at 760 mm, flash p 170°F, d 0.9406 at 20°, vapor pressure 1.1 mm at 20°, viscosity 0.032 poise at 20°, np 1.42146 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 cong a carbonyl group, as an additive to NG, which acts as an acceler in the gelatinization of NC.


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
Diacetotetrazacyclooctane, $\text{C}_9\text{H}_{16}\text{N}_4\text{O}_2$, 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-tetraza
cylooctane, 

$$
\begin{align*}
\text{H}_2\text{C} & \overset{\text{N} (\text{NO}_2)}{\text{N}} \overset{\text{CH}_2}{\text{CH}_2} \\
\text{CH}_3 \cdot \text{CO, N} & \overset{\text{NOC, CH}_3}{\text{CH}_2} \\
\text{H}_2 \text{C} & \overset{\text{N} (\text{NO}_2)}{\text{N}} \overset{\text{CH}_2}{\text{CH}_2}
\end{align*}
$$

m w $290.24$, N $28.96\%$, crys (from boiling nitromethane), mp 265-66 $^\circ$C, was obtd as one of the products of hexamine nitrolysis, but more specifically by treating the 3-
nitroso-7-nitroderiv with HNO$_3$ & (NH$_4$)$_2$ SO$_4$ at 2-3 $^\circ$. The dinitroderiv was converted in 25% yield to HMX by overnight treatment with 99% HNO$_3$


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

Diacetoxy-diazapentane, $\text{C}_7\text{H}_{14}\text{N}_2\text{O}_4$, 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, $\text{CH}_2\text{COO, CH}_2\text{N(NO}_2)$

$$
\begin{align*}
\text{CH}_2 & \overset{\text{3}}{\text{CH}_2} \\
\text{CH}_3\text{COO, CH}_2\text{N(NO}_2) & \overset{\text{1, 2}}{\text{5}}
\end{align*}
$$

ch $280.20$, N $20.00\%$, crys (from acet + alc), mp 90-92 $^\circ$ & 103-04 $^\circ$, was obtd from a mixt of methylene-

dinitramine in formalin, anhyd Na acetate & acetanhydride warmed to 95 $^\circ$ 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 spatuila and burned like
cordite (Refs 1 & 2)


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-
triazahexa

Diacetoxy-tetrazanonane, $\text{C}_9\text{H}_{20}\text{N}_4\text{O}_8$, may be considered the parent compd for its tetra-
nitro deriv but is not used to prepare it: 1, 9-Diacetoxy-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), $\text{CH}_3\text{COO, CH}_2\text{N(NO}_2)\cdot\text{CH}_2\text{N(NO}_2)$$

$$
\begin{align*}
\text{CH}_2 & \overset{\text{5}}{\text{CH}_2} \\
\text{CH}_3\text{COO, CH}_2\text{N(NO}_2) \cdot \text{CH}_2\text{N(NO}_2) & \overset{\text{9, 8, 7, 6}}{\text{5}}
\end{align*}
$$

col crys (from Nm or acetone), mp 186.5-
187.2 $^\circ$, was prepd by reaction of DPT (1, 5-endomethylene-3, 7-dinitro-1, 3, 5, 7-
tetrazacyclooctane), acetic anhydride & 99.6% HNO$_3$ over 40min at 44 $^\circ$. 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 dest-
royed 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-Dinitroxyc-2, 4, 6, 8-
tetranitro-2, 4,-
6, 8-tetrazanonane (qv)


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)


& CA 47, 12095(1953)(chromatographic props)

Diacetoxyltriazoleheptane, C$_2$H$_{17}$N$_3$O$_4$, may be considered the parent compd of its tri-nitro deriv but is not used to prepare it:

1,7-Diacetoxyl-2,4,6-trinitro-2,4,6-triazoleheptane; 1, 7-Diacetoxyl-1, 3, 5, 7-tetramethylene-2, 4, 6-trinitramine; or 2, 4, 6-Trinitro-2, 4, 6-triazazoleheptane-1, 7-diol Diacetate (Code name BSX), CH$_3$COO.CH$_2$.N(NO$_2$)$_2$.CH$_2$\(^{4}$ N(NO$_2$)

CH$_3$COO.CH$_2$.N(NO$_2$)$_2$.CH$_2$; 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 prpd 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 prpd 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 prpd to give a dinitro deriv which is a very powerful & very sensitive expl.


Dinitroxy-BSX or 1, 7-Dinitroxy-2, 4, 6-trinitro-2, 4, 6-triazoleheptane, O$_3$N.CH$_2$.N(NO$_2$)$_2$.CH$_2$\(^{4}$ N(NO$_2$)

CH$_3$COO.CH$_2$.N(NO$_2$)$_2$.CH$_2$; mw 354.24, N 23.73%; col plates (from CCl$_4$) mp 154-55°, was prpd by nitratng BSX with 99% HNO$_3$ at 0°, one hr at 20°, and drowning the soln in water. It was obtd also by nitratng 1-acetoxyl-7-ethoxy-2, 4, 6-trinitro-2, 4, 6-triazoleheptane with 99% HNO$_3$ at 0°, 200 min at 20°, and drowning in water (Ref 5, 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


1, 7-Diacetoxyl-1, 3, 5-trimethylene-2, 4-dinitramine. See under Diacetoxyl-diazapentane.

Diacetylaminophenetrol, C$_{10}$H$_{11}$NO$_6$, may be considered the parent compd of its mononitro deriv but is not used to prepare it: Diacetilnitraaminophenetrol or 3-Amino-6-nitro-1, 2, 4, 5-benzzenetetrol Diacetyl Deriv. (CH$_3$CO$_2$)C$_6$(OH)$_2$NH$_2$(NO$_2$)$_2$; mw 286.20, N 9.79%; its yel ndls (from alc), mp darkens at 208-10° & defigr at 214°, was prpd by treating nitraminophenetrol
Diacytelynedicarbonic Acid or Hexadiyne-dioic Acid (called Hexadiniidesäure; Butadiin-dicarbonsäure or Diacetylen-dicarbonsäure in Ger), HOOC.C.C.C.COOL; mw 138.08, O 46.35%; rhombic tablets (from eth + petr eth) with 1H2O, mp darkened at 100°, exploded at 177°, changed in light to purple mass; was prepd by oxidation of the Cu compd of propionic 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 Dibhydate, It yel monoclinic ndls (from w or aq eth & petr eth), mp darkened at 95-100°, decomposed on further heating w/o expln. The crystals 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)

Diacytelylene or Butadiyne (called Diacetylen or Butadien in Ger), HC.C.C.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°, nD 1.4386 at 0.8°; can be prepd by the action of cupric chloride soln on cuprous acetylide, followed by treatment of the Cu diacetyl-ylene, thus formed, with dil HCl; and by action of excess alcoholic KOH at 150-60° on butadiene tetralobromide; and by other methods (Refs 1 & 2)

The critical pressure of expn of Diacytelylene is 30-33mm; the critical concn (by vol %) of it in mixts at 700mm with other gases is 22-25% with N, 37% with NH3, 16.5-17% with acetylene, 39% with natural gas, 35% with CO2, 30% with CO, and 30% with H (Ref 4)

It readily yields expn metallic derivs, such as Silver salts, Ag2C4, citron-yel ppt, very expl; and AgC4H4+AgNO3, citron-yel ppt, explodes by friction; and with iodine in KI it forms the very expl compd Diodo-diacytelylene (qv) (Ref 1). See also Ref 3

Diacytelylene Dihydroperoxides. Organic peroxides which contain two acetylenic groups in conjugation with two hydroperoxyl 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 cmpds 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:

\[
\text{HOO} \quad \text{OOH}
\]

\[
\text{C}_6\text{H}_10 \quad \text{C} = \text{C} = \text{C} = \text{C} = \text{C}_6\text{H}_10; \\
7 \quad 8 \quad 8 \quad 7^1
\]

mw 278.34, O 22.99%, col ndls (from benzene + pentane), mp 96-97° dec; was prepd by reacting in the cold (0°) H2O2, H2SO4 benzene & anhydrous ether, adding 7,71-dicyclohexyl-7,71-butadiyniol-6,6 (mp 173°) and allowing the temp to rise to 25°

3, 8-Dimethyl-4, 6-decadiyn-3, 8-dihydroperoxide,
It has found use as a nitrating agent.
A. Pictet used it to prep Tetranitroacetyl-diphenylamine (See Vol 1, p A58)
Its Ag salt, called Diargentic Acetonitrile, (AgO)2N(OH)(OOC.C.H3), col crystals, explodes about 172°, was prepared by dissolving AgNO3 in glacial AcOH
Refs: 1) Beil 2, 271, [175] & [382]

Diacetyl Peroxide or Acetyl Peroxide (called Diacetylperoxyl or Acetylperoxyl in Ger), CH3CO.O.O.COCH3; mw 118.09, O 54.20%; col ndls (from ether), mp 30°, bp 63 at 21mm, flash p (OC) 113°E; explodes on further heating & on shock, sol in alc & eth; sl sol in w; can be prepared by action of acetyl anhydride or acetyl chloride on Na2O2, BaO2 or H2O2 in etherial soln. A relatively safe procedure for its prep 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 phthalate, 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 Lallement(1935), 137 2) O.J. Walker & G.L.E. Wild, JCS 1937, 1132-36 & CA 31, 8508-09(1937)(Thermal & photo-
chem decompn of acetyl peroxide)
3) L.F. Fieser & A.E. Oxford, JACS 64, 2060-65(1942)(Prepn of diacetyl peroxide and the alklylation of para quinones)
4) M.S. Kharasch et al, JACS 65, 15-17(1943) & CA 37, 1385(1943)(Decomp of acetyl peroxide in aliphatic acids) 5) M.S. Kharasch et al, JOC 10, 386-93(1945) & CA 40, 1783(1946)(Decomp 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)

**Dialkylacetlenes.** A convenient method for the prepn of dialkylacetlenes, RC=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:

<table>
<thead>
<tr>
<th>Compound</th>
<th>°C/bp</th>
<th>mm d at 25 °C</th>
<th>nD25</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diethylacetylene</td>
<td>74.5</td>
<td>600 0.7263</td>
<td>1.4112</td>
</tr>
<tr>
<td>Dipropylacetylene</td>
<td>116.7</td>
<td>498 0.7474</td>
<td>1.4225</td>
</tr>
<tr>
<td>Dibutylacetylene</td>
<td>106.3</td>
<td>80 0.7673</td>
<td>1.4311</td>
</tr>
<tr>
<td>Dimethylacetylene</td>
<td>110</td>
<td>23 0.7816</td>
<td>1.4374</td>
</tr>
<tr>
<td>Ethylbutylacetylene</td>
<td>118.5</td>
<td>498 0.7501</td>
<td>1.4230</td>
</tr>
</tbody>
</table>

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,

\[
\begin{align*}
\text{CH}_3 & \quad \text{N} \quad \text{N} \\
\text{C} & \quad \text{N} \quad \text{H} \\
\end{align*}
\]

mw 113.13, N 61.91%; hygr crs (from benz, and methylene chloride-petr eth), mp 41-43°C, bp 88°C at 1mm; was obtd by dissolving the hydrochloride (mp 202-95°C) 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 

*Pircate*, C₉H₁₀N₂O₇, crs (from alc), mp 186-87°C; *Hydrobromide*, C₃H₇N₅Br, crs (from 98% isopropyl alc), mp decmpd 188-89°C; and *Nitrate*, C₃H₇N₅O₇, crs (from alc), mp 153-5-54.5°C (*Refs 1 & 2*)

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


**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 dialkyl-
phthalates completely. These facts form the basis for an analysis of proplants conng dibutyl- or diamyl-phthalates, centralite & possibly phthalide

Procedure: Extract a sample of proplant 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 hydroxyethylbenzoate. Dry the ether extract, which contains phthalates & centralites, and weigh the residue. Treat this mixed residue with hot aq 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% H₂SO₄ and heat to recover (2) to phthalide. Extract the phthalide with ether. Determine NG present by difference.

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

N¹, N⁴-Dialky-triethylene-tetranitramines. These are compds of the general formula R-N(NO₂)-CH₂-CH₂-N(NO₂)-CH₂-CH₂-N(NO₂)-CH₂-CH₂-N(NO₂)-R. They were patented by Blomquist and Fiedorek (Ref 3) for use in propellants as explosive, practically non-volatile, plasticizers for NC. The dialky-triethylene tetranitramines are obtained, together with compds of general formula R-N(NO₂)-CH₂-CH₂=N=N(O)-O-CH₂-CH₂-N(NO₂)-CH₂-CH₂-N(NO₂)-R, when N-alkylthylene dinitramines are treated with ethylene dibromide as described by Franchimon & Klobbie (Ref 2).

As an example of such tetranitramines may be cited N¹, N⁴-dimethyl-triethylene-tetranitramine (qv)


Diallylbenzidine and Derivatives

Diallylbenzidine or 4, 4¹-Diallyldiamino-diphenyl (called Diallylbenzidin or 4, 4¹-Diallyldiamino-diphenyl in Ger), CH₂·CH₂·CH₂·HNC₆H₄·H₄·C₆H₄·NH·CH₂·CH₂·CH₂; mw 264.36, N 10.60%; may be considered the parent compd of its nitroderivs but is not used to prep them

N¹, N⁴-Diallyl-3, 5, 3¹, 5¹-tetranitrobenzidine,
C₃H₅·HNC₆H₄·(NO₂)₂·C₆H₄·(NO₂)₂·NH·C₆H₅; mw 444.36, N 18.91%; orn-red nds (from phenol or salicylic acid methyl ester), mp 205⁰ (dec); was prepd by heating at 100⁰ in a sealed tube 3, 5, 3¹, 5¹-tetranitro-4, 4¹-dimethoxy (or dioctoxy) -diphenyl with allylamine in alcohol (Refs 1 & 3)

N, N¹-Diallyl-N, N¹, 3, 5, 3¹, 5¹-hexanitrobenzidine,
(C₆H₅H(NO₂)₂·N(NO₂)·CH₂·CH₂·CH₂)·2; mw 534.35, N 20.97%; nearly col crystals, mp dec ca 100⁰; was obtd by treatment of the tetranitro deriv w/ concd HNO₃ (Refs 2 & 3)


Diallylether or Allyl Ether (called Diallyl-ether in Ger),
CH₂·CH₂·CH₂·O·CH₂·CH₂·CH₂; mw 98.14, O 16.30%; col liq, bp 94.3⁰, d 0.805, vap d 3.38, nD 1.4163 at 20⁰ 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 H₂SO₄, with explosive violence.

Refs: Beil 1, 438, [477] & [1883]

2) J.B. Niederl et al, JACS 53, 3393-94 (1931) 3) Thorpe l(1937), 258
4) Kink & 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 B128-1, where this compd is called erroneously Diallylpentaerythritol. The Dinitrate of this Ether was suggested as a possible gelatinizer for NC
Diallylphosphite, \((\text{CH}_2\text{CH}_2\text{CH}_2\text{O})_2\text{P.OH}\); mw 162.05, \(\Delta 29.62\%\); yell oil, bp 58-62°C at 0.5-mm & 98°C at 2-mm, d 1.0793 at 20°C, \(n_D 1.4430\) at 20°C; can be prepd by reaction of allyl alcohol & \(\text{PCl}_3\) 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 prep 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).


Diallyl-o-phthalate, \(C_6H_4(CO_2\text{CH}_2\text{CH}_2\text{CH}_2)\); mw 246.25, \(\Delta 25.99\%\); syrupy liq, bp 183°C at 15-mm, d 1.118 at 20°C, \(n_D 1.5189\) at 25°C, Qcomb at C$_v$ 1653.5 kcal/mol, Qform 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$_2$SO$_4$ (Ref 3).


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, i.e. the decompn of a compd by an electric current. Electrodialysis is the combined process of dialysis & electrolysis using semipermeable membranes to confine the soln under treatment.


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

**Diamides.** These comps are the dibasic acid derivs of ammonia or amines, and contain two -CONH₂ groups. Numerous combinations of dicarboxylic acids, diamines, and amino acids have been interacted. Copolymers containing various proportions of two or more dicarboxylic acids have been prepd (Ref 2). The diamide of carbonic acid is called urea, H₂NCONH₂. It is a product of animal metabolism and is prepd industrially by reaction of CO₂ with excess NH₃ 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)NHCH₂[AN(NO₂)]₉CH₂A′.NHC(O)R

(where R & R′ are alkyl radicals)
A & A′ are alkylene radicals, and n is 2-4)

as suitable main expl changes in shells, missiles & blasting devices, or as O donors & modifiers for proplnt fuels. These nitrodiamides are prepd by condensing the appropriate polynitrodiamines, derived from nitroso disocyanates & strong mineral acids, with acid anhydrides. The polynitrodiamides prepd & cited are as follows:

N, N′-diacetyl-3, 6-dinitrazo-1, 8-octanediimine, mp 178-80°;
N, N′-diacetyl-3-nitrazo-1, 5-pentanediamine, mp 197-98°;
N, N′-dibexanoyl-4-nitrazo-1, 7-heptanediamine;
N, N′-dipentanoyl-4, 7-dinitrazo-1, 10-decanediamine; and
N-propionyl-N′-butanoyl-3-nitrazo-1, 5-pentanediamine

Compds of the general structure

RC(O)R′(NO₂)₂.C₂H₅.C(NO₂)₂CH₂.A. 
N(NO₂).C(O)R where R is alkyl and A is alkylene, were also patented by Frankel & Klager (Ref 4) for use as HE chgs or proplnt additives. These nitrodiamides are prepd by heating the appropriate dinitrodiamines with acyl anhydrides and treating the product with HNO₃. Thus, the following N-nitrodiamides 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′-dibexanoyl-6, 6-dinitro-1, 11-undecanediamine


**Diamidides.** These are comps having the structure N-C-N-C-N, such as NR:CPN:NR₉: CPh:NR₉ and NRR₉:CPN:CPN:NR₉, 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 NRR₉:CPN:CPN:NR₉ yield normal salts, but di- & tri-amidides derived from NR:CPN:NR₉:CPN:NR₉ readily decomp by acids (HX) to NH₃:CPN:NR₉ & X:CPN:NR₉. 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 NH₂: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

Diamin. Ger designation for Ethylene Dinitrate (EDD)

Diamines. Aliphatic amines may be regarded as derived from hydrocarbons by replacement of two H atoms by two amino (-NH₂) groups. The most important aliphatic amines are the ω-diamines of the normal hydrocarbons, NH₂(CH₂)n-NH₂, some of which are produced by putrefaction of animal matter. They are also prep'd by a) heating alkyene dibromides with alc ammonia at 100° b) reduction of dicyanides, dioximes, and dihydrozones of dialdehydes or diketones, or dinitrosoamines c) from dicarboxylic acids thru Hofmann or Curtius degradations and d) hydrolysis of alkylene-diphthalimidines. 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 prep'd 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 prep'n of Nitro-Diamines of the structure

\[ \text{R}^1 \text{H} \quad \text{NO}_2 \quad \text{H} \quad \text{R}^1 \]

\[ \text{R}^2 \quad \text{C} \quad \text{N} \quad \text{CH}_2 \text{C} \quad \text{CH}_2 \text{N} \quad \text{C} \quad \text{R}^2 \]

\[ \text{R}^1 \quad \text{R} \quad \text{R}^1 \]

where R may be aryl, alkyl, H, or X(balogen); R¹ may be H, CH₂OH, or alkyl; and R² may be aryl, H, aralkyl, alkyl, or CH₂OH by treating nitrates with primary amines. These compds were unobtainable by interaction of nitro alkanes & methylol amines (Ref's: 1) Thorpe 1(1937), 314(Aliphatic); 315 (Aromatic) 2) Sidgewick, Org Chem of N(1937), 43 (Aliphatic); 79 (aromatic) 3) Kirk & Other 1X(1954), 185 (Reactions with succinic acid & succinic anhydride); 827 (Aromatic as solv for polyacrylonitrile)

Diamino-1-alkene. Doss & Bost (Ref 1) describe the prep'n of 1, 2-bis(dimethylamino) ethylene (bp 38-40° at 15mm & nD 1.4649), its hydrogenation in a Parr bomb at 40lb/sq inch to the ethane (nD 1.4170) and the use of the ethane deriv as a component of hypergolic & fast-burning rocket fuels

Mahan (Ref 2) also prep'd 1, 2-diamino-1-alkene derivs and tested their rocket fuel props of hypergolic & ignition delay. The product N, N, N¹, N¹-tetramethyl-1, 2-diaminoethylene was found to have an ignition delay of 3.6 millisec at 75°F with white, fuming HNO₃ as compared to 6.9 millisec for N, N, N¹, N¹-tetramethyl-1, 2-propanediamine, the best of many hypergols similarly tested. Also prep'd and tested are the hypergols N, N, N¹, N¹-tetraethyl-1, 2-diaminoethylene and N, N¹-dimethyl-N, N¹-dietethyl-1, 2-diaminoethylene (Ref's: 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-aryl (or 3-pentyl)-s-triazolo[4, 3-c]-s-triazine,
DIAMINOANISOLE AND DERIVATIVES

Diaminoanisole or Methoxyphenylentediamine
(called Diamino-phenol-methyläther or Diamino-anisol in Ger), (H₂N)₂C₆H₃O·CH₃;
mw 138.17, N 20.28%. Three isomers are
described in Beil, sometimes as the HCl
salt: 2, 3-Diamino-anisole, C₇H₇N₂O +
2HCl, crysts (from alc + eth), mp ca 250°;
to gm liq (Ref 1); 2, 4-Diamino-anisole,
ndls (from eth), mp 67-68°(Ref 2);
C₇H₁₀N₂O + 2HCl, crysts, sol in alc + eth
(Ref 3); and 3, 5-Diamino-anisole, C₇H₁₀N₂O
+ 2HCl, crysts, mp dec ca 220°, its addn
compd with 1, 3, 5-TNB, C₇H₁₂N₂O +
C₆H₃N₃O₆, dk red-bm 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, (H₂N₂)₂C₆H₅(NO₂).
O.CH₃; 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, (H₂N)₂C₆H₅(NO₂)₂O.CH₃;
mw 228.17, N 24.56%. Two isomers are
described in the literature:
3, 5-Dinitro-2, 4-diaminoanisole, red-violet
cysts (from alc or Nitrobenz), mp 250-55°
& 260-61°, sol in alc or Nitrobenz; diffe sol
in alc or AcOH; was prepd by heating on a
water bath 2, 3, 5-trinitro-4-aminoanisole
with alcoholic NH₃ and by other methods
(Refs 1, 3, 4 & 5)
3, 5-Dinitro-2, 6-diaminoanisole, gm-red ndls
(from ethyl acet), mp 237°; diffe 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
antifoggent in photographic Ag halide emuls.
ions. 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
2775211(1957) & CA 51, 4187(1957)
5) Kodak Soc Anon, BelgP 553885(1957) &
CA 54,134(1960)

Trinitrodiaminoanisole, C₇H₇N₅O₇, not found
in Beil or in CA thru 1961

DIAMINOANTHRACINONE AND DERIVATIVES

Diaminoanthraquinone (called Diamino-anthrachinon in Ger), H₂N.C₆H₅(CO)₂C₆H₃,NH₂ or
C₆H₄(CO)₂C₆H₂(NH₂)₂; mw 328.24, 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, H₂N.C₆H₅(NO₂)
(CO)₂C₆H₂(NO₂).NH₂; mw 328.24, N 17.67%.
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
plts (from Nitrobenz), mp above 330°; diffe
sol in all solvs (Ref 2)
4, 5-Dinitro-1, 8-diaminoanthraquinone, yel
cysts, 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
cysts (from NB), mp above 300°(Ref 4)

Other props & methods of prep 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)

Dinitrodinitriminoanthraquinone,
O₂N.HN.C₆H₅(NO₂)(CO)₂C₆H₅(NO₂).NH.NO₂;
mw 418.24, N 20.10%
4,8-Dinitro-1, 5-dinitrodinitriminoanthraquinone,
cyst yel powder, mp explodes ca 134°; sl
sol in concd H₂SO₄ giving a red color; insol
in w; can be prepd by treating 1, 5-
dinitrimino-antbraquinone with HNO₃ at 0°.
Its dibromo & some other salts are also
expl (Ref 1)
X, X-Dinitro-1, 5-dinitraminoanthraquinone, gm-yel powder, mp explodes ca 121°C on rapid heating, dec on prolonged slow heating at 116°C, delg in presence of a little 
H₂SO₄; sol in excess H₂SO₄ giving a green color; insol or diff sol in cold org solvs; obtd with other products by action of HNO₃ on 1, 5-diaminoanthraquinone at -12°C 
(Refs 2 & 3)

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

**Tetranitrodiminoanthraquinone,**

H₂N.C₆H(NO₂)₂(CO)₂C₆H(NO₂)₂NH₂;

mw 418.24, N 20.10%

2, 4, 6, 8-Tetranitro-1, 5-diaminoanthraquinone, dark-brown violet powd, mp not given; sol in pyridine with a crimson-red color; sol in 40% fuming H₂SO₄ with an olive-green col; sol in concd H₂SO₄ with a crimson-red col, becoming violet-red on warming with boric acid; was prep'd by heating 2, 4, 6, 8-tetranitroanthracenylene-1, 5-diurethane with concd H₂SO₄ 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 H₂SO₄ giving a yellow-brown col; in concd H₂SO₄ with a reddish-brown color, becoming claret-red on warming with boric acid; was obtd as above by heating the appropriate urethane deriv with concd H₂SO₄ at 80°C

(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,

O₂N.HN.C₆H(NO₂)₂(CO)₂C₆H(NO₂)₂NH.NO₂;

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 brown color; in alc giving a brown color; sol in Na carbonate giving olive-brown; sol in caustic soda giving brown; sol in H₂SO₄ giving brick-red, becoming reddish-brown on adding boric acid; mod sol in water giving a brown color; was prep'd by treating 1, 5-diamino-anthraquinone with an excess of HNO₃ (d 1.52) and cooling (Refs)

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

**Diaminoazobenzene and Derivatives**

Diaminoazobenzene, Phenylazophenylene or Azodianiline, C₁₂H₁₂N₄, mw 212.25, N 26.40%. Several isomers are known: 2, 2', 3, 3'; and 4, 4'-Diaminoazobenzene (called Diamino-azoazobenzol or Azoanilin in Ger), H₂N.C₆H₄.N:N.C₆H₄.NH₂. See Azodianiline in Vol 1 of this Encycl, p A646-L 2, 4-Diamino-azobenzene, C₆H₅.N:N.C₆H₃(NH₂)₂. Called 2, 4-Diamino-azoazobenzol; 4-Benzolazo-phenylenediamin or Base des Chrysoïdins in Ger. See Chrysoïdine, Vol 3, p C321-R

4-Amino-diacaaminobenzene, C₆H₅.N:NH, C₆H₄.NH₂; bln-yel nds (from dil alc), mp 157°C defigt; sl sol in cold alc; diff sol in eth; insol in petr eth & w; was prep'd by treating 4-acetamidio-diazeobenzene with Na ethylate (Refs 1 & 2)

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

Azido, C₁₂H₁₁N₇, and Diazido, C₁₂H₁₀N₁₀, derivs of Diaminoazobenzene were not found in Beil

Mononitrodiamino-azobenzene, C₁₂H₁₁N₅O₂;

mw 257.25, N 27.23%. Four derivs are found in the literature:

3'-Nitro-2, 4-diamino-azobenzene, O₂N.C₆H₄.N:N.C₆H₃(NH₂)₂; yel crysts (from dil alc), mp 204°C (Ref 1)

4'-Nitro-2, 4-diamino-azobenzene, above formula, red-brown shiny nds (from Nitrobenz or benzene homologs), mp 230°C; 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 Nitro-chrysoïdine, C₆H₅.N:N.C₆H₂(NO₂)₂.NH₂)₂; obtd as HCl salt, bln crysts having a metallic lustre, mp not reported; its aq soln is om 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,
H.C —(NH₂) —C-N: N.C₆H₅
H.C —(NO₂) —C.NH₂ ; no description given, mp 193.5°, was prepd by coupling
PhN₂Cl with 4-nitro-1, 3-phenylenediamine (Ref 5)
Refs: 1) Beil 16, 384  2) Beil 16, (204)
3) Beil 16, (205)  4) A. Korczyński &
S. Piascecki, 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
& CA 53, 4173(1959)

Dinitrodiamino-azo-benzene, C₁₂H₁₀N₆O₄;
mw 302.25, N 27.81%. Two isomers are
found in the literature:
5, 4'-Dinitro-2, 4-diamino-azo-benzene,
O₂N.C₆H₄.N:N.C₆H₅(NO₂)(NH₂)₂; orn-colored
powd, colors accent rayon a golden yel;
was prepd by coupling 4-nitro-1, 3-diamino-
benzene with 4-nitroaniline (Ref 1)
3, 3'-Dinitro-4, 4'-diamino-azo-benzene,
H₂N.(O₂N).C₆H₄.N:N.C₆H₅(NO₂).NH₂; red
plates or om ndls (from phenol + ethanol),
mp 340-41°, was obtd by hydrolysis with
H₂SO₄ of 4, 4'-dicacetamido-3, 3'-dinitro-
azo-benzene, 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 Diamino-
azo-benzene were not found in Beil or in CA
thru 1961

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

DIAMINOBENZENE AND DERIVATIVES
Diaminobenzene, Phenylendiamine, or Amino-
aniline (called Diamino-benzol or Phenyl-
diamin in Ger), H₂N.C₆H₄.NH₂; mw 106.12,
N 26.40%. Three isomers are known:

1, 2-Diaminobenzene, col monoclinic crystals,
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 numerus salts & addn compds.
The Perchlorate salt, C₆H₂N₂+2HClO₄,
crysts, explodes by impact or friction (Ref 2)
1, 4-Diaminobenzene, col to lt purple crystals,
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-diaminobenzens 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, C₆H₇N₃, and Diazido, C₆H₆N₈, 
derivs of Diaminobenzene were not found
in Beil or in CA thru 1961

Monodinitrodiamonobenzene, H₃N.C₆H₃(NO₂).NH₂;
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 159-59°; diffic sol in w
(Ref 1);
4-Nitro-1, 2-diaminobenzene, dk-red ndls,
mp 199-200°(Ref 2);
4-Nitro-1, 3-diaminobenzene, om-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


1, 3-Dinitroaminobenzene [called 1,3 Dinitroaminobenzol or m-Phenylendinitramin (?) in Ger], C₆H₃(NH₂NO₂)₂ or C₆H₄[N::N::O].OH₂ (?); 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-phenylene diamine nitrate with AlCl₃

Ref: Beil 16, 676. No later refs found

1, 4-Dinitrosodihydroxylaminobenzene [Called 1,4-Bis-nitrosohydroxylaminobenzol in Ger] [called N, N -Dihydroxy-N, N'-dinitroso-phenyldiamine and N, N'-p-phenylenebis(N-nitrosohydroxylamine) in CA], ON(HO)N.C₆H₄(NO)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.H₂O.C₆H₄(NO)ONa, brn-yel crysts, deigs on heating on Pt foil (Ref 3). Danzig et al (Ref 4) also prep'd the Na salt & others and found them to be exp in nature. Its Fe salt, dk-red ppt, exists ca 215°, almost insol in pyridine & acet (Ref 3)


Dinitrodiaminobenzene, H₂N.C₆H₄(NO₂)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, om-yel ndls (from alc) or lt yel-brown ndls (from glacial AcOH), mp 260-64; diffic 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 glacial AcOH (Ref 3);

2, 3-Dinitro-1, 4-diaminobenzene, brn-yel ndls (from benz or alc), mp 241-50° (dec); diffic sol in alc, eth, benz or chlff (Ref 4); and

2, 5-Dinitro-1, 4-diaminobenzene, reddish-brown ndls (from alc), mp 295-300° (dec); sol in acet & H₂SO₄; sl sol in chlff, CCl₄ & eth; insol in Benz (Ref 5)

Other props & methods of prepn are given in Refs.


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-phenylenediamin (1,3) in Ger; and 2, 4, 6-Trinitrometaphenylendiamine in Fr; H₂N.C₆H₄(NO₂)₃.NH₂; mw 243.14, N 28.81%; yel crysts (glac AcOH), mp 287-301° (dec); sl sol in glacial AcOH; v sl sol in other solvs; can be prep'd by nitratig m-dichlorobenzene to 2, 4, 6-trinitro-1, 3-dichlorobenzene and aminating in methanol soln; or by the aminating of 2, 4, 6-trinitro-3-aminoanilide (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 phosphorus oxytrichloride & dipyrindimium styrphane 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 NH₃

The expnl 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 proplets (Ref 11)

D 1131

USP 2679538(1954) & CA 49, 4715(1955)

DIAMINOBENZIL AND DERIVATIVES
Diaminobenzil, H₂N.C₆H₅.CO.CO.C₆H₅.NH₂; mw 240.25, N 11.66%. The p₁p₁ -Diamino deriv, yel ndls (from boiling water or alc), 166-69°, was patented by Anderson (Ref 3) by reducing p₁, p₁ -dinitrobenzil using FeSO₄, (NH₄)₂S or Fe(OH)₂ as reducing agents, or using catalytic reducing agents. A similar method of prep the 4,4₁-Diamino deriv had been reported earlier by Kuhn et al (Ref 2). An improved prep of the 4,4₁-Diamino deriv from p-aminobenzaldehyde which, after acetylation, is converted to 4,4₁-diacetamidobenzoin which is oxidized to the benzil deriv and hydrolyzed with HCl is reported by Gee & Harley-Mason(Ref 4)
4,4₁-Diaminobenzil was found by Kuhn et al (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)

Dinitrodiaminobenzil, C₁₄H₁₀N₆O₆, not found in Beil or in CA thru 1961

3,3₁,5,5₁ -Tetranitro-4,4₁ -diaminobenzil
H₂N.C₆H₅(NO₂)₂.CO.CO.C₆H₅(NO₂)₂.NH₂; mw 420.25, N 20.00%; bm-yel crysts (from NB), mp 271°; insol in most org solvs; was prep by heating of 3,3₁, 5,5₁ -tetrinitro-4,4₁ -dimehoxybenzil with aq alc NH₃ in a tube at 100°
Refs: 1) Beil 14, [98] 2) J. van Alphen, Rec 48, 1118(1929) & CA 24, 844(1930)
Hexanitrodiaminobenzil, C₁₄H₅N₈O₁₄, not found in Beil or in CA thru 1961

The Amm salt, called 2,4,6-Trinitro-1 -ammoniumtrinitramino-3-aminobenzene or NH₃+ 2,4,6-Trinitro-3-aminophenyltrinitramine (in CA), H₂N.C₆H₅(NO₂)₃-N(NO₂)₂.NH₄, canary-yel crystals (from boiling alc), mp dec at 180° when heated rapidly and at 300° when heated slowly, explodes when heated in air, was prep by Macciotta & Orani (Ref 3) by dissolving 2,3,4,6(tetranitrophénylnitrïnamine, (O₂N)₄C₆H₅(NO₂)₂H, in w, filtering, and making s/alk with NH₄OH. With excess NH₄OH sym-Trinitrophenylene-diamine (DATNB) is formed
4,6-Diaminobenzimidazole,
\[
\begin{align*}
H_2N.C==CH & \quad C==NH \quad CH \\
| & \quad | & \quad | & \quad \\
HC & \quad C.NH_2 & \quad \cdots & \quad C.NH_2 & \quad \cdots & \quad N
\end{align*}
\]
mw 148.17, N 37.82%. The Dihydrochloride deriv, C_7H_12N_4Cl_2, wh ndls (from HCl), mp 335° dec; was prpbd by addg formic acid to freshlly 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_7H_11N_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 & prtg with water.

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


Diaminobenzofurazan,
\[
\begin{align*}
HC==C.NH_2 & \quad C==N \\
| & \quad | & \quad \\
H.C & \quad C.NH_2 & \quad \cdots & \quad C.NH_2
\end{align*}
\]
I

\[
\begin{align*}
HC==C.NH_2 & \quad C==N \\
| & \quad | & \quad \cdots & \quad | & \quad \cdots & \quad O
\end{align*}
\]
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 benzofurazan in MeOH. The 4,7-Diamino deriv, red crysts (from hot w), mp 193-94°, sol in methanol, acet, chl & 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 prepbd, 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 plts (from dill alc) or yel rods (from benz), mp 132-35°, forms salts & addn compds (Ref 1);
2,3'-Diamino-, golden-yel plts or ndls (from dill alc), mp 80°, sol in alc; less sol in w; forms a hydrochloride salt (Ref 1);
2,4'-Diamino-, straw colored ndls (from w), mp 128-20°, 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 dill 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 dill alc), mp 241-45°, readily sol in alc; mod sol in hot w & is decmpd by boiling w; insol in cold w (Ref 3)

Other props & methods of prep of diamino benzophenones 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_4(NO_2)_2.CO.C_6H_4(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 prepbd 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_4(NO_2)_2.CO; mw 392.24, N 21.43%; lt-yel, woolly crysts (from NB), mp dec 250°,
D 1133

mp 324° by Maquenne block; sol in alc, benz, AcOH & H₂SO₃; was prepd by heating the 4,4¹-dichlorotetranitrobenzophenone deriv with alc NH₃; and by reaction of 4,4¹-dimethylamino-3,5,3¹,5¹-tetranitro-diphenylmethane or benzophenone with chronic 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

4,4¹-Diaminobiphenyl. See Benzidine in Vol 2, p B63-R

**Diaminobiphenyl, Nitrated Derivatives.**

In addn to the nitrated derivs of Benzidine or 4,4¹-Diaminobiphenyl given in Vol 2, p B64 of this Encyclopedia, the following nitrated derivs of Diaminobiphenyl are described in the literature:

**Dinitrodiaminobiphenyl,**

H₂N·C₆H₅(NO₂)₂·C₆H₅(NO₂)·NH₂; mw 274.23, N 20.43%;

5,5¹-Dinitro-2,2¹-diaminobiphenyl; golden-yel prisms having a violet glaze (from phenol & alc), mp becomes bm & sinters at 285° & 303°, sol in hot phenol; diff sol in alc; v sl sol in chf; insol in eth, benz & petr eth; was prepd by nitration of diacetyl-2,2¹-diaminobiphenyl and hydrolysis of the dinitro deriv with HCl & alc (Refs 1 & 2);

4,4¹-Dinitro-2,2¹-diaminobiphenyl, formula as above, orn crystals (from alc), mp 249-50°;

was obtd by heating of disuccinyl-2,2¹-diamino-4,4¹-dinitrobi phenyl with 10% KOH (Ref 2);

4,5¹-Dinitro-2,2¹-diaminobiphenyl formula as above, rhomb crystals + AcOH (from AcOH), mp 180°, on heating in air for several hrs or on washing with alc the crystals 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 crystals 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);

4,3¹-Dinitro-2,4¹-diaminobiphenyl, formula as above; bright-red crystals (from alc), mp 199-200°; sol in concd but not in dil acids;

was formed on saponification of 2,4¹-diacetylamino-4,3¹-dinitrobi phenyl by boiling in a mixt of concd H₂SO₄ & 50% alc for 6 hrs, pouring into w and washing the ppt with aq HCl (Ref 3);

5,3¹-Dinitro-2,4¹-diaminobiphenyl, formula as above, red crystals, mp 242-43°; was obtd on saponification of 2,4¹-di(acetylamino)-3¹,5-dinitrobi phenyl with a mixt of dil H₂SO₄ & alc for six hrs (Ref 4); and

6,6¹-Dinitro-2,2¹-diaminobiphenyl, formula as above, orn blades (from alc), mp 241° & 242-44° (Ref 2); was obtd by boiling 2,6,2¹,6¹-tetranitrobi phenyl 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) Bci 13, 210 2) S. Sako, MemColl-EngKyushuImpUniv 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. Holr & A.N. Hughes, JCS 1960, 3219 & CA 55, 1526(1961)

**4,6,4¹,6¹-tetranitro-3,3¹-diaminobiphenyl,**

H₂N·C₆H₅(NO₂)₂·C₆H₅(NO₂)·NH₂; mw 364.23, N 23.08%; red-bm crystals, mp 297°; was prepd by heating 5,5¹-dichloro-2,4,2¹,4¹-tetranitrobi phenyl with alc NH₃ at 110° (Ref 1 & 2). The expl props of this tetrani tro deriv were not reported

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

**2,4,6,2¹,4¹,6¹-Hexanitro-3,3¹-diaminobiphenyl** or **2,4,6,2¹,4¹,6¹-Hexanitro-3,3¹-biphenylidiamine; also called Dipicramide** (by Dacons et al),

H₂N·C₆H(NO₂)₃·C₆H(NO₂)₃·NH₂;
mw 454.23, N 24.67%; microcryst-yel powd (from acet + alc), mp 299° dec; explosive compd; was prepd by addg portionwise during 1 hr at a temp below 12° 3,3'-dichloro-2,4,6,2',4',6'-hexanitrophenyl to abs éthanol saturated with dry NH₃; anhyd NH₃ slowly bubbled thru the blood-red soln for an addnl 15 mins and the soln slowly heated to reflux; refluxed for 30 mins with loss of NH₃ & alc; cooled to 10° and the ppt washed with 3% aq HCl, alc & eth; the compd was purified by treating a hot aceton soln with charcoal, addg hot alc & concentrating to a small vol (Ref 2)


1(5-Diaminobuiuret)(called Diamino-biuret; Imino-dicarbonsäure-dihydrazid; Iminodialimeisensüre-dihydrazid; or Ammoniakdicarbonsäuredihydrazid in Ger), HN(CO.NH.NH₂)₂; mw 133.12, N 52.61%; col prisms (from w + alc) or ndls (from glc AcOH), mp 200-05° dec; readily sol in w; mod sol in hot glc AcOH; sl sol in alc & eth; was prepd by reaction of N-tricarbonylic ester & 42% hydrazine hydrate (Refs 1 & 2). Its relative basicity was detd by Rochlin et al (Ref 3)


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

DIAMINOBUTANE AND DERIVATIVES
Diaminobutane or Butanediamine, C₄H₁₂N₂; mw 116.17, N 48.23% All possible isomers are known:

1,2-Diaminobutane, CH₃.CH₂.CH(NH₂).CH₂.NH₂ 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, CH₃.CH(NH₂).CH₂.CH₂.NH₂ forms numerous salts, some of which are unstable on heating; and exists as an oil, bp 140.5-141.5° at 738mm Hg (Ref 2);

1,4-Diaminobutane, H₂N.CH₂.CH₂.CH₂.CH₂.NH₂ forms many salts, some of which are unstable on heating; exists as ptls, mp 26-28° (Ref 3); and

2,3-Diaminobutane, CH₃.CH(NH₂).CH(NH₂).CH₃ 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


Azido, C₄H₁₁N₅, and Diazido, C₄H₁₀N₈, derivs of Diaminobutane were not found in Beil or in CA thru 1961

Mononitrodiaminobutane, C₄H₁₁N₃O₂, not found in Beil or in CA thru 1961

Dinitroaminobutane, C₄H₁₀N₄O₄; mw 178.15, N 31.45%. The following derivs are reported in the literature:

1,3-Dinitroaminobutane, CH₃.CH(NH₂).NO₂; CH₂.CH₂.NH.NO₂; col crysrs (from benz), mp 81.6-82.2° was prepd by dissolving 1,3-dinitro-4-methyl-1,3-diazacvclohexanone-2 in 10% aq NaOH and acidifying this soln with concd HCl (Ref 3); 1,4-Dinitroaminobutane, O₂N.NH.CH₂.CH₂.CH₂.CH₂.NH.NO₂; col crysrs (from w), mp 162.2° dec; mod sol in hot w; decompd by boiling with H₂SO₄; was prepd from N,N,N-dinitro-N,N-dicarbomethoxy-tetramethylene & aq NH₃ (Refs 1 & 2); and by hydrolysis of 1,3-dinitro-1,3-diazacycloheptone-2 (Ref 3)

NOTE: Higher nitrated derivs of Diamino-
butane were not found in Beil or in CA thru
1961

**DIAMINODIHYDRO-s-TETRAZINE
AND DERIVATIVES**

**Diaminodihydro-s-tetrazine, C\textsubscript{2}H\textsubscript{6}N\textsubscript{6}; mw
114.12, N 23.65%.** The 3,6-Diamo-1.4-
dihydro-s-tetrazine,

\[
\begin{array}{c}
\text{NH} \quad 1 \\
\text{H}_2\text{N} \quad \text{C} \\
\text{6} \\
\text{3} \quad \text{C} \quad \text{NH}_2 \\
\text{or} \\
\text{NH} \quad 4 \\
\text{H}_2\text{N} \quad \text{C} \\
\text{N} \\
\text{H}_2\text{N} \quad \text{C} \\
\text{NH}_2 \\
\text{N} \quad \text{N}
\end{array}
\]

is known, dk-red amorphous solid, mp 360°, was obtd
when S-methylisothiosemicarbazide hydro-
iodide, H\textsubscript{2}N\text{HNC}(\text{NH})\text{.S.CH}_3\text{.HI}, was con-
densed in a heterocyclic amine, such as
piperidine, pyrrolidine or morpholine (Ref 3).
Its sulfate & picrate were also prep'd.

Walter (Ref 2) patented the use of this
compd, in the manuf of aminoplasts by its
reaction with aldehydes, prep'd by recrystal-
lization from H\textsubscript{2}O of the reaction of
H\textsubscript{2}N\text{COOC}_2\text{H}_5 and an aq soln of 85%
N\textsubscript{2}H\textsubscript{4}.H\textsubscript{2}O.

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-Diamo-3,6-dihydro-s-tetrazine Dini-
trote** (called Nitrato de diaminotetrazène
in Fr).

\[
\begin{array}{c}
\text{NH}_2 \quad \text{H}_2\text{N} \text{HC} \\
\text{6} \\
\text{3} \quad \text{CH} \quad \text{NH}_2 \quad \text{HNO}_3 \\
\text{N} \quad \text{N}
\end{array}
\]

mw 240.14, N 46.67%; defigr weakly in a
flame, explodes at 150° when heated in a
tube; was prep'd 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 compds
(Ref 2)

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

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

**Diaminodimethylamine,**

\[
\begin{array}{c}
\text{H}_2\text{N} \quad \text{C} \\
\text{6} \\
\text{3} \quad \text{C} \quad \text{NH}_2 \\
\text{or} \\
\text{NH} \quad 4 \\
\text{H}_2\text{N} \quad \text{C} \\
\text{N} \\
\text{H}_2\text{N} \quad \text{C} \\
\text{NH}_2 \\
\text{N} \quad \text{N}
\end{array}
\]

Its trinitro deriv, 1,1'-Dinitramino-N-nitro
dimethylamine,

\[
\begin{array}{c}
\text{O}_2\text{N} \quad \text{N} \\
\text{H}_2\text{N} \quad \text{C} \\
\text{6} \\
\text{3} \quad \text{CH}_2\text{NH}_2 \text{NO}_2 \\
\text{or} \\
\text{NH} \quad 4 \\
\text{H}_2\text{N} \quad \text{C} \\
\text{N} \\
\text{H}_2\text{N} \quad \text{C} \\
\text{NH}_2 \\
\text{N} \quad \text{N}
\end{array}
\]

mw 210.12, N 40.00%; was found as a second-
ary 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\textsubscript{16}H\textsubscript{14}N\textsubscript{2}O\textsubscript{2};
mw 266.29, N 10.52%.** Two isomers are
found in the literature:

2,4-Diamo-1,3-dimethylanthraquinone,
C\textsubscript{8}H\textsubscript{4}(CO)\textsubscript{2}C\textsubscript{6}(CH\textsubscript{3})\textsubscript{2}NH\textsubscript{2}2, dk-red crys-
t (from glac AcOH), mp 230° dec; obtd by re-
duction of 2,4-dinitro-1,3-dimethylan-
thaquinone with hot aq Na sulfide soln (Ref 2)

1,8-Diamo-2,7-dimethylanthraquinone,
H\textsubscript{2}N.C\textsubscript{6}H\textsubscript{2}(CH\textsubscript{3})(CO)\textsubscript{2}C\textsubscript{6}H\textsubscript{2}(CH\textsubscript{3})\textsubscript{2}NH\textsubscript{2}2
grn-
red crys, mp 208-09°, obtd by reduction
of 1,8-dinitro-2,7-dimethylanthraquinone
with ZnCl\textsubscript{2} & KOH (Ref 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\textsubscript{16}H\textsubscript{12}N\textsubscript{4}O\textsubscript{6}, and Tetranitro,
C\textsubscript{16}H\textsubscript{10}N\textsubscript{6}O\textsubscript{10}, derivs of Diaminodimethyl-
anthaquinone 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 Diaminoxyylene
(called Diamino-dimethyl-benzol; Diaminooxyli and Dimethyl-phenylenediamin in Ger);
C₆H₄(NH₂)₂(CH₃)₂; mw 136.19, N 20.57%.
All possible isomers are described in Beil:
3,4-Diamino-1,2-dimethylbenzene, ptdts, 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 prisms (from benz), mp 116° (Ref 2);
4,5-Diamino-1,2-dimethylbenzene, ptlts + H₂O, 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, lfrs (from benz + petr eth), mp 103-04° (Ref 4);
4,5-Diamino-1,3-dimethylbenzene, ndls (from petr eth) or lfrs (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 lfrs (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 prep 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,
(CH₃)₂C₆H(NO₂)(NH₂)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; diff sol in w; was prepd by reduct. of 4,6-dinitro-2-amino-m-xylene with Amm hydrosulfide (Ref 1);
2-Nitro-6,8-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 obt 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,
1-red ndls (from benz or dil aq alc), mp 169°; mod sol in eth & gluc AcOH; sl sol in hot w; v sl sol in benz; was obt 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,
CH₃.CH ——— CH ——— CH₂.2HClO₄
|     |     |     |
NH₂ | CH₃ | NH₂

an expl listed as Compd No 212 in ADL.
PureExplComps, Part II, 1947, pp 62, 69, 70 & 149

DIAMINOETHANE
AND DERIVATIVES

1,2-Diaminoethane or Ethylenediamine
(called 1,2-Diamino-äthan or Äthylen-dia-
amin in Ger), H₂N.CH₂.CH₂.NH₂; mw 60.10, N 46.62%; col liq, fr p 10.9°, bp 117.2° at
760mm, d 0.8995 at 20°, pD 1.4565 at 20°,
flash p (COC) 150°F; completely miscible with w; forms azoetropes w, toluene & methyl cellosolve; was prepd for the first time in 1871 by heating ethylene dichloride with alc NH₃ 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 explo.

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. 


N-Nitro-diaminoethane or N-Nitro-ethylendiamine (c called β-Aminoethylinitramine by Hall & Wright), H₂N.CH₂.CH₂NH₂NO₂; mw 105.10, N 39.98%; col crys (from w), mp 240-45° dec; insol in alc; was obtd by treatment of propyl-β-chloroethylaminonitrocarbonate with alc KOH or by soln of β-aminoethylinitramine in aq NaOH, refluxing, neutralizing with concd HCl & evaporating the filtrate to dryness (Ref 2). McKay et al (Ref 3) prep'd the compd by aq hydrolysis of 1-nitro-2-nitriminoimidazolidine


N₃N⁻¹-Dinitro-diaminoethane; N₃N⁻¹-Dinitro-ethylenediamine or Ethylenedinitramine (abbrd EDNA) and HALEITE (called 1,2-Dinitramino-áthan; N₃N⁻¹-Dinitro-ethylenediamine; and áthylendinitramin in Ger), O₂N₂.H₂O₂Cl₂.H₂O₂.NH₂NO₂; mw 150.10, N 37.33%; col ndls, mp 174-79° dec; d 1.75 at 20°Qcomb, 2506 cal/g at const-pres; readily sol in alc & acet; mod sol in w; sl sol in eth, chlf & benz; was prep'd by aq hydrolysis of N₃N⁻¹-dinitroethylene urea (Refs 1 & 3). Hall & Wright (Ref 4) converted β-aminoethylinitramine to N₃N⁻¹-Dinitrodiminoethane via the dichloramine deriv

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

Booster sensitivity, 2.09 inches wax for 50% detonations (TNT value 1.68 inches)

Brisance by sand test, 5.23 g 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, 7570 m/sec at d 1.49 g/cc

Explosion temperature, 216° in 1 sec, dec 189° in 5 sec, and 178° in 10 sec

Flammability index, 138 (TNT = 100)

Friction pendulum, unaffected by either fiber or steel shoe

Gas evolved on explosion, 908 cc/g

Heat of combustion, 2477 cal/g at const vol

Heat of explosion, 1276 cal/g

Heat of formation, 134 cal/g

Heat test at 100°C, 0.2% loss 1st 48 hrs, 0.3% loss 2nd 48 hrs, no expn in 100 hrs

Hygroscopicity, 0.1%

Impact sensitivity, with 2 kg wt, 14 inches for 17 mg sample by PA App; 48 cm for 20 mg sample by BM App

International heat test at 75°C, 0.01% loss in 48 hrs

Power by ballistic mortar test, 139% of TNT; and by Trauzl test 122% TNT

Rifle bullet impact test, 60% partials, 20% burned and 20% unaffected in 5 trials

Sensitivity to initiation, 0.21 g MF or 0.13 g LA req'd for 0.4 g sample

Stability in storage, withstands storage at 50°C

Vacuum stability test, 0.5 cc gas evolved in 40 hrs at 100°C, 1.5 cc at 120°C and 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 KClO₃ & NH₄ClO₄

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)
\( \text{\textbf{D 1138}} \)


\( \text{\textbf{Diaminoethane Dichlorate,}} \)
\( (H_2N.CH_2.CH_2.NH_2).2HCIO_3, \) an expl listed as Compd No. 16 in ADL PureExp1Comps, Part 1(1947), p 72. No props of this compd are reported

\( \text{\textbf{Di(aminooethyl)-amine or Diethylentetramine.}} \)
See Bis(aminooethyl)-amine and Derivatives in this Encycl, Vol 2, p B128-L

\( \text{\textbf{N,N'-Di(\beta-aminooethylamino)-ethane or}} \)
\( \text{\textbf{N,N'-Di(\beta-aminooethyl)-ethylenediamine.}} \)
See 1,2-Bis(\( 2^-\text{ami} \)noethylamino)-ethane, Vol 2, p B129-L

\( \text{\textbf{Diaminoethylene Dichlorate or Ethylenediaminedichlorate,}} \)
\( O_3CH.H_2N.CH_2.CH_2.NH_2.HCIO_3, \) an expl listed as Compd No. 16 in ADL PureExp1Comps, Part 1(1947), p 72. No props of this compd are reported

\( \text{\textbf{N,N'-Di(\beta-aminooethyl)-ethylenediamine;}} \)
\( \text{\textbf{1,4,7,10-Tetrazadecane or Triethylentetramine abbr as TETA.}} \)
See Bis(aminooethyl-amino)-ethane in Vol 2, p B129-L

\( \text{\textbf{Sym-Di(aminooethyl)-urea.}} \)
See 1,3-Bis(aminooethyl)-urea in Vol 2, p B130-L

\( \text{\textbf{N,N'-Di(\beta-aminooethyl)-propanediamine.}} \)
See Bis(aminooethylamino)-propane in Vol 2, p B130-L

\( \text{\textbf{1,3-Diaminoguanidine} (called N.N'-Diamino-}
\text{guanidin in Ger), H}_2N.NH.C(NH).NH.NH.H_2; \)
mw 89.11, N 78.60%; known in the form of its salts, ie Hydrobromide, CH_2N_5 + HBr, ppltts (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-

\( \text{\textbf{Diaminoguanidine Azide, N}_3.C(NH).NH.NH.H}_2; \)
mw 100.09, N 83.97%; compd predicted by reduction of azidonitroaminoguanidine, N_3C(NH).NH.NO_2, using Na hydrosulphite, however, nitroguanidine, H_2N.C(NH).NH.NO_2, always resulted (Ref 2)

\( \text{\textit{Re)s:} 1) \) Beil-not found 2) F.L. Scott et al, JAppiChem(London) 2, 368(1952)

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

\( \text{\textbf{Di(aminoguanidinium)-1,6-dinitrobiguanidine.}} \)
See Bis(aminoguanidinium)-1,6-dinitroguanidine in Vol 1, p A214-R

\( \text{\textbf{Diaminohydroxypropane.}} \)
See Diaminopropanol

\( \text{\textbf{Diaminohydroxytriazine Picrate.}} \)
See Ameline Picrate in Vol 1, p A274-R

\( \text{\textbf{Diaminoimidazolidine, C}_3H_9N_4, \text{ may be}} \)
\text{considered as a parent compd of the following}
\text{substance:}
\( (2^-\text{Amino-2-nitramino-1-nitro}-\text{imidazolidine,}} \)
\( H_2C—N(NO_2)\text{—C(NH}_2)(NH.NO_2) \)
\( H_2C—\text{———NH; mw 192.15} \)
N 43.68%. Cryst, mp 184.8-185.3°, with dec-
\text{compn. Was prep’d from 1-nitro-2-nitriminoimi-

dazolididine-2 (Ref 3) by the method de-
\text{scribed in Ref 2} \)
D 1139

Refs: 1) Beilnot 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, 1986(1952)

DIAMINOMETHANE
AND DERIVATIVES

Diaminomethane or Methylene diamine (called Diaminomethan or Methylendiamin in Ger), H₂N.CH₂.NH₂; mw 58.08, N 48.23%; exists in the form of salts, ie Dihydrochloride, CH₃N₂ + 2HCl , prisms, dec on soln in w; Dinitratae, CH₃N₂ + 2HNO₃, prisms expl Mildly on heating w/o melting; Sulfamic Chloride, CH₃N₂ + 2HCl + NaCl, prisms, dec above 300° w/o melting; and Sulfate, CH₃N₂ + H₂SO₄, crys, dec 183-93°
Ref: Beil 1, (305), [648] & [1596]

Mononitrodiaminomethane, CH₃N₃O₂, was not found in Beil or in CA thru 1961

N,N¹-Dinitro-diaminomethane; N,N¹-Dinitro-
methanediamine; Dinitromethanomethane or N,N¹-
Dinitromethylene diamine (called Dinitromethan-
omethan or Methylendinitramin in Ger),
O₂N.HN.CH₂.NH.NO₂; mw 104.07, N 53.84%;
crys (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 decomp in aq soln 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 & Thom (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, recryst or evap 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 stabili-

lity. Other examples using 9/1 ethylene chloride/isopropanol mix 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 & Urbanski (Ref 14) as follows:


Explosion by 2kg falling wt, Height, cm, for
Salt 50% explns
Pb(II) 12
Hg 10
Ag 10
Ba insensitive
Na insensitive

Ignition point 5°C
Pb(II) 238°
Hg 195 deflgr
Ag 195 explodes
Ba 162 slow decomps
Na 198 dec

Data similar to the above were obtd and compared to the props of the salts of methylene-
diisonitrile, HON:N(O)CH₂NO₂:CH₂N(O)N.OH
(UV absorption spectra) 6) J. Barrott et al., JCS 1953, 1998 & CA 48, 10541(1954)
12) R. Reed, Jr., JOC 23, 496(1958) & CA 53, 816(1959)(Reactions with dianinoalkanes)

NOTE: Higher nitrated derivs of Diamino-
methane were not found in Beil or in CA thru
1961

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

Its nitrated derivative:
2,2-(Nitraminomethyl)-1,3-propanedinitro-
methyl or Pentaoxytetranitramine,
C(CH₂N₂N₂O₂)₄; mw 312.21, N 42.29%, Ob to
CO₂ -41%. This compd is listed by ADL
(Ref 2) as compd No 232 and is called
Tetrad nitraminomethyl)methane. The follow-
ing props are reported by ADL, now declass-
IFIED (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 187% 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, Pure Expl-
Comps, Part 1(1947), pp 35, 148, 178 and
Part 4(1952), p 586

2,4-Diamo-no-6-methyl-s-triazine (called 4,6-
Diamo-no-2-methyl-1,3,5-triazin; 4,6-Diamo-
2-methyl-tetrahydro-1,3,5-triazin or Aceto-
guanamin in Ger),

HN=C
| N= C(CH₃)
| N= C(CH₃)
NH or H₂N.C

mw 125.14, N 55.97%; shiny wh plts or ndds
(from w), mp 265-74°C; readily sol in hot w or
alc; less sol in cold w; can be prepd by heating
cyanoguanidin & acetamidine hydrochlori-
de or acetonitrile in a tube at 225-30°C or by
heating biguanide with acetonitrile and by
other methods given in the Refs

Many addn compds & salts have been
prep'd, some of which are unstable on heating.
For example, the Nitrate, C₄H₇N₅ + HNO₃
prisms, explodes on heating. Many other re-
actions & props of the Diamo-no-methyl-s-
triazines 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, 1016(1943)(Prepn & also
of other guanamine derive) 4) J.K. Simmons & W.I. Weaver, USP 2408694 (1946) & CA 41, 1240(1947)(Prepn)
8) Amer Cyanamid, BritP 642409(1950) & CA 45, 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)
(1953)(UV absorption spectra) 12) K. Rohne, Monatsh 86, 65(1955)(Prepn & paper-
(Improved prepn)

Diamino-oxalic Acid Dihydrazone; Oxalhydro-
zone Oxamide (called Oxalidimidsäure-
dihydrazid; "carbodihyrazimin" or "cyanh-
dyrazin" in Ger),
(H₂N₃N):(H₂N)(C.NH₂)(HN.NH₂) or
(H₂N)(H)(H):C.(NH)(NH.NH₂) mw 116.13,
N 72.37%; col ndls (from dil alc), mp dec &
becomes orn-yel at 160°C, and does not melt to
250°C; readily sol in w; sl sol in alc; insol in
eth; obd by combining cyanogen with hydrazone hydrate (Refs 1, 2 & 3).
This compd is also obd from dihydroxamide
in 20% alc & N₂H₄.H₂O (Ref 4)

Salts of the compd were prepd by De-
dichen (Ref), some of which are expl:
Hydrochloride, C₂H₂N₆.2HCl, yel ndls, ex-
plodes by impact but not by heating
Sulfate, C₂H₂N₆.H₂SO₄, wh powder
Nitrate, C₂H₂N₆.2HNO₃, mother-of-pearl
crstals. Many other salts, similar to those
of NH₃, having characteristic colors are also
formed
Refs: 1) Beil 2, 560 2) A. Angeli, Gaz 23 11, 103(1893) & JCS 66 1, 150(1894)
3) T. Curtius & G.M. Dedichen, JPraktChem 50, [2], 254(1894) & JCS 68 1, 93(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 Diaminoxybenzene**
(called Diamino-oxy-benzol or Diamino-phen-
ol in Ger), (H₂N)₂C₆H₄.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
C₆H₅N₂O₂ + 2HCl, ndls, (Ref 3)
2,6-Diaminophenol, as Hydrochloride,
C₆H₅N₂O₂ + 2HCl, ndls, or Sulfate, C₆H₅N₂O₂ +
H₂SO₄, lft-yel ndls (Ref 4)
5,4-Diaminophenol, mp 167-68°, dec under
rapid heating (Ref 5)
3,5-Diaminophenol, prisms, mp 168-70°
(Ref 6)

Other props & methods of prep of Di-
aminophenols are given the Refs
Refts: 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** (H₂N)₂C₆H₅(NO₂)OH
mw 169.14, N 24.85%. Two isomers are known:
4-Nitro-2,6-diaminophenol, dk-yel ndls or thin
lfts + H₂O (from water), mp 169° dec (Ref 3)
260°(Ref 1); sol in acet, ethyl acetate, benz,
alc, water, chlf, dil acids & dl alk; was
prepd by passing H₂S into Picric Acid in hot
dil NH₄OH until no ppt was given by addo
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 Refts
Refts: 1) Beil 13, 563, (209) & [316]
2) A. Koczyuski & S. Plasecki, AnzAkadWiss-
Kraakau 1917, 176 & CA 16, 2124(1922)
3) J. English, Jr et al, JACS 62, 354(1940)
& CA 34, 2348(1940) 4) Societe Monsavon-
1'Oréal, FrP 1137922(1957) & CA 53,
18405(1959)
Dinitrodiaminophenol, $C_6H_8N_4O_5$, 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 crys (from glc AcOe), mp 270$^\circ$; was obt by Blanksma (Refs 1 & 2) by treating pentanitrophenol, tetrinitroresorcin-monomethylether, or 5-chloro-2,3,4,6-tetranitrophenol (Ref 3) with alcoholic ammonia.

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

Refs:  1) Beil 13, 168  2) J. J. Blanksma, Rec 21, 263(1902)  3) J. J. Blanksma, Rec 27, 36(1908)

**DIAMINOPROPANE AND DERIVATIVES**

Diaminopropane; Propandiamine; N-Methyl-ethenediamine and Trimethylenediamine; $C_3H_8N_2$, mw 74.13, N 37.80%. Two isomers are known:

1,2-Diaminopropane (called 1,2-Diaminopropan or Propyldiamine in Ger), CH$_3$.CH(NH$_2$).CH$_2$.NH$_2$; exis as d-form, l-form & dl-form all of which form numerous crys salts, some of which are unstable on heating (Ref 1)

1,3-Diaminopropane (called 1,3-Diaminopropan or Trimethylenediamine in Ger), H$_2$N.CH$_2$.CH$_2$.NH$_2$; liq, fp 23.5$^\circ$ bp 131$^\circ$, forms numerous salts, the perchlorate, $C_3H_8N_2 + 2HClO_4$, explodes when heated to 310$^\circ$ (Ref 2)

N-Methyl-ethylenediamine [called N-Methyl-ethylenediamine or Methyl-(2-amino-ethyl)-amin in Ger] CH$_3$.NH.CH$_2$.CH$_2$.NH$_2$; liq, bp 115-117$^\circ$, forms numerous salts, such as the Cu perchlorate, [Cu(C$_3$H$_7$N$_2$)$_2$](ClO$_4$)$_2$, blue-violet crys, defgl on heating (Ref 3)

Other props & methods of prepn are given in theRefs


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-nitriminopropane by McKay & Viron (Ref 2)

CH$_3$.CH(NH$_2$).CH$_2$.NH.NO$_2$, crys(from w + alc), mp 239.5-40.9$^\circ$ 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-nitraminoethy)3-nitro-urea with water until gassing ceases (Ref 2)

N-Nitro-N-methyl-ethylenediamine Salts [called 3-Nitrazaburylammonium Nitrate or Chloride by Frankel & Klager (Ref 3)], CH$_3$.N(NO$_2$).CH$_2$.CH$_2$.NH$_2$.HX, where X = NO$_2$ or ClI, 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-Dinitrodiaminopropane or Propyldinitramine, CH$_3$.CH(NH.NO$_2$).CH$_2$.NH.NO$_2$; mw 164.13, N 34.14%; crys from w, mp 109.8-12.0$^\circ$, 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$^\circ$*  
*Hygroscopicity: 0.7% at 30$^\circ$C & 90%RH; 2.42% at 30$^\circ$C & 100%RH*  
*Impact sensitivity: 63 cm vs 48-50 for RDX*  
*Power: 116% TNT*  
*Stability tests: International at 75$^\circ$ & 2.0% loss in 24 hrs*

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

1,3-Dinitroaminopropene; N,N'-Dinitrottrimethyl-ndimine; or N,N'-Dinitro-1,3-propanedimine, O_2N.H.N.CH_2.CH_2.CH_2.NH.NO_2, prisms (from w), mp 76.2-68.5\(^\circ\)C readily sol in w & alc; less sol in eth & chlft; obtb by hydrolyzing 1,3-dinitro-1,3-diazacyclohexa-
none-2 (Ref 2). Its decomp 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, 1411(1950) 5) C. Lindley &
J.C. Speakman, JACS 149, 1657 & CA 44,
1411(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)_2.CH_2.CH_2.NH.NO_2; cot
crysts (from w), mp 121-22\(^\circ\)C readily sol in w;
less sol in eth & chlft; was first obtb by
Francihmont & Klobbie (Refs 1 & 2), in
addn to dimethyl-ethylenedinitramine, by
action of methyl iodide on ethylenedinitra-
ine. Some other methods of prepn & props
are given in the Refs. The expl props were
not reported

Ref's: 1) Beil 4, 573 2) A.P.N. Franchi-
mont & E.A. Klobbie, Rec 7, 347(1888) &
JCS 56, 492(1889) 3) R.N. Jones &
G.D. Thom, 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, 5968(1954)(Prepn)
6) L.W. Kissinger & M. Schwartz, JOC 23,
1344(1958) & CA 53, 15968(1959)(Prepn)

NOTE: Higher nitrated derivs of Diamino-
propene were not found in Beil or in CA thru 1961

DIAMINOPROPANOL
AND DERIVATIVES

Diaminopropanol or Diaminohydroxypropane,
C_3.H_10.N_2.O_1; mw 90.13, N 31.08%. Three
isomers are listed in the literature:
1,3-Diamino-1-propanol [caled 1,3-Diamino-
propanol-(1) or α-Hydroxy-trimethylenediamin
in Ger], H_2.N.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 [caled 1,3-Diamino-
propanol-(2); 1,3-Diamino-2-hydroxy-propan;
or β-Hydroxy-trimethylenediamin in Ger],
H_2.N.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 [caled 2,3-Diamino-
propanol-(1); 2,3-Diamino-1-oxo-propan;
β,γ-Diamino-propyl-alcohol; or γ-Oxypoly-
propylenediamin in Ger],
H_2.N.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
Ref's: 1) Beil 4, 200, [739] & [766]
2) Beil 4, (436) & [736] 3) Beil 4, [875]
4) A.Y. Mattlau, USP 2287118(1942) & CA
37, 254(1943)

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 [caled 1,5-
Dinitro-3-nitroxy-1,5-diazapentane by Jones
& Thorn (Ref 4)],
O_2N.NH.CH_2.CH(O.NO_2), CH_2.NH.NO_2; mw
225.49, N 31.22%, OB to CO_2.-17.8%; crysrs
(from Nitromethane), mp 160-5\(^\circ\)C (Ref 3) &
164-65\(^\circ\) dec (Ref 5), degr at 230\(^\circ\); can be
prepd by reaction of 1,3-diamino-2-propanol
with CICO_2.C_2.H_5 followed by nitrin &
ammonolysis (Ref 5)

This compd is a very powerful explosive
(142% of TNT by Ballistic Mortar Test); Impact
Sensitivity, using Bruceton No 3
Mononitrodiamino-tetramethylbenzene,
\( \text{O}_2\text{N-C}_6\text{H}_4[N(\text{CH}_3)]_2 \); mp 209.24, N 20.08%.
Two isomers are reported in the literature:

4-Nitro-1,2-(diamino-N,N,N',N'-tetramethylbenzene, crysTs (from alc), mp 62-63°, was
prepd from 2,5-(COONH)(C_6H_4N(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'-tetramethylbenzene, om-red plts, mp 79-81°, was
prepd from 3,4-dinitrodimethylaniline & dimethyl-
amine 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. Thittle,
USP 2652331(1953) & CA 48, 12595(1954)
(Use as a developer in color photography)
4) I.I. Levkov et al, ZhObshchKim 24,
280(1934) & CA 49, 4628(1955); JGenChem
(Russia) 24, 283(1934) & CA 49, 8248(1955)
(English translation)

Dinitrodiamino-tetramethylbenzene,
\( (\text{O}_2\text{N})_2\text{C}_6\text{H}_4[N(\text{CH}_3)_2]_2 \); mp 254-24, N 22.04%.
Three isomers have been reported:

X,X-Dinitro-1,3-(diamino-N,N,N',N'-tetramethylbenzene, crysTs (from dil alc or
Glac AcOH); was obt'd by nitration of N,N,N',N'-tetramethyl-pheniene-1,3-diamo and
using dil
\( \text{H}_2\text{SO}_4 \) & \( \text{HNO}_3 \) (Ref 1)

4,6-Dinitro-1,3-(diamino-N,N,N',N'-tetra-
 methylbenzene, yel nds (from chlf + alc),
mp 191°, sol in chlf; sl sol in hot alc;
was prepd by heating 1,3-dinitro-4,6-dichloro-
benzene with dimethylanidine in alc; or
from 2,4-dinitro-5-chloro-N,N-imethylan-
iline & NH(CH_3)_2 (Ref 2)

2,6-Dinitro-1,4-(diamino-N,N,N',N'-tetra-
methylbenzene, long, slender deep purple
ndls (from alc), mp 176°; was formed when
2,6-dinitrodimethyl-p-anisidine was heated
in a sealed tube with excess dimethylanine
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)

DIAMINOTETRAMETHYLBENZENE
AND DERIVATIVES

Diaminotetramethylbenzene, Tetramethyl-
diaminobenzene or Tetramethylphenyl-
diamine (called Tetramethyl-phenylenediamine
in Fr and Tetramethyl-phenylenediam in Ger),
\( \text{C}_6\text{H}_4[N(\text{CH}_3)]_2 \); mp 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 crys
salts (Ref 1)

1,3-(Diamino-N,N,N',N'-tetramethyl)-benzene,
liq, fr p -2°, bp 256.7° at 761mm press, d
0.992 at 15°, forms crys salts (Ref 2)

1,4-(Diamino-N,N,N',N'-tetramethyl)-benzene,
liq (from dil alc or pete eth), mp 52°, bp 260°,
d 0.8795 at 99.3°, forms crys 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, \( \text{C}_6\text{H}_3\text{N}_3 \), and Diazido, \( \text{C}_6\text{H}_4\text{N}_8 \),
derivs of Diaminotetramethylbenzene were
not found in Beil or in CA thru 1961
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-Dimino-1,2,3,6-tetrahydro-1,2,4,5-tetrazin in Ger),

H₂N.C₆ 3, C.NH₂ or

HN:C

NH—NH

C:NH

HN—C—S—C:NH

H₂N.C—S—C.NH₂

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 coned mineral acid from which it may be repped unchanged on dilution with water; was prepd by an unequivocal synthesis in which 1,2,4,5-tetrazine-3,6-dicarboxyl azide was degraded thru Curtius rearrangement; also obtd by action of HNO₂ on dianimoguanidine 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 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, C₄H₄N₆.H₂CO₃, orn-yel prlfts, mp dec at 100° into CO₂ & water
Hydrobromide, C₂H₄N₆.HBr, yel lifts, mp explodes ca 120°
Hydrochloride, C₂H₄N₆.HCl, orn-yel lifts (from acq acet), mp dec ca 200°
Nitrate, C₂H₄N₆.HNO₃ + ½H₂O, orn-yel lifts (from w); anhyd sak, yel-bm crysts dec 180-80°
Oxalate, 2C₂H₄N₆.C₂H₂O₄, orn-red prisms, mp dec ca 205°

Picate, C₂H₄N₆.C₆H₃N₃O₇, yel amorph powd, mp expl
4) L. Paoloni, Gazz 87, 313-28(1957) & CA 51, 14407(1957)

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

DIAIMINO THIODIAZOLE
AND DERIVATIVES

2,5-Diamino-1,3,4-thiodiazole (called 2,5-Diamino-1,3,4-thiodiazol or 2,5-Dimino-1,3,4-thiodiazolidin in Ger)
HN:C—S —C:NH

H₂N.C—S—C.NH₂

mw 116.08, N 48.27%; almost col prisms (from w), mp 210-12°; readily sol in w; was obtd by treating hydrazine-N₄N₁-bis-thiocarboxic acid amide with H₂O₂ in hotaq soln or with hydroxylamine. Other methods of preps & props are given in Beil
Ref: Beil 27, 667, (598) & [752]

5-Nitrosoamino-2-amino-1,3,4-thiodiazole
ON.N:C —S —C:NH

ON.HN.C—S—C.NH₂

mw 177.21, N 39.52%; bm crysts; mp explodes at 160-90°; diff sol in alc; insol in w & eth; was prepd by treating the parent compd with isoamylnitrile in alc, or with NaNO₂ in acetic acid soln in the cold

NOTE: Mononitro, C₂H₃N₅O₂S, and Dinitro, C₂H₂N₄O₄S, derivs of Diamoathiodiazole were not found in Beil or in CA thru 1961

DIAIMINOTOLUENE
AND DERIVATIVES

Diaminotoluene or Toluenediamine (called Diamino-толуol; Methyl-phenylendiamin; or
Toluylendiamin in Ger), (H₂N)₂C₆H₃.CH₃; mw 122.17, N 22.93%. All possible isomers are known:

2,3-Diaminotoluene, crsysts, mp 61-64⁰, bp 255⁰ (Ref 1)

2,4-Diaminotoluene, ndls (from w) or rhomb prisms (from alc), mp 99⁰, bp 280-85⁰ (Ref 2)

2,5-Diaminotoluene, plts (from benz), mp 64⁰, bp 273-74⁰ (Ref 3)

2,6-Diaminotoluene, prisms (from w), mp 105⁰; prisms (from benz), mp 106⁰ (Ref 4)

3,4-Diaminotoluene, lfts (from petr eth), mp 88.5-90⁰, bp 265⁰ (Ref 5)

3,5-Diaminotoluene, liq, bp 283-85⁰ (Ref 6)

2,1-Diaminotoluene, H₂N.C₆H₄.CH₂.NH₂, crsysts, mp dec under distillation, given off NH₃ (Ref 7)

3,1-Diaminotoluene, liq, forms a crystal picrate (Ref 8)

4,1-Diaminotoluene, liq, bp 268-70⁰, d 1.08 at 20⁰ (Ref 9)

All of the Diaminotoluenes form crystal salts and addn comds. 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, (H₂N)₂C₆H₃(NO₂).CH₃; mw 167.17, N 25.14%. The following isomers are known:

5-Nitro-2,3-diaminotoluene, om-red ndls (from dil alc), mp 185⁰ & red ndls (from w), mp 175⁰; 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 154⁰; sl sol in hot w; sol in hot alc (Ref 2)

6-Nitro-2,4-diaminotoluene, red prisms (from w), mp 132⁰ & om-red crysts (from benz or petr eth), mp 130-31⁰; sol in dil HCl (Ref 3)

6-Nitro-2,5-diaminotoluene, red to dk-brn bronze luster ndls (from w), mp 173⁰ (Ref 4)

5-Nitro-3,4-diaminotoluene, dk-red ndls (from w), mp 158⁰; 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, [56]

Dinitro-diaminotoluene, (H₂N)₂C₆H₂(NO₂)₂.CH₃; mw 212.17, N 26.41%. The following isomers are reported in the literature:

3,5-Dinitro-2,4-diaminotoluene, golden-yel ndls, mp 240⁰; diff sol in common org solvs; sol in NaOH; can be prepd by reaction of NH₃ on 2,4-dichloro-3,5-dinitrotoluene at 160⁰ or on 2,4-dibromo-3,5-dinitrotoluene at 150⁰, and by heating on a steam bath alc NH₃ & 3,4,5-trinitro-2-methoxytoluene (Ref 1)

3,5-Dinitro-2,6-diaminotoluene, lt-bn crstys, mp 298⁰; v sl sol in alc; was prepd by heating 2,6-dibromo-3,5-dinitrotoluene with alc NH₃ at 150⁰ (Ref 2)

2,4-Dinitro-3,5-diaminotoluene, yel crstys, mp 199⁰ & om-yel ndls (from alc), mp 210⁰; v sl sol in alc; was prepd by reaction of alc NH₃ on 3,5-dichloro-2,4-dinitrotoluene or 3,5-dibromo-2,4-dinitrotoluene at 125⁰, and on 3,5-dichloro-2,4-dinitrophenylacetic acid at 145⁰ (Refs 3 & 5)

2,6-Dinitro-3,3-diaminotoluene, crstys, mp 196⁰ (Ref 6); prepd by heating 6 ps of 3,5-dibromo-2,6-dinitrotoluene with alc NH₃ at 160⁰ (Refs 4 & 5)

The expl props of the dinitro derivs were not detd


Trinitro-diaminotoluene, (H₂N)₂C₆(NO₂)₃.CH₃; mw 257.17, N 27.24%. Only one isomer is known:

2,4,6-Trinitro-3,5-diaminotoluene, yel prisms (from alc), mp 222⁰ & 225⁰ (Ref 3); was prepd by heating 8 ps of 3,5-dibromo-2,4,6-trinitrotoluene with alc NH₃ under pressure on a steam bath (Refs 1 & 2). No expl props of this comd were detd


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

\[
\begin{align*}
\text{NH} & \quad \text{C} \quad (\text{NH})_3 \\
\text{HN} & \quad \text{C} \\
\text{H}_2 & \quad \text{N} \\
\text{N} & \quad \text{C} \quad (\text{NH}_2)
\end{align*}
\]

or

\[
\begin{align*}
\text{NH} & \quad \text{C} \quad (\text{NH})_3 \\
\text{HN} & \quad \text{C} \\
\text{H}_2 & \quad \text{N} \\
\text{N} & \quad \text{C} \quad (\text{NH}_2)
\end{align*}
\]

; mw 111.11, N 63.04%; ndls (from w), mp 329°, mp 318° (Ref 8); sol in 20% hot w, dil HCl or warm dil H₂SO₄; sol in alc; was prepd by heating guanidine formate to 200°; or by treating biguanide hydrochloride with abs alc KOH & chlor under cooling; as well as by heating anhyd biguanide sulfate with Na formate 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).

Formoguanamin 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.


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

\[
\begin{align*}
\text{N} & \quad \text{C} \quad (\text{NH}_2) \\
\text{H} & \quad \text{N} \\
\text{N} & \quad \text{C} \quad (\text{NH}_2)
\end{align*}
\]

; mw 151.14, N 64.88%; col ndls, mp infusible; was prepd by addg NaN(CN)₂ & H₂N.C.(NH)NH.CN to KOH in methyl cellosolve, heating at 130° for 2hrs, stirring, addg w, filterng, 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 flame-proofing agents.


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-carbonitrile.

(4,6-Diamino-s-triazine-2-yl)-guanidine; or Guanylelamine.
on a w bath (Refs). Some derivs of Diamino-
triazole are expl
Refs: 1) Beil 26, 193 & (57) 2) G. Pellizzari,
Gazz 24(1), 491(1894) & JCS 66(1),
518(1894) 3) K.A. Hofmann & O. Ehrhart,
Ber 45, 2733(1912) 4) R. Stolle & K. Krauch,
JPraktChem 88, 310-11(1913) 5) R. Stolle
& W. Dietrich, JPraktChem 139, 193-210
(1934) & CA 28, 2714(1934)
3,4-Diamo-4H-1,2,4-triazole or 3,4-Diamo-
y-s-triazole,
\[
\text{H}_2\text{NN}—\text{N} \text{NH} \quad \text{or ONN}—\text{N} \quad \text{NH}
\]

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,
965-68 (English) & CA 54, 8286(1960)

DIAMINOTRIAZOLE
AND DERIVATIVES
3,5-Diamo-a-s-triazole, Aminoaminotriazole
or Guanazole (called 3,5-Diamo-1,2,4-
triazolidin; Urazol-dimid; 3,5-Diamo-1,2,4-
triazol; or Guanazol in Ger),
\[
\begin{array}{c}
\text{H}_2\text{N.C—NH—N} \\
\text{N—C.NH}_2
\end{array}
\]

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 & hydrazinohy-
drochloride; also prepd in good yield by heating
a mixt of dicyandiamide & hydrazinehydrate

\[
\text{H}_2\text{NN}—\text{C.NH}_2
\]

on a w bath (Refs). Some derivs of Diamino-
triazole are expl
Refs: 1) Beil 26, 193 & (57) 2) G. Pellizzari,
Gazz 24(1), 491(1894) & JCS 66(1),
518(1894) 3) K.A. Hofmann & O. Ehrhart,
Ber 45, 2733(1912) 4) R. Stolle & K. Krauch,
JPraktChem 88, 310-11(1913) 5) R. Stolle
& W. Dietrich, JPraktChem 139, 193-210
(1934) & CA 28, 2714(1934)
3,4-Diamo-4H-1,2,4-triazole or 3,4-Diamo-
y-s-triazole,
\[
\text{H}_2\text{NN}—\text{C.NH}_2 \quad \text{large col plates (from}
\]

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,
965-68 (English) & CA 54, 8286(1960)

DIAMINOTRIAZOLE
AND DERIVATIVES
3,5-Diamo-a-s-triazole, Aminoaminotriazole
or Guanazole (called 3,5-Diamo-1,2,4-
triazolidin; Urazol-dimid; 3,5-Diamo-1,2,4-
triazol; or Guanazol in Ger),
\[
\begin{array}{c}
\text{H}_2\text{N.C—NH—N} \\
\text{N—C.NH}_2
\end{array}
\]

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 & hydrazinohy-
drochloride; also prepd in good yield by heating
a mixt of dicyandiamide & hydrazinehydrate

\[
\text{H}_2\text{NN}—\text{C.NH}_2
\]

on a w bath (Refs). Some derivs of Diamino-
triazole are expl
Refs: 1) Beil 26, 193 & (57) 2) G. Pellizzari,
Gazz 24(1), 491(1894) & JCS 66(1),
518(1894) 3) K.A. Hofmann & O. Ehrhart,
Ber 45, 2733(1912) 4) R. Stolle & K. Krauch,
JPraktChem 88, 310-11(1913) 5) R. Stolle
& W. Dietrich, JPraktChem 139, 193-210
(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],

\[
\begin{align*}
\text{ON.HN.C} & \equiv \text{N} - \text{N} \\
\text{HN} & \equiv \text{C.NH.NO} \\
4 & \quad 3
\end{align*}
\]

mw 157.10, N 62.42%; orm-red amor solid, mp dec 187\(^\circ\)C, was prep'd from guanazole in 7N aq-HCl & NH\(_4\)NO\(_2\).

Reduction of the dinitro compd with SnCl\(_2\) & concd HCl gives 3-Amino-5-hydra-

zino-1,2,4-triazole di-HCl, mp 217\(^\circ\)C (dec), which in an ice-cold aq soln & NaNO\(_2\) gives 5-Azido-3-nitrosamino-1,2,4-triazole, an other amor solid, mp dcrmatizes at 134\(^\circ\)C (Ref 2).

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


Mononitrminoaminotriazoles, 3-Nitramino-

2-aminos-s-triazole,

\[
\begin{align*}
\text{HC} & \equiv \text{N} - \text{N.NH}_2 \\
\text{HC} & \equiv \text{N} - \text{N.NH.NO}_2 \\
\text{N} & \equiv \text{C.NH.NO}_2 \\
\text{N} & \equiv \text{C.NH}_2
\end{align*}
\]

mw 144.10, N 58.33%; obtd from the nitrate deriv by treatment with H\(_2\)SO\(_4\); its impact sensitivity expressed as FI is 47\% of PA and its power & brisanse are less than that of PA (Ref 2).


3,5-Dinitroamino-α-triazole Salts,

\[
\begin{align*}
\text{O}_2\text{N.HN.C} & \equiv \text{N} - \text{N} \\
\text{N} & \equiv \text{C.NH.NO}_2
\end{align*}
\]

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

Monoaminoguanidinium, C\(_3\)H\(_5\)N\(_4\)O\(_4\), rosettes of ndls (from w), mp 180\(^\circ\)C dec

Monoammonium, C\(_2\)H\(_4\)N\(_8\)O\(_4\), felted rosettes of fine wh ndls (from w), mp 182-84\(^\circ\)C decomp

Monoguanidinium, C\(_3\)H\(_4\)N\(_2\)O\(_4\), crysts (from w), mp 176-79\(^\circ\)C & 186-87\(^\circ\)C dec

Monopotassium, C\(_2\)H\(_2\)N\(_7\)O\(_4\)K, crysts (from w), mp 199-200\(^\circ\)C dec

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

Stolle & Dietrich (Ref 2) report a Di-
nitrate compd, pale-yel crysts, mp exploding when heated rapidly at 145\(^\circ\)C, was prep'd by treating a soln of guanazole in 2NH\(_4\)Cl with 65\% HNO\(_3\)


Diammine-cadmium-nitrate. See under Ammnes 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 1, 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 fuze and related items. This facility is now called Harry Diamond Laboratories

Ref: OrdTechTerm(1962), p 99

Diamylamine Perchlorate or Dipentylamine Perchlorate, [CH\(_3\).(CH\(_2\))\(_4\)]\(_2\)NH.HClO\(_4\), crysts, mp explodes at 325\(^\circ\)C was prep'd by double decomp of diamylamine HCl & Ag perchlorate (Ref 2)
Diamylether. See Amylether in Vol 1, p A396-L

\(\alpha\)-Diamyleose, \((C_6H_{10}O_5)_2\); mw 324.28; O 49.34%; was prep'd by Leibowiz & Silmann (Ref 5); Qcomb. 4285 cal/g (Ref 4); X-ray spectrum (Ref 6); L & S also prep'd some nitrated derivs which are expl: a Diamyleose tetrinate, \([C_6H_8O_3(NO_2)_2]_2\); amr solid, not isolated from reaction mixt; was present in alc extract of crude hexanate; a Diamyleose hexanate, \([C_6H_7O_2(NO_2)_3]_2\), cryts (from acet), mp defgr at 206-070°, was prep'd by nitration of a-diamylene or as the final product of the nitrization of tetramyleose.

Compare with Amylose in Vol 1, p A398-R


Diisophthalate, \(C_8H_8O_4\), 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), \(C_6H_4(OOC.CH_2.CH_2.CH_2.CH(CH_3)\_2\), col liq, bp 330-380° (dec) at 744 mm, d 1.0220 at 15.67/15.6°, nD 1.4871 at 20°, smells sl of isooamylalcohol; can be prep'd by ester interchange or "transesterification" of diethyl phthalate & C_6H_12 ONa (Refs 1 & 12)

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

CA 44, 2306 (1950) (Vapor press-temp rel-relationships and latent heat of vaporization)
6a) Kirk & Othmer 3 (1949), 407-08
7) J. Haslam & W. Soppe, J Appl Chem (London) 1, 112-19 (1951) & CA 45, 6869 (1951)
8) C.A. Powers & G.B. Ayres, USP 2694487 (1954) & CA 49, 3481 (1955)
10) K. Büchner, USP 2780643 (1957) & CA 51, 132924 (1957)
11) Sax (1957, 544)
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 Bell

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

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

a,β-Dianilino-ethane or N,N'-Diphenyl-ethylenediamine. 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

a-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 Dimethylxyphiphophenyl (called Dimerathoxy-diphenyl or Dianisol in Ger), CH3.O.C6H4.C6H4.O.CH3; mw 214.35, O 14.94%. Exists as
8α- and 8β-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, chl, glc acet ac & thiocarbonic acid, CH3(OH)2; insol in w (Ref 2)

p,p-Dianisole, crystals (from alc) or pltl (from benz), mp 172-73°; readily sol in benz, chl, hot alc; sl sol in eth; insol in petr eth & w (Ref 3)

Other props & methods of prepn are given in Refs


Dinitroanisole, [-C6H3(NO2)O.C6H3]2; mw 304.25, N 9.21%. Several isomers are known:
3,5'-Dinitro-o,o'-dianisole or 3,5'-Dinitro-2,2'-dimethoxybiphenyl, crys, mp 133-35° (Ref 3)
4,4'-Dinitro-o,o'-dianisole or 4,4'-Dinitro-2,2'-dimethoxybiphenyl, crys, 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, chl & alc; sol in benz; sl sol in glc acet & hot acet; insol in petr eth & water (Ref 1)
6,6'-Dinitro-o,o'-dianisol or 6,6'-Dinitro-2,2'-dimethoxybiphenyl, yel crys, mp 226-28° (Ref 6) & 231-32° (Ref 8)
2,2'-Dinitro-p,p'-dianisole or 2,2'-Dinitro-4,4'-dimethoxybiphenyl, crys, mp 136-37° (Ref 4 & 7)
3,3'-Dinitro-p,p'-dianisole or 3,3'-Dinitro-4,4'-dimethoxybiphenyl, crys, mp 221° (Ref 2) & 214° (Ref 5)
2,6-Dinitro-3,5-dimethoxybiphenyl, C6H3.C6H4(NO2)2.OCH3); pale-yel prisms (from ethyl acetate), mp 260-61° (Ref 10)

Other props & methods of prepn are given in the Refs

Tetranitrodianisole, [-C₆H₂(NO₂)₂.O.CH₃]₂; mw 394.25, N 14.21%. The following isomers are reported in the literature:
3,3,3',5'-Tetranitro-2,2'-dimethoxybiphenyl, exists in two forms: lower-melting crystals (from acet or acel), mp 179° & higher-melting crystals, mp 191°, which are mutually interconvertible; was prep'd by reaction of 2,2'-dimethoxybiphenyl & ethyl nitrate in concd H₂SO₄ 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 prep & props are given in the above Refs.
4,4',6,6'-Tetranitro-2,2'-dimethoxybiphenyl, exists in two forms: lower-melting crystals, mp 168° convertible to higher-melting crystals (from acel), mp 180°; was prep'd by nitrating guaiacol [o-methoxyphenol, C₆H₅OH(OH)CH₃] to the dinitro deriv, which was converted to the 2,3,5-Cl(O₂N)₂C₆H₄OCH₃, and by boiling in Nitrobenez with Cu bronze for 1hr (Ref 9, p 453).
4,4',6,6'-Tetranitro-3,3'-dimethoxybiphenyl, dry crystals, mp 244°; was prep'd from 3,3'-dichlorobiphenyl which was first nitratd to 4,4',6,6'-tetranitro-3,3'-dichlorobiphenyl, and by action of Na methanol in boiling MeOH on the tetrachloro deriv (Ref 9, p 361).
3,3',5'-Tetranitro-4,4'-dimethoxybiphenyl, almost col ndls (from concd HNO₃), mp 242-55°; was prep'd by nitrating 4,4'-dimethoxybiphenyl with HNO₃ (Refs 2 & 7).

No expl props of the tetranitro derivs were reported.

References:

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

Dianisoyl Peroxide or Anisoyl Peroxide, (call'd Dianisoylperoxyd in Ger), CH₃O.C₆H₄.CO.OO.OC.C₆H₄.OCH₃; mw 182.17, O 52.70%; the p-deriv is colorless & tasteless, crystals (from eth acet), mp 128°; degr by heating on a Pt foil or in a capillary tube; insensitive to impact or pressure; degr in contact with concd H₂SO₄; sol in eth acet; diff sol in petr eth; can be prep'd by adding dropwise a soln of 3% H₂O₂ in pyridine to a soln of anisoyl chloride in acetone, and allowing the mix to stand for 12hrs (Ref 1 & 2). Hey & Walker (Ref 3) reported a prep by reaction of aryl nuclei with acyl peroxides. See also Refs 4, 5, 7, 9, 10 & 13 for prep & decomp in soln.

Its IR absorption spectrum is given by Davidson (Ref 6).

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

**Diaphragm Gauges.** See under Gauges

**Diallyl Phthalates in Propellants.** Substances such as diphenylylphthalate (qv) were proposed for coating the grains of NC or NG propInts to serve as detergents, 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 (nitratd 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

**Diazo.** A prefix denoting replacement of two ring carbons by nitrogen(s), and applied to names of heterocyclic comps derived from names of carbocyclic comps

*See also:* Aza, Vol 1, p A516-R

**Diaza Alkanes, Polynitro.** They are prepd by acidifying the appropriate amine with HNO3 to form comps useful as expls. Thus the compd bis(1,1,1-trinitroethy1-2-N- nitroethylen-imine)-dinitromethane, [(CH2)2NH—CH2.C(NO2)2]2C(NO2)2, was added to a 5% soln of 100% HNO3 & AcOH. The soln was poured on ice, and the wh ppt was washed with w, and recryst from hot 100% HNO3. The product, [(CH2)2N(NO2)2CH2.C(NO2)3]2C(NO2)2, mp 170-75°, Qcombstn 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-dinitromethyl-3-N-nitroethylen-imine)-dinitromethane, [(CH2)2N(NO2)2CH2.C(NO2)2]. CH3C(NO2)2, was similarly prepd from the appropriate amine

*Ref:* M.B. Frankel, USP 2978510(1961) & CA 55, 15934(1961)

**1,4-Diaza cyclohexane.** See Piperazine

**Diaza cyclohexanone.** See Tetrahydropryrimidone

**1,3-Diaza cyclohexene.** See Tetrahydropryrimidine

**1,3-Diaza cyclopentane.** Same as Cyclo-2,4,5-trimethylene-1,3-diamine (qv) or Imidazolidine, Vol 3, p C610-R
Diazacyclopentanone. See Imidazolidone or Ethyleneurea

1,3-Diazacyclopentene. See Imidazoline

4,7-Diazadecane; 2,2,4,7,9,9-Hexanitró., \([\text{H}_2\text{C} \cdot \text{C(NO)}_2 \cdot \text{CH}_2 \cdot \text{N(NO)}_2 \cdot \text{CH}_2 \cdot \text{J}_2]_2\);

mw 428.26, N 29.44%. An exptl 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-Diazahexane; 1,1,1,3,5,7,7-Octanitro; \(\text{CH}_2 \cdot \text{N(NO)}_2 \cdot \text{CH}_2 \cdot \text{C(NO)}_2 \cdot \text{3J}_2\); mw 546.23, N 41.03%; crysts (from cyclohexane), mp 84-85\(^\circ\), Qcombstn 1423 cal/g, Trauzl Pb block value 196% (TNT = 100), Ballistic Mortar value 156% (TNT = 100); was prepd by condensing 2-trinitro-1-ethanol, \(\text{C(NO)}_2 \cdot \text{3J}_2 \cdot \text{CH}_2 \cdot \text{OH}\), with \(\text{CH}_2 \cdot \text{N(H)}_2 \cdot \text{2J}_2\) to form the hexanitro compd, \(\text{CH}_2 \cdot \text{N(H)} \cdot \text{CH}_2 \cdot \text{C(NO)}_2 \cdot \text{3J}_2\), which was acidified with HNO\(_3\) to yield the useful exp1 octanitro compd


2,5-Diazahexane. Same as N,N\(^\prime\)-Dimethyl-ethylenediamine

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 Phthaly Diazide under Phthalic Acid

Diazido. A prefix indicating the presence of two azido groups, \(-\text{N} \cdot \text{N} \cdot \text{N}\) also designates \(-\text{N}_2\) in org compds. See also Azides, Organic in Vol 1, p A626-L

\(\alpha\)-Diazido dipic Acid or Ditriazo dipic Acid (called by Bertho & Maier \(\alpha\)-\(\alpha\)-Diazido dipicinsäure),

\(\text{HO} \cdot \text{OC} \cdot \text{CH(N)}_3 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CH(N)}_3 \cdot \text{CO} \cdot \text{OH}\);

mw 228.17, N 36.84%; col crysts (from w) mp 147\(^\circ\) (decompn); was prepd from ethyl-\(\alpha\),\(\alpha\)-dibromoadipate and \(\text{Na}\) azide in ethanol with subsequent alk hydrolysis. Its exp1 props were not examined.

(Compare with Adipyldiazide, described under Adipic Acid, p A104-R in Vol 1 of this Encycl Refs: 1) Beill-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(\(\alpha\)-azidobenzylidene)-azine. (called in Ger Dibenzhydrazidazid). See Bis(\(\alpha\)-azidobenzylidene)-azine in Vol 2, p B133-L

Di(\(\alpha\)-azidobenzylidene)-hydrazine. (called in Ger Dibenzyhydrazidhydrazid). See Bis \(\alpha\)-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

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

Diazooacetamide. (called Diazoessigsäure-amid or Diazooacetamid in Ger), N=NCH.CO.NH₂; mw 85.07, N 49.40%; golden-yel prisms (from alc), mp 114° (dec); sol in w, alc & concd aq NH₃; prep & other props are given in Beil Ref: Beil 3, (215-16)

Diazooacetic Acid. (called Diazooessigsäure in Ger), N₂CH.CO.OH; mw 86.05, N 32.56%; The free acid is not stable, but it forms some metallic salts which are expl: Potassium salt, KC₂HN₂O₂, yel ndls or lemon-yel lfts, expls violently with a loud noise on heating (Refs 1 & 2) and Sodium salt, NaC₂HN₂O₂, yel crys (from w), gm-yel ndls (from w + alc), 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)

Diazooacetic Acid, Ethyl Ester; Ethyldiazooacetate or Diazooacetic Ester (called Diazo-essigsäure-äthylester or Diazooessigester in Ger), N₂CH.CO₂.C₂H₅; 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°, mp 1.4616; v sol in alc, benz, ligroin & eth; sl sol in w; was prep'd by action of aq NaNO₂, in dil H₂SO₄, on glycine ethyl ester hydrochloride, H₂N.CH₂.CO.O.C₂H₅.HCl (Ref 1)

Its Mercury salt, Hg(C₄H₅N₂O₂)₂, yel rhmb crys (from eth), mp 104°(dec); sl sol in alc & eth; prep'd by treating diazoacetic ester with yel HgO, while cooling with ice; deflagrates or explodes mildly on strong impact (Ref 1, p 215)
Diazoacetic Acid, Methyl Ester or Methyl diazoacetate (called Diazoessigsäure-methylester in Ger), N₂CH.CO₂.CH₃; mw 100.08, N 27.99%; lt-yel expl liq, bp 31° at 12 mm Hg; distillation, even under reduced pressure, is extremely dangerous, since heat causes the compd to detonate violently; d 1.158 at 25°, nD₂ 1.4515 & nD₂ 1.4676 at 20.6°; was prep'd by diazotization of aminoaetic acid methyl ester with HNO₂ (Ref 1, 2 & 3). Its Mercury salt, Hg(C₂H₃N₂O₂)₂, sulfur-yel crys (from eth), mp 125° (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)


Diazoacetone or Diazo-2-propanone (called Diazoacetone, 1-Diazo-propanon(2), or Acetyl-diazomethan in Ger), N₂CH.CO.CH₃; mw 84.08, N 33.32%; lt-yel oil, fr p does not solidify at -18°; forms lt-yel crys with liq CO₂, bp 45-47° at 15 mm of Hg; explodes on heating, d 1.0661 at 21.7°; miscible in equal proportions of a mix of alc, eth & w; was prep'd from acetyl chloride & diazomethane in ether or from diacetyl diamethane & dil NaOH at 0° (Ref)

Ref: Beil 1, (396), [823] & [3093]

Diazoacetonitrile (called Diazoacetonitril or Diazoessigsäurenitril in Ger), N₂CH-CN; mw 67.05, N 62.67%; orn-yel, sl elec conductive liq, fr p does not solidify at -18°, bp 46.5° at 14-15 mm 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 obt'd in low yield by reaction of HNO₂ 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


DIAZOAMINOINZEN AND DERIVATIVES

Diazoaminobenzene, Diazobenzeneanilide, or Benzeneazoanilide (called Diazoomido benzene by Sax (Ref 5); called Diazooaminobenzol or 1,3-Diphenyl-triazien-(1) in Ger), C₆H₅.N:NH.C₆H₅; mw 197.23, N 21.31%; yel crys (from alc) or plts (from benz), mp 98-100°, bp explodes when heated to 150° (Ref 6); sol in eth & alc; med sol in hot alc; v sl sol in cold alc; insol in w; has been prep'd by the action of NaN₂ on aminline sulfate or aniline hydrochloride, by the action of NaN₂ & 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


Mononitrodiazoinobenzene, O₂N.C₆H₄.N:NH.C₆H₅; mw 242.23, N 23.13%. The 2-Nitro, orn-yel nds (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 decomp) are described in Beil

Ref: Beil 16, 696, 697-99, (406) & [354]

Dinitridiazoinobenzene, O₂N₂.C₆H₄.N:NH.C₆H₄.NO₂; mw 287.23,
N 24.38%. The following derivs are the more important dinitro compds:
2,2'-Dinitro-diazooaminobenzene, yel ndls or scales (from alc), mp 196-96.5°; diff sol in boiling alc (Refs 1, 3 & 4)
3,3'-Dinitro-diazoaminobenzene, straw-yel
ndls (from alc), mp 195-96° (violent decomp); 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 decomp); sl sol in chlf & benz; diffic
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 Amino-
diazoaminobenzene in Vol 1, p A195-L

6-Diazo-3-aminobenzoesäure in Ger),
O — CO
N 2, C — C — CH

CH — CH = C.NH₂ mw 163.13, N 25.76%;
brass-yel ndls or lfts + 3.H₂O; loses w of
crystm by heating on a water bath, mp exp
plodes on heating; mod sol in hot w; sl sol in
hot alc; insol in eth; was prepd by treating
2,5-diaminobenzoeic acid with nitrous
acid. This compd does not exhibit the char
acteristics of an acid. It possesses 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

Diazoamin Compounds. They are described,
with few exceptions under individual diazo-
amino compds. However, several general Refs
on this subject are given below
Refs: 1) A. Mangini & I. Dejudicibus, Gazz-Chimital 63, 601-12 (1933) & CA 28,
1672-74 (1934) 2) A. Mangini, Gazz-Chimital 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'-Diazo-
amino-di-1-H-Tetrazole [called 5,5'-Diazo-
aminotetrazol or 1,3- Di-[tetrazolyl-(5)]-triazene
in Ger],
N — C. NH.N:N.C — — N
N — N — NH
NH—N — N

mw 181.13, N 85.07%; almost col, lustrous,
thin doubly refractive plates + 1 mole H₂O;
was obtd by treating aminoguanidine nitrate
with NaNO₂ in the presence of dil acetic
acid, or by diazotizing aminotetrazole (Refs 1 & 2)
Its Copper salt, Cu₃(C₂N₁₁)₂+ 2NH₃,
dk-grn plts, explodes by friction, impact or
heating; this salt with KClO₃ is a more
powerful primer mixt than MF·KClO₃ (Ref 2);
Silver salt, Ag₂C₂N₁₁ + H₂O, powd,
explodes on heating or strong friction; and
Barium salt, Ba₃(C₂N₁₁)₂ + 8H₂O, yel plts
(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)
**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

**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.

---

**Diazaminomethane.** Same as Dimethyltriazene

**Diazaminonaphthalene [called 1,1'-or a,a'-] Diazaminonaphthalin or 1,3-Di-a-naphthyltriazene-(1) in Ger], C_{10}H_{7}:N:NH.C_{10}H_{7}; mw 297.34, N 14.13%; yel-bm tiny plates 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 a-naphthyl-amine hydrochloride
Ref: 1) Beil 16, 716 2) Sax(1957), 716

**Diazaminonaphthalene Bromide Hydrobromide,** See Aminodiazaminonaphthalene Bromide Hydrobromide in Vol 1, p A195-L

**Diazoanhydride.** Same as 1,2,3-Oxadiazo1

**4'-Diazooazobenzene-4-sulfonic Acid [called 4'-Diazooazobenzol-sulfonsäure-(4) in Ger], N:N.C_{6}H_{5}.N:C_{6}H_{4}.O_{2}S; mw 288.24, N 19.44%**

---

pale-yel ndls, mp - explodes on heating; sol in concd KOH; insol in org solvs; was prep'd by Griess (Ref 2) on passing nitrating 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)
Diazocamphor. See Vol 2, p C23-R

Diazocarbazole. See Vol 2, p C47-L


See also Diazocompounds

Diazochloronitrophenol. See Mononitrodiazochlorobenzoquinone and Derivative, Vol 3, 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 prepd 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, Diazodinitrochlorogluinol 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 Diazochromestry, Diazohycrocarbons and Diazonium Salts


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

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

4-Diato-2,5-cyclohexadien-1-one (called p-Benzquinonediazide by Anderson et al (Refs 2 & 3) and also Diazophenol (Ref 1)), CH—C(=O)—CH
\[CH—C(=N)—CH\]
\[CH—C(=N)—CH\]

CH 1.08.10, N 25.92%; colorless ndls + 4H2O, mp 38-390, explodes violently at 750, sl sol in w & alc; diffc sol in eth & benz; was obtd by digesting an abs alcoholic soln of p-hydroxybenzenediazoinium chloride with Ag oxide and cooling the filtrate to -300. This compd & its derivs are light-sensitive material. Its salt, C6H4N2O + C2H2, explodes at 1680 (Ref 1).

The Mononitro derius are described under Benzenediazioxide, Vol 2, p B58


Diazocyrolmethane. See under Cyclodiazo Compounds in Vol 3, p C593-L
Diazocyclopentadiene, CH\(_2\)C(=N N) = CH
\[\text{CH} \quad \text{C(=N N) = CH}\]
mw 92.10, N 30.42%; red liq, bp 52-53° at 50mm, d 1.059, n\(_D\) 1.6150; yel crys (from alc, then pentane at -70°), mp -23 to 22°; was prepd by reaction of a suspension of cyclopentadienylium with a cooled, stirred soln of p-toluenesulfonamid 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 prep, they reported, a violent expln took place after distillation.


Diazocymol, C\(_{10}\)H\(_4\)N\(_2\)O. Its expl deriv is 5-Nitroso-p-cymol-2-diazonium nitrate or 6-Nitroso-p-cymol-3-diazonium nitrate,

\[\text{CH}_3\]
ON.C\(_6\)H\(_2\)N(=N).O.NO\(_2\)

\[\text{CH(CH}_3)_2\]
mw 252.23, N 22.22%; canary-yel lt flakes, mp 56-60° (dec) and explodes on heating at higher temp and on impact; difff soln in org solv; was prepd by action of nitrogen dioxide on p-dinitrosocymol, or on quinonedioxime.

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

Diazoo-o-dihydroxybenzene. See Diazopyrocatechol

Diazoo-m-dihydroxybenzene. See Diazoresorcinol

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

Diazodinitrophenol, Analytical Procedures. Qualitative Tests

Accdg to StdMethodsChemAnalysis, 28 (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-brown 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 crys 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 hotn 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 1/4 x 1/4 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 crystals 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 (W₁), 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, (W₂) and calculate the bulk density from the formula: Bulk d = W₂ - W₁

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 hr; 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: \( B = \text{weight of AgCl in crucible (B); and} \)
\( A = \text{wt of dry sample in crucible (A)} \)

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 chloride (percentages are not given). Its US military requirements were covered by Specification MIL-S-10641A (Oct) (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.

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.

Percentage of DADNPh = 100(A-B-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 Compan M11A1 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 dis-appearance 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.

Percentage of K Chlorate = 100(A-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°C for 2 hrs, cool in a desiccator and weigh.

Percentage of NS = 100(B-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 calc'd from the following equation:

Percentage of Wood Charcoal = 100(C-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 calc'd by subtracting from 100 combined percentages of DADNPh, NS and charcoal, after detg the charcoal content.
2,2'-Diazoo-3,3'-dioxo-4,6,4',6'-tetrinitromethyl-biphenol or Bis(2-diazo-3-oxy-4,6-dinitro)-m,m'-biphenol. See Vol 2, p B122-L

DIAZIDIPHENYLAMINE AND DERIVATIVES

4-Diazodiphenylamine (called 4-Diazodiphenylamino-formaldehyde or p-Chinon-anil-diazid in Ger), C_6H_5N:C_6H_4:N_2; mw 195.22, N 21.53%; bnyel 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-amino-aminodiphenylamine with NaNO_3 & excess HCl and allowing the cold diazoniumchloride soln to stand with dil NH_3 added

Refs: 1) Beil 16, 603 2) A. Hantzsche, 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-Diazolo-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-amino-diphenylamine hydrochloride soln was diazotized, and well-cooled NH_3 was added to a Na acetate soln of the diazo-

4-Diazolo-4 nitrodiphenylamine, red crstls having a Cu luster, mp - explodes when heated to 60-65° or by friction; was prepd by diazotizing 4'-nitro-4-amino-diphenylamine and treating the diazonium salt with NH_3

Refs: 1 & 2, p 11

4-Diazol-2',4'-dinitrodiphenylamine, (O_2N)_2C_6H_3.N:C_6H_4:N_2; mw 285.22, N 24.56%; red crstls, decmpn on exposure to light, mp - expl 110-15°; was prepd by treating an aq soln of 1-(2,4-dinitroanilino)-benzenediazoniumchloride with KHC_3

Ref: 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%; bnm-red crstls (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:NN:MW 56.07, N 49.92%; gas, bp -19 to -17° at 89,5 mm press; can be prepd by reaction of N-nitromso-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 Diazooäthan or Diethylhyponitrit in Ger), C_2H_5.O:N.O.C_2H_5; mw 118.14, N 23.71%; oil lighter than w, bp - on heating begins to decmp and explodes at 80-82°; cannot be dis-
tilled even at low press; readily sol in alc, acet & benz.; insol in water; d 0.953 at 16°, d 1.0326 at 20°; n_2 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


1-Diazoethylbenzene of Methylphenyl-diazomethane (called (α-Diazo-ethyl)-benzol or Methyl-phenyl-diazomethane in Ger), C_6H_5.CN.2.CH_3mw 132.16, N 21.20%;
It red crysrs (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; soln in org solvs; was obsd 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 Diazooethylbenzene
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. See Cyanoazo-
guanidine in Vol 3, p C587-R

Diazoguanidine Picrate. CH₄N₅ + C₆H₅N₃O₇,
This salt is mentioned as one of the ingre-
dients of a detonator chge. It may be mixed
with Tetryl. The detonator chge is made by
mixing a primary initiation chge (L-A 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 Austra-
alia & New Zealand, AustralianP 102202
(Oct 1937) & CA 32, 2753 (1938)

Diazoguaniloxazide or Nitroso-aminoguanil-
oxide (called Diazoguaniloid or Nitroso-
aminoguaniloid in Ger),
N₃...C(NH)...NH.N.NH₂ or N₃...C(NH)...NH.
NH.NO3; known only in the form of its salts
such as:
Sodium salt, NaCHN₇O, obtb by treating
guanyl-diazoguaniloxazide with dil NaOH.
The add of Cu acetate gives the Copper
salt, CuCHN₇O, light greenish-blue plts,
very expl salt
Refs: 1) Beil 3, (60) & 12391 2) K.A.
Hofmann et al, Ber 43, 1093 (1910) & JCS
98 I, 446 (1910)

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

Diazohydrocarbons. 1-Substituted-1-nitroso-
3-nitourgauinidines when treated w/ aq KOH
gave diazohydrocarbons. McKay et al (Ref)
used this method to prepare Diazomethane,
Diazoothene, Diazoo-propane and other
Diazocompds. See Ref 2 for their decomp
studies.

See also Diazochemistry and Diazo-
compounds
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)
(Decomp of aliphatic Diazocompds by tri-
methyl borate)

Diazohydrazides. This name was given by
Wohl & Schiff (Ref 2) to compds of the general
formula R¹.N:N.R².NH₂, where R¹ is an H or
alkyl & R² is an ary1 group. These compds
may be considered as derivatives of hypotheti-
ical "tetrazene", HN:N.NH₂, and they re-
ssemble the compd

```
H₃C
N.N:N=N
C₆H₅
```

prep'd earlier by Fischer (Ref 1) and called
"Dimethylidiphenyltetrazene". Wohl & Schiff
(Ref 2) prep'd a number of similar compds and
suggested that the name "Diazohydrazides" was
simpler. It allowed isomersides to be
readily distinguished and was in better agree-
ment with their chem nature

Diazohydrazides are crys substances
which explode when heated but not when sub-
jected 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 Bisdiaizotetrazones, R N:N.NR² .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, 274-58 (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) (Diazohydra-
zide from Diazotetrazole)
5-Diazo-2-hydroxybenzoic Acid or 5-Diazo-salicylic Acid [called 5-Diazo-salicylsäure or Benzochinin-(1,4)-diazid-(4)-carbonsäure-(2) in Ger]

\[
\begin{align*}
\text{COO-} & \quad \text{COOH} \\
\text{HC} & \quad \text{C} \equiv \text{C.OH} & \quad \text{HC} & \quad \text{C} \equiv \text{C}:0 \\
N_2C & \quad \text{CH}=\text{CH} & \quad \text{or} & \quad N_2C & \quad \text{CH}=\text{CH} \\
\end{align*}
\]

m.w. 164.12, N 17.07%; yell ppt (from w) or blue-gray ndls; mp - explodes 155-162⁰; mod sol in hot w & aq Na₂CO₃; sl sol in cold w & org solvs; was prepd by diazotizing the hydrochloride of 5- amino-salicylic acid with NaNO₂ 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-oxo-benzoësäure or Benzochinin-(1,4)-diazid-(1)-carbonsäure-(2) in Ger]

\[
\begin{align*}
\text{COO-} & \quad \text{COOH} \\
\text{HC} & \quad \text{C} \equiv \text{C.OH} & \quad \text{HC} & \quad \text{C} \equiv \text{C}:0 \\
N_2C & \quad \text{CH}=\text{CH} & \quad \text{or} & \quad N_2C & \quad \text{CH}=\text{CH} \\
\end{align*}
\]

m.w. 164.12, N 17.07%; ndls, mp - explodes at 169⁰; diffc sol in cold w; was prepd by diazotizing 6-amino-3-hydroxybenzoic acid with NaNO₂ in ice-cold dil HCl


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

Diaziomide of Benzenesulfonfyltolylene. See Benzenesulfonfyltolylenediaziomide in Vol 2, p B62-R

Diaziomide of Benzenesulfonfylxylylene. See Benzenesulfonfylxylylenediaziomide in Vol 2, p B63-L

Diaziomide. 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-Diazo or Pyrazole (called Pyrazol in Ger),

\[
\text{HC} \quad \text{NH} \quad \text{N} \\
\]

HC ———— CH; m.w. 68.08, N 41.15%; ndls or prisms (from petr eth), mp 69-70⁰, bp 184-85⁰ at 719mm of Hg; d 1.0012 at 99.8⁰; sol in w, alc, eth & benz; can be prepd by reaction of a-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-Diazo or 4-Nitro-pyrazole (called 4-Pyrazol in Ger),

\[
\begin{align*}
\text{HC} & \quad \text{NH} \quad \text{N} \\
\end{align*}
\]

\[
\text{O}_2\text{N.C} ———— \text{CH}; \\
\]

m.w. 113.08, N 37.16%; ndls (from benz), mp 162⁰, explodes when heated on a Pt foil, bp 323⁰, sol in alc & eth; sl sol in cold w & benz; was prepd by nitration of 1,2-diazo with mixed HNO₃ - H₂SO₄ acids and by other methods (Ref). Other props are given in Beil

Refs: 1) Beil 23, 44 2) F. Reicheneder & K. Dury, BelgP 660636(1962) & CA 63, 18096(1965)

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

1,3-DIAZOLE AND DERIVATIVES

1,3-Diazo, Glyxoxaline or Imidazole (called Imidazol or Glyoxaline in Ger),

\[
\text{HC} — \text{NH} — \text{CH} \\
\]

\[
\text{HC} — \text{N} ; \text{m.w. 68.08, N 41.15%}; \text{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 & chl; mod sol in w & alc; sl sol in eth; can be prepd from glyxal & NH₃ with or without the addn of formaldehyde; from imidazol-thione-2 & HNO₃; 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, 2C3H4N2+ AgNO3, crysts (from alc), mp 129-31°; explodes on heating to a higher temp (Ref 1) 


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

\[
\text{HC} \equiv \text{NH} \equiv \text{CH} \quad \text{O}_2 \text{N}_2 \text{C} \equiv \text{NH} \equiv \text{CH}
\]

\[
\text{O}_2 \text{N} \text{C} \equiv \text{N} \quad \text{or} \quad \text{HC} \equiv \text{N} \equiv \text{N} \equiv \text{H}
\]

mw 113.08, N 37.16%; ndls (from alc or glacial acetic acid), mp 312-13°, sol in hot HCl & glacial acetic acid; sl sol in hot w & alc; insol in eth & chlf; can be prepd by heating imidazole with ntriosyl 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 nitratd derivs of 1,3-Diazole were not found in Beil or in CA thru 1961.

Diazomethane (called Diazomethane in Ger), CH2:NN:; 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 prep are from nitroso-methyleurea, 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


Iso-Diazomethane (called Isodiazomethane in Ger)

\[
\begin{align*}
\text{H} & \equiv \text{C} \equiv \text{N} \quad \text{or} \quad \text{H} \equiv \text{C} \equiv \text{N} \\
\text{H} & \equiv \text{C} \equiv \text{N} \equiv \text{H} \\
\text{N} & \equiv \text{N} \equiv \text{H}
\end{align*}
\]

mw 42.04, N 66.64%; pale-yel oil of low viscosity, fr p - begins to decom at 15° and then explodes at 35-40°; can be synthesized from an ether suspension of Diazomethyl lithium, (CH2N2)Li, by acid hydrolysis. Diazomethyl lithium explodes on contact with air; is obtbd by reaction of N2O & CH3Li or from diazomethane & CH3Li (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)


Diazomethanedisulfonic Acid or Diazomethionic Acid (called Diazomethanedisulfonsäure or Diazomethionsäure in Ger), N:N=CSO3H2; obtbd in the form of its Potassium salt, K2CO3N2S + H2O, yel crysts or om-yel ndls or prisms, mp - explodes ca 210°; mod sol in w; was prepd by treating K aminomethanedisulfonate with HNO2 and by other methods

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}, yellow amorphous
powder, mp - explodes violently at 110°; diffe-
sol in w
nitrato: C_{14}H_{10}SN_{3}.NO_{3}, yellow-brown ppt which
explodes in dry state at 145°
chromate: C_{14}H_{10}SN_{3}.HCrO_{4}, yellow ppt which
detonates at 120°
carbonate: C_{14}H_{10}SN_{3}.HCO_{3}, red-brown amorph-
ous powder, mp 128° with weak explosion
nitroprusside: (C_{14}H_{10}SN_{3})_{2}[Fe(CN)_{5}NO],
ltr yellow 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-
axylyl-3-diazotoluene by Hodgson & Ward),
C(N_O) — C(O) — C — CH_{3}
\[\begin{array}{c}
\text{CH} \\
\text{C(NO) — CH} \text{; mw 179,13, N} \\
23,46\%; lr-yel crysts (from hot w), mp 123°
\text{ (decomp)}; was obtb as an intermediate when
5-nitro-3-amino-ocresol was diazotized
\end{array}\]
Refs: 1) Beil-not found 2) H.H. Hodgson
& F.R. Ward, JCS 1945, 665 & CA 40,
1149 (1946)

6-Diazo-5-methyl-2-nitro-2,4-cyclohexadiene
-1-one (CA nomenclature) (called 3-Methyl-
6-nitro-1-chinon-2-diaizd in Ger),
C(N_O) — C(O) — C.NO_{2}
\[\begin{array}{c}
\text{CH_{3}C} \\
\text{CH} \\
\text{CH} \text{; mw 179,13, N} \\
23,46\%; rust-brown, flat ndls, mp 128-29°; 
explodes violently on rapid heating; was obtb
\end{array}\]
by adding KI dropwise in water to 2,3,4-H_{2}N.
(NO_{2})_{2}C_{6}H_{5}.CH_{3} in concd H_{2}SO_{4}, after
treatment with HSO_{3}NO_{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

Diazonaphthalen or Naphthalenediazonium
Hydroxide, C_{10}H_{7}N_{2}OH, 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)-Diazonaphthale [called 1(or a)-
Diazonaphthalin or Naphthalin-diazonium-
hydroxyd-(1) in Ger], C_{10}H_{7}N_{2}(N).OH; forms
the following unstable salts:
Borofluoride, C_{10}H_{7}N_{2}.BF_{4}, mp - dec at 113°
Chloride, C_{10}H_{7}N_{2}.Cl, om-yel crysts (from alc),
mp 96° (dec) and explodes on further
heating
Dichloroiodide, C_{10}H_{7}N_{2}.Cl_{2}, lr-yel fine
cryst powd, mp 98° (dec)
Mercuric chloride, C_{10}H_{7}N_{2}Cl + HgCl_{2} lr-
yel crysts, mp 120-21° (dec)
a-Naphthalenethiosulfonate, C_{10}H_{7}N_{2}.SS.O_{2}.
C_{10}H_{7}, brn crysts, very unstable
Nitrato, C_{10}H_{7}N_{2}O.NO_{2}, col crysts, mp explodes on heating, and detonates violently
by impact or friction
Perchlorate, C_{10}H_{7}N_{2}.ClO_{4}, double-refractive
crysts, very expl when dry
Sulfate, C_{10}H_{7}O.SO_{3}H, yel crysts (from alc +
eth), mp 115° (dec) and exploding mildly

2(or beta)-Diazonaphthale [called 2(or beta)-
Diazonaphthalin or Naphthalin-diazonium-
hydroxyd-(2) in Ger], C_{10}H_{7}N_{2}(N).OH; forms,
with bases, some expl salts:
Benzedithiosulfonate, C_{10}H_{7}.N_{2}.SS.O_{2}.C_{6}H_{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 acetic), 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\textsubscript{10}H\textsubscript{7}.N\textsubscript{2}.Cl\textsubscript{2}I, lt-yel crystals, mp 90° (dec)
Mercuric chloride, C\textsubscript{10}H\textsubscript{7}.N\textsubscript{2}.Cl + H\textsubscript{2}Cl\textsubscript{2}, yel crystals, mp 120-25° (dec)
a-Naphthalenetrisulfonate, C\textsubscript{10}H\textsubscript{7}N\textsubscript{2}.S\textsubscript{3}SO\textsubscript{2}C\textsubscript{10}H\textsubscript{7}, lt-yel crystals, very unstable
Nitrile, C\textsubscript{10}H\textsubscript{7}.N\textsubscript{2}.O.NO\textsubscript{2}, col ndls (from glacial acetic acid or MeOH), explodes on heating or impact
Perchlorate, C\textsubscript{10}H\textsubscript{7}N\textsubscript{2}.ClO\textsubscript{4}, crystals, very expl when dry
Sulfate, C\textsubscript{10}H\textsubscript{7}.N\textsubscript{2}.O.SO\textsubscript{3}H, wh plts (from MeOH + eth), mp, explodes on heating
p-Toluenesulfonate, C\textsubscript{10}H\textsubscript{7}.N\textsubscript{2}.S\textsubscript{2}O\textsubscript{2}.C\textsubscript{6}H\textsubscript{4}.CH\textsubscript{3}, yel ndls, very unstable
\textit{Unnamed Salt}, C\textsubscript{10}H\textsubscript{7}.N\textsubscript{2}.Br + 2CuBr, fiery red ppt, dec on standing at RT
\textit{Unnamed salt}, 2C\textsubscript{10}H\textsubscript{7}.N\textsubscript{2}.SCN + CO(SCN)\textsubscript{2}, pale-green ppt, mp - dec at 70° and explodes on contact with H\textsubscript{2}SO\textsubscript{4} (See Ref 2)

\textbf{iso-Diazonaphthalene} [called \textit{a\& \textbeta}-Naphthalin-isodialdehydydroxyl or \textit{a\& \textbeta}-Naphthalin-anti-diazodyhydroxyd in Ger],
C\textsubscript{10}H\textsubscript{7}.N OH; forms with bases, some expl salts:
The \textit{\textbeta}-\textit{Silver salt}, AgC\textsubscript{10}H\textsubscript{7}N\textsubscript{2}O, wh crystals, explodes on heating rapidly (See Ref 3)

\textbf{Nitro Derivatives of Diazonaphthalene}
\textbf{Meronitro Diazonaphthalene}, O\textsubscript{2}N.C\textsubscript{10}H\textsubscript{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
\textit{Nitrate}, lt-yel ndls, explodes mildly on heating (Ref 1)
5-Nitro-1-diazonaphthalene, the normal sodium salt, ppt (from alc + eth), begins to decom below 100°; the isodialdehyde salt, dk-yel ndls (from alc + eth), dec at 165° (Ref 2)
1-Nitro-2-diazonaphthalene or 1-Nitronaphthalene-2-diazoniumhydroxide, its \textit{Nitrate salt}, O\textsubscript{2}N.C\textsubscript{10}H\textsubscript{6}.N\textsubscript{2}.ONO\textsubscript{2}, lt-yel ndls, explodes on heating; the \textit{Sulfate salt}, O\textsubscript{2}N.C\textsubscript{10}H\textsubscript{6}.N\textsubscript{2}.O.SO\textsubscript{3}H, or-yel ndls explodes mildly on heating (Ref 3)
6-Nitro-2-diazonaphthalene or 6-Nitronaphthalene-2-diazoniumhydroxide, forms a Sulfate pt, which decom in acid soln at RT (Ref 2)

\textbf{NOTE:} Higher nitrated derivs of Diazonaphthalene were not found in Beil or in CA thru 1961

\textbf{DIAZONAPHTHOL AND DERIVATIVES}
\textbf{Diazonaphthol}, N\textsubscript{2}.C\textsubscript{10}H\textsubscript{6}.OH; mw 170.16, N 16.46%. Two isomers are known:
2-Diazo-1-naphthol [called 2-Diazo-naphthol-(1) or Naphthochinin-(1,2)-(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\textsubscript{2} on 2-amino-1-naphthol in HCl in the presence of Cu sulfate, and by other methods (Ref 1). Its 4-Bromo deriv yol ndls (from w), decmp violently at 148-51°
1-Diazo-2-naphthol [called 1-Diazo-naphthol-(2) or Naphthochinin-(1,2)-(2) in Ger], wine-yel or yol-bm prisms from pret eth or liq 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)

\textbf{Nitroderivatives of Diazonaphthol}
\textbf{Meronitro Diazonaphthol}, O\textsubscript{2}N(N\textsubscript{2}).C\textsubscript{10}H\textsubscript{4}.OH; mw 215.16, N 19.53%. Several isomers are known:
3-Nitro-2-diazo-1-naphthol, crys (from benz), mp 187° (dec); was prepd by diazotization of 1-chloro-3-Nitro-2-naphthylamine and pouring the diazonium soln inaq Na acetate (Ref 9)
6-Nitr02-diazo-1-naphthol [called 6-Nitro-2-
diazonaphthalen-1(2)-diazid(1) in Ger), golden-yel prts or ndls from acet., mp - explodes at 142-45°; sol in chl, acet & acetic acid; sl sol in alc, benz & petr eth; insol in w; was prep 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-diazonaphthalen-1(2)-diazid(1) in Ger], It-om prisms from acet., mp - explodes violently at 155-60°; was prep 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); prep 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 acetic acid & benz; mod sol in alc; sl sol in w; was prep by diazotizing 2,4-dinitro-1-naphthylamine (Refs 1, 4 & 6).

7-Nitro-1-diazo-2-naphthol, crys from dioxane, mp 160-62° (dec); was obt when the 2,7-dinitro-1-naphthylamine diazotized salt was poured into water contg Na acetate (Ref 8).


**NOTE:** Higher nitrat derivs of Diazonaphthols were not found in Beil or in CA thru 1961.

**Diazonaphtholphydroxylic Acid, N₂(HO)-C₁₀H₄-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 prts from MeOH, mp - deflag rates at 151°; obt together with 1,2-naphthoquinone-4-carboxylic acid by diazotizing 4-amino-3-hydroxy-1-naphthoic acid with NaNO₂ & CuSO₄ in water (Ref 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); prep by diazotizing 4-amino-3-hydroxy-2-naphthoic acid with NaNO₂ & CuSO₄ in w or with NaNO₂ & HCl (Refs 1 & 2).
Diazonium Derivatives of Benzaminobenzene. See Benzaminobenzene-diazonium Derivatives in Vol 2, p B41-L

Diazonium Derivatives of Benzaldehyde. 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, 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 prep (Ref 1), Johnson & Oswald prepd 3-Pyridyldiazonium fluoroborate, C₅H₄N₂BF₄, and spread the salt on Ag 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 decomp point on a small sample of any new diazonium fluoroborate before allowing the bulk of the material to dry. They suggest that if a decomp point is above 100°, the salt can be safely dried and stored, however, if the decomp point is between RT & 100°, the salt may be dangerous. Any diazonium fluoroborates which show signs of decomp at or below RT should never be allowed to dry completely.

2) Anon, C & EN 45, No 44, p 44 (16 Oct 1967)

Diazonium Compounds. See Diazocompounds

Diazonium Compounds & Sodium Sulfide. Diazonium compds., such as diazotized 4-chloro-o-toluidine treated with Na₂S, NaHS or Na polysulfide solns, form explosive expls (Ref 1). Hodgson (Ref 2) reported that the addn of o-nitrophenyldiazonium chloride to Na₂S₂ soln at 0° gives a red ppt which explodes when touched with a glass rod. Similarly, when diazotized m-chloroaniline is added dropwise to Na₂S₂ soln, every drop reacts violently.


Diazonium Derivatives of Anilinobenzene. See Anilinobenzene-diazonium Hydroxide and Derivatives in Vol 1, p A421

Diazonium Derivatives of Azobenzene. See Azobenzene-diazonium Derivatives in Vol 1, p A650

Diazonium Chloride of Aminodicarboxybinphenyl. See Aminodicarboxybinphenyl-diazonium Chloride in Vol 1, p A195-L

Diazonium Compounds. See Diazocompounds

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 Derivatives of Benzaminobenzene. See Benzaminobenzene-diazonium Derivatives in Vol 2, p B41-L

Diazonium Derivatives of Benzaldehyde. See Vol 2, p B41-L

Diazonium Derivatives of Benzoquinone. See Vol 2, p B81-L

5-Diazo-4-iso-nitrosomethyl-uracil, Nitro Derivative. See under Diazouracil and Derivatives

Diazonium Chloride of Aminodicarboxybinphenyl. See Aminodicarboxybinphenyl-diazonium 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 Na₂S, NaHS or Na polysulfide solns, form explosive expls (Ref 1). Hodgson (Ref 2) reported that the addn of o-nitrophenyldiazonium chloride to Na₂S₂ soln at 0° gives a red ppt which explodes when touched with a glass rod. Similarly, when diazotized m-chloroaniline is added dropwise to Na₂S₂ soln, every drop reacts violently.


Diazonium Derivatives of Anilinobenzene. See Anilinobenzene-diazonium Hydroxide and Derivatives in Vol 1, p A421

Diazonium Derivatives of Azobenzene. See Azobenzene-diazonium Derivatives in Vol 1, p A650
Diazonium Salts. These are substances having the general formula $\text{RN}^+\cdot\text{A}$ where $\text{R}$ is a radical such as $\text{C}_6\text{H}_5^-$, $\text{C}_6\text{H}_5\text{O}^-$, and $\text{A}$ is an acid radical such as $\text{Cl}^-$, $\text{ClO}_4^-$, $\text{NO}_3^-$. 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 $\text{N}$ as in $\text{Ar.N.}^+(\text{Br,Cl})\rightleftharpoons\text{Ar.N.$^+$}$

Thus, the colored expl diazohalids 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 exps suitable as primer compds in detonators. He suggested also that the perchloric acid diazo compds of mono- & polynitroated 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 Diazocompounds and Diazotization.

Rejs: 1) A. Hantzsch, Ber 33, 2179-88 (1900) & JCS 78 I, 568 (1900)
2) E. Herz, GerP 258679 (1911) & CA 7, 2687 (1913);
3) BritP 27198 (1912) & CA 8, 1672 (1914)
4) R.P. Johnson & J.P. Oswald, C & EN 45, No 44, p 44 (16 Oct 1967)

2-Diazo-3-oxidobenzoic Acid, 4-Nitro Derivative. (called 6-Diazo-4-nitro-5-oxo-1,3-Cyclohexadiene-1-carboxylic Acid in CA)

$\text{CH}=\text{C(OOH)}\rightleftharpoons\text{C:N:N}

\text{CH}=\text{C(NO}_2\rightleftharpoons\text{C:O} ; \text{mw } 209.12, \text{ N}

20,10%; omn-colored powd + H$_2$O, stable in darkness, turns brn-red in light, mp - loses H$_2$O at 80$^\circ$ & deフラgrates at 176$^\circ$; decomp in boiling w; sol in alc & eth; sl sol in cold w; insol in benz; was prep by adding HNO$_3$ to 4-nitroanthranilic acid in conc H$_2$SO$_4$ below -5$^\circ$, pouring the ppt on ice and extracting with eth (Ref 2)

Rejs: 1) Beil-not found 2) G. Berti et al, Gazz 85, 1634, 1641-42 (1955) & CA 50, 10041 (1956)
Diazophenol and Derivatives

Diazophenol, O.C₆H₄.N:N; mw 120.11, N 23.33%.
2-Diazophenol. See Benzenediazo-oxide, Vol 2, p B58-L.
4-Diazophenol. See Benzenediazo-oxide, 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 Mononitrobenzenediazo-oxide, Vol 2, p B58-R
2-Nitro-4-diazophenol. See 2-Nitrobenzene-4-diazo-1-oxide under Mononitrobenzenediazo-oxide, Vol 2, p B58-R
3-Nitro-4-diazophenol. See 3-Nitrobenzene-4-diazo-1-oxide under Mononitrobenzenediazo-oxide, Vol 2, p B58-R
Dinitroderivs.
4,6-Dinitro-2-diazophenol. See Dinitrobenzenediazo-oxide or Dianzodinitrophenol, 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],
O₂N.C₆H₂ON₂,N(N)Cl, mw 225.58, N 31.05%; obtd from 4-nitro-2,6-diaminophenol by using an excess of HNO₂ & HCl. Its Chloroplatinate salt, 2C₆H₃N₂O₃Cl + PrCl₄, yel plts, is expl
Refs: 1) Beil 16, 532 2) P. Griess, Ber 19, 318 (1886) & JCS 50 1, 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 KNO₃ forms the Potassium salt of 2,4-dinitro-6-(tetrazeno)phenol, dk-red crysts. Both salts decom with expl violence on heating to 200°. They are claimed to be useful in prim compns (Ref)
Ref: J.F. Kenney, USP 2728760 (Dec 1955) & CA 50, 7462-63(1956)

5( or 3)-Diazao-3( or 5)-phenyl-1,2,4-triazole
[called 5( or 3)-Nitroaminino-3( or 5)-phenyl-1,2,4-triazolin or 5( or 3)-Diazao-3( or 5)-phenyl-1,2,4-triazol in Ger],
C₆H₅.C := N -- NH C₆H₅.C -- NH -- NH
|             | C:N:NO                     | N --------- C:N:NO
or

C₆H₅.C := N -- NH
|             | C:N:OH                     |
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 prep'd by diazotizing 5-amino-3-phenyl-1,2,4-triazole with NaNO₂ & HCl in the cold (Refs 1 & 2)
Ref s: 1) Beil 26, (46) 2) W. Manchot, Ber 43, 1313(1910) & CA 4, 2479(1910)

Diazopropane, C₃H₅N₂; mw 70.09, N 39.97%.
Two isomers are known:
1-Diazopropane (called 1-Diazo-propan in Ger),
CH₃,C₂H₂,CH₂:N:N, liq, bp -8.0 to -7.5° at 41.5mm of press; its ether soln is deep yellow in color (Ref 1)
2-Diazopropane (called 2-Diazo-propan or Dimethylidiazomethan in Ger), (CH₃)₂-C:N:N,
mobilie red liq of unpleasant, overpowering odor, bp -31.2° at 14mm press; dec at RT with evolution of heat & loss of N; may def&lgr suddenly; reacts violently with acids (Refs 2 & 3)
Other props & methods of prep are
given in Beil.
Refs: 1) Beil 1 (334) & [2691]  2) Beil 1
(344) & [2745]  3) H. Staudinger & A. Gaulke,
Ber 49, 1905 (1916) & CA 11, 596 (1917)

Diazopropanone or Acetyldiazomethane. See
Diazonacetone

Diazopropene, Diazopropylene or Vinylc大妈-
methane. (called Diazopropen, Vinyl-diazo-
methan or Diazopropyl en in Ger),
\[
\text{CH}_2\text{CH}:\text{CH}=\text{NN} \text{; mw} 68.08, \text{N} 41.15% \text{; not}
\]
isolated in the free state, gives a wine-red
ech soln; isomerizes to pyrazole; can be
prep 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. Nird-
linger & S.F. Acrec, 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_9N_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)

DIAZOPYROCA TEOHL AND DERIVATIVES

Diazopyrocatehol or Diazo-o-dihydroxyben-
zene, N_2C_6H_5(OH)_2, may be considered the
parent cmpd of the following derivs, al-
though it was not found in the literature and
was not used to prep them:

6-Nitro-4-diazopyrocatehol [called 6-Nitro-
4-diazobenzenechin or 6-Nitro-2-oxo-benzo-
chinon-(1,4)-diazid-(4) in Ger],
\[
\begin{align*}
\text{O}_2\text{N} & \equiv \text{C} \equiv \text{O} \equiv \text{C} \equiv \text{N} \equiv \text{CH} \\
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} & \equiv \text{or}
\end{align*}
\]

5-Nitro-3-diazopyrocatehol-1-methyl ether
[ called 5-Nitro-3-diazo-benzenechin-1-
methyläther or 5-Nitro-3-methoxy-o-chinon-
diazid-(1) in Ger; and called 6-Methoxy-4-
nitro-o-guinoinediazide in CA Collective
Formula Index 14-40]
\[
\begin{align*}
\text{CH} & \equiv \text{C} \equiv \text{O} \equiv \text{CH} \equiv \text{C} \equiv \text{O} \\
\text{O}_2\text{N} & \equiv \text{C} \equiv \text{O} \equiv \text{CH} \equiv \text{C} \equiv \text{O} \\
\end{align*}
\]

3-Diazo-4(3H)-quinolone or Quinolone-3,4-
dione-3-diazide,
\[
\begin{align*}
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} \\
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} & \equiv \text{or}
\end{align*}
\]

\[
\begin{align*}
\text{O}_2\text{N} & \equiv \text{C} \equiv \text{O} \equiv \text{CH} \\
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} & \equiv \text{or}
\end{align*}
\]

\[
\begin{align*}
\text{O}_2\text{N} & \equiv \text{C} \equiv \text{O} \equiv \text{CH} \\
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} & \equiv \text{or}
\end{align*}
\]

\[
\begin{align*}
\text{O}_2\text{N} & \equiv \text{C} \equiv \text{O} \equiv \text{CH} \\
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} & \equiv \text{or}
\end{align*}
\]

\[
\begin{align*}
\text{O}_2\text{N} & \equiv \text{C} \equiv \text{O} \equiv \text{CH} \\
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} & \equiv \text{or}
\end{align*}
\]

\[
\begin{align*}
\text{O}_2\text{N} & \equiv \text{C} \equiv \text{O} \equiv \text{CH} \\
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} & \equiv \text{or}
\end{align*}
\]

\[
\begin{align*}
\text{O}_2\text{N} & \equiv \text{C} \equiv \text{O} \equiv \text{CH} \\
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} & \equiv \text{or}
\end{align*}
\]

\[
\begin{align*}
\text{O}_2\text{N} & \equiv \text{C} \equiv \text{O} \equiv \text{CH} \\
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} & \equiv \text{or}
\end{align*}
\]

\[
\begin{align*}
\text{O}_2\text{N} & \equiv \text{C} \equiv \text{O} \equiv \text{CH} \\
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} & \equiv \text{or}
\end{align*}
\]

\[
\begin{align*}
\text{O}_2\text{N} & \equiv \text{C} \equiv \text{O} \equiv \text{CH} \\
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} & \equiv \text{or}
\end{align*}
\]

\[
\begin{align*}
\text{O}_2\text{N} & \equiv \text{C} \equiv \text{O} \equiv \text{CH} \\
\text{CH} & \equiv \text{C} \equiv \text{N} \equiv \text{CH} & \equiv \text{or}
\end{align*}
\]
and many of its unstable derivs, prepd by Süss & Glos (Ref 3), were patented as sensitizing agents on lithographic printing plates


**DIAZORESORCINOL AND DERIVATIVES**

Diazoresorcinol or Diazo-m-dihydroxybenzene, CH—C(=O) — CH CH—C(=O) = CH

\[
\text{CH} = \text{C}(\text{NO}) — \text{C}.\text{O} \quad \text{or} \quad \text{CH} = \text{C}(\text{NO}) — \text{C}.\text{OH} ;
\]

mw 165.11, N 24.45%; gray-gren ndls (from acet), mp - begins to decom 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-tetraminoresorcinol-HCl (Refs 1 & 2)

Refs: 1) Beil 16, 541 2) H. Kauffmann & E. de Pay, Ber 39, 324 (1906) & JCS 90 I, 158 (1906)

4,6-Dinitro-2-diazoresorcinol [called 4,6-Dinitro-2-diazoresorcin or 4,6-Dinitro-3-oxy-chinon-diazid(2) in Ger], O₂N.C — C(=O) — C.N₂

\[
\text{CH} = \text{C}(\text{NO}) — \text{C}.\text{OH} ;
\]

mw 226.11, N 24.78%; yel crystls; obtd from its K salt by treating with concd H₂SO₄ Potassium salt, KC₆H₇N₄O₆, yel ndls (from very concd aq soln)

**Potassium salt monohydrate, KC₆H₇N₄O₆·H₂O, bms 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-diazeresorcin; 3,5-Dinitro-2-oxy-p-chinon-diazid(1); or 3,5-Dinitro-4-oxy-o-chinon-diazid(1) in Ger],

\[
\text{O₂N.C — C(=O) — C.NO} \quad \text{or}
\]

O₂N.C — C(=O) — C.NO₂

\[
\text{CH} = \text{C}(\text{NO}) — \text{C}.\text{OH} ;
\]

CH — C(=O) — C.O

(mw 226.11, N 24.78%; golden-yel prts, 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)


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], C₇H₇N₃O₄Br; mw 274.04, N 15.33%; onyl crystls, mp 189°, explodes when heated above its mp; was obtd from 4-nitro-3,5-dibromo-anisole-diazonium-2-hydroxide by treating with H₂SO₄ or acetic acid soln

Ref: Beil 16, 536 & (365)

4-Nitro-5-bromo-2-diazoresorcinol-1-methyl ether, C₇H₆N₄O₃Br; mw 274.04, N 15.33%; onyl crystls, mp 189°, explodes when heated above its mp; was obtd from 4-nitro-3,5-dibromo-anisole-diazonium-2-hydroxide by treating with H₂SO₄ or acetic acid soln

Ref: Beil 16, (365)

2,5-Dinitro-4-diazoresorcinol-1-methyl ether, C₇H₆N₄O₆; mw 240.13, N 23.33%; orn prisms (from acet), mp 197-98°, defigr 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, C₇H₆N₄O₆, mw 240.13, N 23.33%; yel prisms
(from acet ac), mp 191-94\(^\circ\) (dec); was prep by diazotizing 2,3,6-trinitro-4-aminoanisole
Ref: Beil 16, (366)

**DIAZOSALICYLIC ACID
AND DERIVATIVES**

3-Diazo-salicylic Acid [called 3-Diazo-salicyl-
äsäre or o-Chinon-diazid-(1)-carbonsäure-
(3) in Ger],

\[
\begin{align*}
\text{CH} & \equiv (\text{COOH}) \equiv \text{C}_3\text{O} \\
\text{CH} & \equiv \text{C} \equiv \text{C}_3\text{N} \\
\text{N}
\end{align*}
\]

or

\[
\begin{align*}
\text{CH} & \equiv (\text{COOH}) \equiv \text{C}_3\text{O} \\
\text{CH} & \equiv \text{C} \equiv \text{C}_3\text{N} \\
\text{N}
\end{align*}
\]

\[
\begin{align*}
\text{CH} & \equiv \text{C} \equiv \text{C}_3\text{N} \equiv \text{N} : \text{mw} 164.12, \text{N} \\
17.07%; \text{yel ndls (from acet), mp - dec at} \text{155}^\circ; \text{was prep by diazotizing 3-aminosalicylic acid in glacial acetic acid}
\end{align*}
\]

Ref's: 1) Beil 16, 553 2) O. Zahn, JPrChem 61, [2], 553 (1900) & JCS 78 1, 549 (1900)

3-Diazo-5-nitro-salicylic Acid [called 5-Nitro-
3-diazo-salicylsäure or 5-Nitro-benzozquinon-
(1,2)-diazid-(1)-carbonsäure-(3) in Ger],

\[
\begin{align*}
\text{O}_2\text{N}\text{C}_6\text{H}_5\text{N}_2\text{O}(\text{COOH}) : \text{mw} 209.12, \text{N} 20.10%; \text{yel ndls (from alc), mp - becomes bm at} 100^\circ \\
& \text{& dec at} 145-50^\circ, \text{explodes violently on rapid} \\
& \text{heating; was prep by diazotizing 5-nitro-3-}
\text{aminosalicylic acid}
\end{align*}
\]

Ref's: 1) Beil 16, (368) 2) R. Meldola et al, JCS 111, 541 (1917)

5-Diazo-salicylic Acid. See 5-Diazo-2-hydroxy-
benzoic Acid

3-Chloro-5-diazo-salicylic Acid [called 3-Chloro-
5-diazo-salicylsäure or 6-Chlor-benzozquinon-
(1,4)-diazid-(4)-carbonsäure-(2) in Ger],

\[
\begin{align*}
\text{CH} & \equiv (\text{COOH}) \equiv \text{C}_3\text{O} \\
\text{C} & \equiv \text{C} \equiv \text{C}_3\text{Cl} \text{ or } \text{C}_3\text{N}_2 \equiv \text{C} \equiv \text{C}_3\text{Cl}
\end{align*}
\]

\[
\begin{align*}
\text{N} & \equiv \text{N} \equiv \text{N} : \text{mw} 166.57, \text{N} 16.82%; \text{ochre-yel scales (from} \\
& \text{acet ac), mp - detonates at} 193^\circ, \text{almost insol} \\
& \text{in} \text{w} \& \text{alc; was prep by diazotizing 3-chloro-
5-aminosalicylic acid}
\end{align*}
\]

Ref's: 1) Beil 16, (369) 2) R. Meldola et al, JCS 111, 543 (1917)

Diazostilbene, C\text{C}_6\text{H}_5\text{CH}:\text{CH}:\text{C}_6\text{H}_4\text{N}_2 \text{; known} \\
in the form of the salts of stilbene diazonium

hydroxide, C\text{C}_6\text{H}_5\text{CH}:\text{CH}:\text{C}_6\text{H}_4\text{N}(\text{N})\text{OH}. \text{The} \\
following salts of Nitrostilbene diazonium \\
hydroxide, C\text{C}_6\text{H}_5\text{CH}:\text{CH}:\text{C}_6\text{H}_3(\text{NO}_2)\text{N}(\text{N})\text{OH}, \\
are either expl or unstable on heating:

4-Nitrostilbene-2-diazoniumhydroxide (Refs 1 & 2):
Chloride, O\text{C}_2\text{N}:\text{C}_1\text{H}_1\text{O}_2\text{N}_2\text{Cl}, \text{dk-yel ndls, mp -} \\
decl at 107^\circ.

Sulfate, O\text{C}_2\text{N}:\text{C}_1\text{H}_1\text{O}_2\text{N}_2\text{O}_3\text{S} \text{H}, \text{lt-yel ppt, } \text{mp} \\
& \text{dec} 135-57^\circ.

2-Nitrostilbene-4-diazoniumhydroxide (Refs 1 & 3):
Chloride, O\text{C}_2\text{N}:\text{C}_1\text{H}_1\text{O}_2\text{N}_2\text{Cl}, \text{dk-violet plts, } \\
& \text{mp - expl 105-110^\circ}

Nitrate, O\text{C}_2\text{N}:\text{C}_1\text{H}_1\text{O}_2\text{N}_2\text{O}_2, \text{lt-bm ndls, } \\
& \text{mp - explodes violently on heating}

Sulfate, O\text{C}_2\text{N}:\text{C}_1\text{H}_1\text{O}_2\text{N}_2\text{O}_3\text{S} \text{H}, \text{lt-bm ndls, } \\
& \text{mp - darkens at 108^\circ and decomp explosively}
\text{at 165^\circ}

The salts are formed by diazotizing in
alcoholic-acid soln the appropriate nitroaminostilbene with amyl nitrite

Ref's: 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-Thiodiazole (called 1,2,3-
Thiodiazol or "Diazosulfid" in Ger),

\[
\begin{align*}
\text{CH} & \equiv \text{S} \equiv \text{N} \\
\text{CH} & \equiv \text{N} : \text{mw} 86.13, \text{N} 32.53%; \text{clear liq,} \\
& \text{bp 83-85^\circ at 66mm press, bp 151^\circ at 742mm,} \\
& \text{volatile with steam, d 1.3292 at 0^\circ, mod sol} \\
& \text{in alc \& eth; sol in 3 parts w at 10^\circ;} \\
& \text{dec on heating in NaOH; was prep by heating} \\
& \text{1,2,3-thiodiazole-4-carboxylic acid to 230-60^\circ}
\end{align*}
\]

It forms salts, such as:

Chloraurate, C\text{H}_2\text{N}_2\text{S} + \text{AuCl}_3, \text{pale-yel ndls} \\
(from alc), mp - darkens at 152^\circ and decomp at \\
165^\circ \text{w/o melting}

Hydrochloride, C\text{H}_2\text{N}_2\text{S} + \text{HCl, hygr ndls} \\
(from eth \& HCl), or prisms (by sublimation), \\
mp 70^\circ

Hydrochloride Chloraurate, C\text{H}_2\text{N}_2\text{S} + \text{HCl} + \\
\text{AuCl}_3, \text{yel ndls, mp - dec 150-60^\circ}

Ref's: 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],
\[
\text{HO.N}_2.C=\text{NH}-\text{N} \quad \text{or} \quad \text{HO.N}_2.C=\text{N}=\text{NH}
\]
\[
\text{N} \quad \text{or} \quad \text{N} = \text{N}
\]

Formula I  
Formula II

\[
\text{N} \quad \text{C} = \text{N} \quad \text{or}
\]
\[
\text{N} \quad \text{N} = \text{N}
\]

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\(^\circ\); 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\(_2\)CON\(_6\), 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: 
\[
\text{Na}_2\text{C}_2\text{N}_10\text{O} + 5\text{H}_2\text{O}, \text{yel plts, explodes with great violence. The corresponding Barium salt, BaC}_2\text{N}_10\text{O} + 4\text{H}_2\text{O}, \text{yel ndls (from w), is also highly expl}
\]
(Refs 1 & 3)


Diazotetrazole-aminoguanidin or [Guanyl (tetrazolyl-5)] - I, IV-tetrazene (called I-Guanyl-4-{tetrazolinyliden-5)}-tetrazene or 1-Guanyl-4-{tetrazolyl45}-tetrazane in Ger),
\[
\text{H}_2\text{N.C} (\text{CNH}) \text{.NH.N=N.C} = \text{N} \quad \text{or}
\]
\[
\text{HN} = \text{N} = \text{N}
\]

\[
\text{H}_2\text{N.C} (\text{CNH}) \text{.NH.N=N.C} = \text{N} \quad \text{or}
\]
\[
\text{HN} = \text{N} = \text{N}
\]

mw 170.15, N 82.33%; yel ndls + H\(_2\)O, mp 142\(^\circ\) (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\(_2\)H\(_6\)N\(_{10}\) + HI + I, blk to bm pleochroic, cubic crysts; explodes gentle when heated, but violently in contact with HNO\(_3\) or AgNO\(_3\)

Silver salt, yel crysts, explosive

Ref.: 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\(_2\)S\(_3\) 30 & Diazotetrazoler-aminoguanidine 15%)
Diazotetrazole-phenylhydrazine. See N\textsuperscript{1}-Amino-N\textsuperscript{2}-phenyl-N\textsuperscript{3}-(tetrazoly1-5)-triazene in Vol 1, p A247-R

Diazotetrazole-semicarbazide or (Tetrazolyl-5)-carbomamide-1, IV-tetrazene I called 4-[(Tetrazolyliden-5)-tetrazen-carbonsäure-(1)-amid or [Tetrazolyl-(5)-tetrazen-carbon-

\[
\begin{array}{c}
\text{H}_2\text{N}.\text{CO}.\text{NH}.\text{NH}.\text{N} : \text{N} : \text{C} \quad \text{HN} \\
\quad \text{HN} \quad \text{N} = \text{N} \quad \text{or}
\end{array}
\]

\[
\begin{array}{c}
\text{H}_2\text{N}.\text{CO}.\text{NH}.\text{NH}.\text{N} : \text{N} : \text{C} \quad \text{HN} \\
\quad \text{HN} \quad \text{N} = \text{N}
\end{array}
\]

mw 171.13, N 73.67%; crystals + H\textsubscript{2}O, mp 122\degree, deflgr when heated at higher temps; diff sol in w, giving an acid soln; was prep'd from 5-Diazotetrazole & semicarbazide or acetone-semicarbazone (Ref 1 & 2)

Ref:s: 1) Beil 26, (123) 2) K.A. Hofmann & H. Hock, Ber 44, 2950-51 (1911) & JCS 100 1, 1048(1911)

Diazotization. This is a reaction by which a diazo compd is obtd from a primary amine.
The reaction is as follows:

R-NH\textsubscript{2} + 2HX + NaNO\textsubscript{2} \rightarrow R-N\textsubscript{2}X + NaX + 2H\textsubscript{2}O

where X = Cl\textsuperscript{-}, Br\textsuperscript{-}, NO\textsubscript{3}\textsuperscript{-}, HSO\textsubscript{4}\textsuperscript{-} & BF\textsubscript{4}\textsuperscript{-}

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\textsubscript{2}SO\textsubscript{4}

R-N\textsubscript{2}X + H\textsubscript{2}O \rightarrow R-\text{OH} + HX + N\textsubscript{2}

b) the diazonium group is replaced with halogen when the double salt formed with cuprous halides is heated

R-N\textsubscript{2}X + Cu\textsubscript{2}Cl\textsubscript{2} + R-X + Cu\textsubscript{2}Cl\textsubscript{2} + N\textsubscript{2}

c) a nitrile is formed when a soln of the diazonium salt is heated with KCN, CuSO\textsubscript{4} or Cu powder

R-N\textsubscript{2}X + KCN = R-CN + KX + N\textsubscript{2}

d) the diazonium group is replaced with H by treating with aliphatic alcohols in alkaline soln

R-N\textsubscript{2}X + C\textsubscript{2}H\textsubscript{5}OH = R-H + CH\textsubscript{3}CHO + HX + N\textsubscript{2}

e) ethers are sometimes formed when a diazo-

nium salt is heated with alcohol in neutral soln

R-N\textsubscript{2}X + CH\textsubscript{3}OH = R.O.CH\textsubscript{3} + HX + N\textsubscript{2}

f) by reduction of a diazonium salt with K bisulfite, hydrazines are obtb

R-N\textsubscript{2}X + 2KHSO\textsubscript{3} + 2H\textsubscript{2}O = R.NH.NH\textsubscript{2}.HX + 2KHSO\textsubscript{4}

Diazotization is an important reaction because of the practical uses of diazo compds. One use of diazo compds is in the prep of light-sensitive printing papers, since most diazo compds are stable in the dark but decom 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-N\textsubscript{2}X + HX \rightarrow R-N=\text{N}-R + HX

where R represents an aryl radical and R represents an alkyl or aryl radical whose conjugate acid HR\textsuperscript{+} is capable of coupling

See also Diazocompounds and Diazonium Salts

Ref:s: 1) W. Fuchs, Rec 42, 511-12(1923) & CA 17, 3489 (1923)(The Witt method of diazotization) 2) Groggins (1952) 135-75 (Chapter 3, "Dizotization and Coupling" by H.E. Woodward; not found in later editions) 3) Kirk & Orthmer, 2nd ed, 2(1963), 868-70 (Diazotization)

**DIAZOTOLUENE AND DERIVATIVES**

**Diazotoluene, Diazomethylbenzene or Toluenediazonium Hydroxide** (called Diazotolul; Diazomethyl-benzol or Toluoldiazoniumhydroxyd in Ger),

\[\text{CH}_3\text{C}_6\text{H}_4.N(\text{N}),\text{OH}; \text{mw 136.15, N 20.58%}\]

The following isomers are known only in the form of their salts:

- (or 2) Diazotoluene Salts (Ref 1):
  - Chloride, CH\textsubscript{3}.C\textsubscript{6}H\textsubscript{4}.N\textsubscript{2}.Cl; wh crys, hygr, mp deflgr on heating; sol in acet, chl & AcOH; was prep'd by diazotizing o-toluidinehydrochloride
  - Lead Tetrachloride, 2CH\textsubscript{3}.C\textsubscript{6}H\textsubscript{4}.N\textsubscript{2}.Cl + PbCl\textsubscript{4}; yel crys, mp - explode at 78-80\degree; stable if kept dry and in absence of air; was prep'd by treating the chloride salt with lead tetrachloride
Nitrates, CH₃C₂H₄N₂O₂ . NO₃; white needles (from alc + eth); mp - highly expl in dry state; v sol in alc & MeOH; sol in acet ac; insol in chl & benz; was prepd by diazotizing o-toluidine with amyl nitrate in the presence of HNO₃

Perchlorate, CH₃C₂H₄N₂ClO₄·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 NaNO₂ in the presence of perchloric acid

Sulfate, CH₃C₂H₄N₂O₂ . SO₃H·ndls; white needles, hygroscopic, mp - decom on heating & explodes mildly on impact; readily sol in acet ac; sol in MeOH; v sl sol in alc; insol in eth, chl, CS₂, petr ether, acet, benz & nitrobenz; was prepd by passing N oxides thru a soln of o-toluidine in alc to which H₂SO₄ was added drop by dropwise

m-(or 3-) Diazotitrotoluene Salts (Ref 2):

Borofluoride, CH₃C₂H₄N₂BF₄·crysts, dec at 108°

Chloride + Bismuth Trichloride, CH₃C₂H₄N₂Cl + BiCl₃; yel ppts, mp 120°(dec);
diff sol in dil HCl

Nitrates, CH₃C₂H₄N₂O . NO₂; white needles, very expl

Sulfate, CH₃C₂H₄N₂O₂ . SO₃H·ndls; white needles, becomes red on exposure to light, very unstable

p-(or 4-) Diazotitrotoluene Salts (Ref 3):

Lead Tetrachloride, 2CH₃C₂H₄N₂Cl + PbCl₄·yel crysts, explodes at 72-75°, insol in w & org solvs

Nitrates, CH₃C₂H₄N₂O . NO₂·ndls, expl; sol in MeOH, alc, acet ac & acet et; v sl sol in chl

Perchlorate, CH₃C₂H₄N₂ClO₄·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, chl, CS₂, acet, benz & nitrobenz. This compd was prepd by von Herz as a chge for use in detonating caps (Ref 3a)

Tetrachloroiodide, CH₃C₂H₄N₂ICl₄·yel crysts (from iodotrichloride in concd HCl), mp 95°(dec)

See also the following Refs for addnl info on Diazotoluenes

Ref s: 1) Beil 16, 495, (358) & (278)
2) Beil 16, 500 & (279)
3) Beil 16, 501, (359) & (280)
4) E. von Herz, USP 1054411 (1913) & CA 7, 1419 (1913)
5) H.R. Lee, JIndEngChem 13, 104950 (1921) & CA 16, 38 (1922) (Decomp of p-diazotoluenyl chloride in acid soln and its use in coupling reactions)
6) A. Algerini, Gazz 62, 145-50 (1932) & CA 27, 2942 (1933) (Diazonium dichloriodofluorides of Diazotoluene)
7) D. Bigiavi & C. Albanese, Gazz 65, 773-88 (1935) & CA 30, 2934-35 (1936) (Reduction of Diazotoluenes in relation to azoxy derivs)
9) E.S. Lewis, ChemBer 91, 2350-58 (1958) & CA 53, 6123 (1959) (Spectroscopic studies)
10) Mononitrodiazotoluenes, CH₃C₂H₃(NO₂) . N₂OH, mw 181.15, N 23.20%; known in the form of their salts:

5-Nitro-2-diazotoluenyl Salts (Ref 1):

Nitrates, CH₃C₂H₃(NO₂) . N₂O . NO₂·straw-yellow needles, dec at 97°; was prepd by diazotizing 5-nitro-2-aminotoluene with amyl nitrite in abs alc in the cold

5-Nitro-2-Isodiazotoluenyl and Its Salts (Refs 1 & 3): yellowish crystals, dec at 40°

Potassium, KC₇H₆N₃O₃ + H₂O; yel needles (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 expl of the material in a desiccator

Sodium, NaC₇H₆N₃O₃·yel crystals (from MeOH), very expl

3-Nitro-4-Isodiazotoluenyl (Ref 2): Lt-yel needles, mp - dec on standing & turning btn

Sodium salt, NaC₇H₆N₃O₃·Lt-yel needles (from alc + eth), deflgr on heating above its mp; v sl sol in alc & w

Ref s: 1) Beil 16, 499, (359) & (279)
2) Beil 16, 506 & (360)
3) J. Meisenheimer & E. Hesse, Ber 58, 1175-77 (1925); 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)
Dinitrodiazotoluene, CH₃C₆H₂(NO₂)₂N₂OH; 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 NaNO₂ & coned
H₂SO₄, or by adding K₂SO₅ & fuming HNO₃
to the amine at -5 to 0° (Ref 1 & 3)
3,5-Dinitro-2-diazotribromotoluene,
CH₃C₆H₂(NO₂)₂Br₂; yel-red crys., mp-
deflgd 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] B. 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 Diazotolosulfo-saure in Ger.], CH₃C₆H₅(N₂O₂)SO₂;
mw 198.21, N 14.14%. The following isomers are known:
4-Diazo-toluene-2-sulfonic Acid, yel or bm
ndls, mp - darkens at 126° & dec at 135°;
explodes when heated on a Pt block or by
impact; insol in alc; was prepd by intro-
ducing nitrous gas into an alc suspension
of 4-aminotoluene-2-sulfonic acid (Ref, p 566)
5-Diazo-toluene-3-sulfonic Acid, listed in Ref,
p 567, but no props are given
4-Diazo-toluene-3-sulfonic Acid, plts, mp -
not reported; sol in w ar 60° w/o decmp;
difficult sol in cold w; prepd by reaction of 4-
aminotoluene-3-sulfonic acid in alc or w at
30-40° & HNO₂ (Ref, p 566)
6-Diazo-toluene-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-Diazo-toluene-4-sulfonic Acid, prisms, burns
brightly when heated on a Pt foil & explodes
by impact; insol in cold w; was prepd by
introducing nitrous gas in an aq alc soln
of 2-aminotoluene-4-sulfonic acid (Ref, p 569)

4-Diazo-toluene-1-sulfonic Acid,
CH₂SO₂
O
C₆H₄
N(N)
, prisms, mp - deflgd
when heated on a Pt foil; readily sol in w;
isol in alcohol; prepd by reaction of nitrous
gas & 4-aminotoluene-1-sulfonic acid in
water (Ref, 570)
Ref: Beil 16, 566-70
Mononitrodiazo-toluene-sulfonic Acid, CH₃-
C₆H₅(N₂O₂)(N₂O₃)SO₂; mw 243.21, N 17.28%.

6-Nitro-4-diazo-toluene-2-sulfonic Acid, dk-red
crys, mp - deflgd when heated; mod sol in
w; sl sol in alc; prepd by treating 4-amino-
toluene-2-sulfonic acid with cold fuming HNO₃
(Ref 1, p 567). Batick (Ref 2) found that
this compd was formed on acidification of the
waste waters from the purification of TNT with Na₂SO₃ 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-diazo-toluene-3-sulfonic Acid, ndls,
mp - explodes violently on heating or by
impact; was prepd from 4-aminotoluene-3-
sulfonic acid & fuming HNO₃ (Ref 1, p 568)

6-Nitro-4-diazo-toluene-3-sulfonic Acid, bm
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 KNO₂ & dil H₂SO₄
(Ref 1, p 568)

3-(or 5) Nitro-2-diazo-toluene-4-sulfonic Acid,
crys, mp - deflgd 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
HNO₃ (Ref 1, p 569)
Refs: 1) Beil 16, 567-69  2) B. Batick,
Chim & Ind, Special No, 960-63 (1933) &
CA 28, 645 (1934)
Dinitrodiazo-toluene-sulfonic Acid,
CH₃C₆H(NO₂)₂N₂O₂SO₂; mw 288.21, N
19.45%

eso-Dinitro-4-Diazo-toluene-2-sulfonic Acid,
Diazotoluenesulfonic Acid
Halogenated Derivatives

4-Bromo-3-(or 5, or 6)-diazotoluene-2-sulfonic Acid,

\[ \text{CH}_3\text{C}_6\text{H}_2\text{Br} \quad \text{SO}_2 \quad \text{O} \quad \text{N} \quad \text{N} \]

crysts, explodes on impact; obtbd 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,

\[ \text{C}_7\text{H}_5\text{N}_2\text{O}_3\text{BrS} \]

brn-red powd, expl on heating or on impact; sol in cold w with slow decomp; dec rapidly in hot w; prepd by action of nitrous gas on a concd qg or alc soln of 5-bromo-4-aminotoluene-3-sulfonic acid (Ref, p 568)

3,5-Dibromo-2-diazotoluene-4-sulfonic Acid,

\[ \text{C}_7\text{H}_4\text{N}_2\text{O}_2\text{Br}_2\text{S} \]

ndls, deflgd violently on heating; almost insol in w & alc; prepd by passing nitrous gas in an q suspension of 3,5-dibromo-2-aminotoluene-4-sulfonic acid (Ref, p 569)

Ref: Beil 16, 567-69; no later refs found

5-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-tetrahydroprymidin or 5-Diazo-2,4-dioxy-pyrimidin in Get).

\[ \text{CH} \quad \text{NH} \quad \text{C} \quad \text{O} \]

\[ \text{HO} \text{N} \text{N} \text{C} \quad \text{C} \quad \text{(O)} \quad \text{NH} \quad \text{or} \]

\[ \text{CH} \quad \text{N} \quad \text{C} \quad \text{O} \quad \text{H} \]

\[ \text{HO} \text{N} \text{N} \text{C} \quad \text{C} \quad \text{(OH)} \quad \text{N} \quad \text{; mw 156.10, N 35.89%; exists in two forms: ruby-red plts (from w), or yel crystals (from w), mp - explodcs when heated on a Pt foil. The red form is obtbd from 5-aminouracil & NaNO}_2 \text{ in dil HCl under cooling, or from 5-diazouracil ethyl ether by hydrolyzing with cold 1% KOH and carefully adding HCl to the K salt. The yel form is obtbd by heating 5-diazouracil ethyl ether with water or by long standing of 5-diazouracilcarboxylic acid at RT.} \]

The Potassium salt, KC\text{H}_3\text{N}_4\text{O}_3, yel-red ndls, explodcs 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 (1893)

5-Diazo-4-isonitosomethyl-uracil [called 5-Diazo-4-isonitosomethyl-uracil; 5-Diazo-2,6-dioxo-4-iminomethyl-tetrahydroprymidin or 5-Diazo-2,6-dioxopyrimidin-aldoxim-(4) in Get.],

\[ \text{C} \quad \text{(O)} \quad \text{NH} \quad \text{C} \quad \text{O} \]

\[ \text{HO} \text{N} \text{N} \text{C} = \text{C} \text{(CH)} \text{(N)} \text{O} \quad \text{NH} \quad \text{or} \]


Diazotrinitrophenol. See Trinitrobenzene-diazoxyde in Vol 2, p B60-L

5-Diazouracil; 5-Diazo-2,4-pyrimidinediol or 5-Diazo-4-hydroxy-2(1H)-pyrimidinone (called 5-Diazo-uracil; 5-Diazo-2,4-dioxo-tetrahydroprymidin or 5-Diazo-2,4-dioxy-pyrimidin in Get).
\[
\text{C(OH)} \rightarrow \text{N} \rightarrow \text{C(OH)}
\]

\[
\text{HO.N: N.C} \equiv \text{C(CH:N,OH)} - \text{N} \quad ; \text{mw 199.13, N 35.17%; ndls (from alc) or prisms + 1/2 H}_{2}\text{O (from w), mp - explodes on heating; readily sol in alkalies & concd H}_{2}\text{SO}_{4}; \text{sl sol in alc; insol in dil acids; was prep by treating 5-amino-4-methyluracil in acid soln with NaNO}_{2}, or from 5-bromo-4-methyluracil by heating with NH}_{3}\text{ in a tube at 150° and reacting the products with NaNO}_{2} \text{ in dil acid soln.}
\]

Its \text{Nitro deriv}, C_{5}H_{4}N_{6}O_{6}, ndls, mp - explodes at 100°; dec on heating in w; was obtd by dissolving 5-diazo-4-isonitrosomethyluracil in red fuming HNO}_{3}.

\text{Refs: Beil 25, 566 2) R. Behrend, Ann 245, 213-223(1888) & JCS 52 II, 809-10(1888)}

\text{5-Diazoauracil-4-carboxylic Acid [called 5-Diazoauracil carbonsäure(4); 5-Diazo-2,6-dioxo-tetrahydropyrimidin-carbonsäure(4) or 5-Diazo-2,6-dioxo-pyrimidin-carbonsäure(4) in Ger],}

\[
\text{C:O} \rightarrow \text{NH} \rightarrow \text{C:O}
\]

\[
\text{HO.N: N.C} \equiv \text{C(CO}_{2}\text{H)} - \text{NH} \quad \text{or}
\]

\[
\text{C(OH)} \equiv \text{N} \rightarrow \text{C(OH)}
\]

\[
\text{HO.N: N.C} \equiv \text{C(CO}_{2}\text{H)} - \text{N} \quad ; \text{mw 200.11, N 28.00%; unstable yel ndls, mp - turns bm at 70-80° & explodes at 130°; sl sol in w with an acid reaction; readily sol in alkalies; was prep by reaction of 5-aminoauracilcarboxylic acid with cold NaNO}_{2} \text{ & HCl. On standing for a long time at RT, the compd is converted into yel Diazauracil.}
\]

\text{Refs: 1) Beil 25, 556 2) R. Behrend & P. Emenr, Ann 258, 349(1890) & JCS 52 II, 1240(1890)}

\text{5-Diazauracil Ethyl Ether [called 5-Diazoauracil-äthyl-äther in Ger],}

\[
\text{C:O} \rightarrow \text{NH} \rightarrow \text{C:O}
\]

\[
\text{C}_{2}\text{H}_{5}.\text{O}:\text{N.C} \equiv \text{CH} \rightarrow \text{NH} \quad \text{or its tautometric form; mw 184.16, N 30.43%; yel-yl lfts (from alc), mp - explodes by heating on a Pt foil; dec on heating with alc at 80-100°; almost insol in cold alc, eth & cold w; is formed when 5-diazouracilcarboxylic acid is boiled with alc.}
\]

\text{Refs: Beil 25, 565 2) R. Behrend & P. Emenr, Ann 258, 354(1890) & JCS 52 II, 1240-41(1890)}

\text{Diazoxylene and Derivatives}

\text{Diazoxylene or Diazodimethylbenzen (called Diazo-xylol, Diazodimethyl-benzol or Xyloidiazonium-hydroxyd in Ger),}

\[
(\text{CH}_{3})_{2}.\text{C}_{6}\text{H}_{3}.\text{N}_{2}.\text{OH}; \text{mw 150.18, N 18.65%; Known in the form of salts of its isomers:}
\]

\text{4-Diazo-m-xylene Salts [Ref, pp 507, (360) & [281]]:}

\text{Borofluoride, C}_{8}\text{H}_{9}.\text{N}_{2}.\text{BF}_{4}; \text{dec at 108°}

\text{Dichloroiodide, C}_{8}\text{H}_{9}.\text{N}_{2}.\text{Cl}_{2}; \text{lt-yel, fine cryst powd, mp 79°(dec)}

\text{2-Diazo-m-xylene Salts [Ref, p [281]]: Chloride, C}_{8}\text{H}_{9}.\text{N}_{2}.\text{Cl; crysts (from alc + eth) &}

\text{2-Diazo-p-xylene Salts (Ref, p 507)}

\text{5-Nitro-4-isodiazao-m-xylene Salts [Ref, p [281]]: Potassium, (CH}_{3})_{2}.\text{C}_{6}\text{H}_{3}.\text{NO}_{3}.\text{N}_{2}.\text{K}; \text{lt-yel pts, defgr on heating rapidly; very sl sol in w}}

\text{3,5-Dinitro-2-diazo-p-xylene Salts [Ref, p (361)]:}

\text{Tribromide, (CH}_{3})_{2}.\text{C}_{6}\text{H(NO}_{2})_{2}.\text{N}_{2}.\text{Br}_{3}; \text{red crysts, dec on storage, especially in sunlight}

\text{3,5-Dichloro-2-diazo-p-xylene Salts (Ref, p 508):}

\text{Tribromide, C}_{6}\text{H}_{5}.\text{Cl}_{2}.\text{N}_{2}.\text{Br}_{3}; \text{lt-yel ndls (from MeOH), mp - dec at 155°}

\text{Chloroplunitate, 2C}_{6}\text{H}_{5}.\text{Cl}_{2}.\text{N}_{2}.\text{Cl + PCl}_{4}; \text{yel-bm pts, mp - dec at 159°}

\text{Ref: Beil 16, 507, 508, (360, 361) & [281]}

\text{NOTE: Higher nitrated derivs were not found in Beil or in CA thru 1961}

\text{Diazoxylenesulfonic Acid and Derivatives}

\text{Diazoxylenesulfonic Acid (called Diazoxyl-sulfonsäure in Ger),}

\[
(\text{CH}_{3})_{2}.\text{C}_{6}\text{H}_{3}.\text{N}_{2}.\text{O}.\text{SO}_{3}; \text{mw 212.16, N 13.21%; The following isomers are found in Beil:}
\]

\text{6-Diazo-m-xylene-4-sulfonic Acid, prisms (from alc) or flesh-colored pts, defgr on heating; mod sol in w; diff sol in abs alc}

\text{4-Diazo-m-xylene-5-sulfonic Acid, prisms}
Dibenzalazobisaminoformamide [called 1,4-Azobis (benzylideneaminoformamide) in CA 5th Decennial Index], 
H₅C₆.CH:NNH.C(NH)N- 
N(CH)NH:CH.C₆H₅; 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-diaminobiguandine was cooled slowly while exposed to air (Ref 2) 

Dibenzal-1,6-diaminobiguandine [called 1,6-Bis (benzylidene-amino)-biguanidine in CA 5th Decennial Index], 
H₅C₆.CH:NNH.C(NH)NH— 
NH.C(NH)NH:CH.C₆H₅; 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 Monopicate 
salt, C₂₂H₂₇N₁₀O₈·3H₂O, crys (from 95% alc), mp - dec at 214-15°; and Picrolonate 
salt, C₂₂H₂₅N₁₁O₈, crys, mp - dec at 248-49°; were both prepd by Henry et al (Ref 2) 

Dibasic Diethyl Lead Styrphate or Bis-basic 
Diethyl Lead Styrphate, 

\[
\text{OPb(CH₂CH₃)₂OH} \\
|| \\
\text{O₂N.C—C≡C.NO₂} \\
\text{HC—C≡C.OPb(CH₂CH₃)₂OH} \\
\text{NO₂} \\
\text{Ph(trivalental) 51.2%, yel crys, mp - explodes at 229°. Can be prepd by treating di-
ethyl lead dichloride in w at RT with equi-
valent amt of di-ammonium styrphate. Was 
apparented by Burrows et al (Ref 2) as a compon-
ent of ignition comps in electric detonators. 
Requires firing current of 0.36 amp in cement-
ed charge} 
\]

Refs: 1) Beil-not found  2) L.A. Burrows et al, USP 2105635 (1935) pp 2-3 (Other Burrows 
comps are listed in Vol 2, pp B362-63)

Dibenzal Diperoxide or Dibenzyldiene Diper-
oxide (called 3,6-Diphenyl-1,2,4,5-tetroxan, 
Dibenzyldendiperoxid or Dibenzal diperoxid in Ger),

\[
\text{C₆H₅.CH} \overset{O}{\text{O}} \overset{O}{\text{O}} \overset{\text{CH₆C₆H₅}}{\text{O}} \\
\text{; mw 244.24,} \\
\text{O 26.20%; prisms or ndls, mp 191° & 202°} \\
\text{(dec); sol in eth; sl sol in warm chlf & acet} \\
\text{ac; diff sol in alc & petr eth; can be prepd by} \\
\text{treating benzaldehyde with H₂O₂ & coned} \\
\text{H₂SO₄ or ozonizing β,γ-dioxy-Δ-phenylvalero-
nitre} 
\]

Refs: 1) Beil 19, 438 & [452]  2) A. Baeyer & V. Valliger, Ber 33, 2484 (1900)  3) F.I. 
& 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
Dibenzaldehyde. See Benzaldehydeazine, Vol 2, p B35-R

Dibenzo[a]azo-azobenzene. See Bis(benzeneazo)-azobenzene in Vol 2 of Encycl, p B134-L

Dibenzo[a]azo-azoxybenzene. See Bis(benzeneazo)-azoxybenzene in Vol 2, p B134-L

Dibenzo[a]azoxybenzene. See Bis(benzeneazoxy)-azoxybenzene in Vol 2, p B134-R

Dibenzo[a]azoxyglycine. See Bis(phenylazo)-glycine in Vol 2, p B153-L

Dibenzylxidiazid. Ger name for compd described as Bis(α-azidobenzylidene)-azine in Vol 2, p B133-L

Dibenzoat des Peroxy XX. The name given by Criegee to 9,9′-Bis(benzoylperoxyfluoroveny)-peroxide. See Vol 2 of Encycl, p B136-L

Dibenzo[a]fur an and Derivatives
Dibenzo[a]fur an, Diphenylene Oxide or Biphenylene Oxide (CA 5th Decennial Subject Index, p 4106s) (called Diphenylbenzoxyl or Dibenzo[a]fur an in Ger),

H₂C = CH₂ = CH = CH₂ = CH₂
H₂C = CH₂ = CH = O = CH = CH₂ = CH₂

mw 168.18, O 9.51%; wh lts or scales (from alc), mp 82-87°C, bp 288°C; d 1.0866 at 99.3°C; nD 1.6079 at 99.3°C; very sol in eth, benz & ac; sol in alc; insol in w. Other props & methods of prep are given in Beil

Ref: Beil 17, 70-71, (30) & [67-68]

Mononitrodibenzo[a]fur an,
C₆H₄ = C₆H₅(NO₂)

; mw 213.18, N 6.57%.

Several isomers are described in the literature:

2-Nitrodibenzo[a]fur an, yel ndls (from acetic acid), mp 181-82°C & 186-186.5°C; diffuse sol in hot alc (Ref 1)

3-Nitrobenzo[a]fur an, yel ndls (from alc or glc acet ac), mp 151-52°C & 158.5-159.5°C; mod sol in glc acet acid; sl sol in alc (Ref 1)

Yamamiro (Ref 2) also prep 1 & 4-mononitro derivs


2,7-Dinitrobenzo[a]fur an (called 2,6-Dinitro-diphenylen oxyl in Ger),
O₂N·C₆H₅

; mw 258.18;

N 10.85%; ndls (from glc acet ac), mp 245°C & 255-56°C; sol in alc; sl sol in alc, glc acet ac & xylene; almost insol in petr eth; insol in benz; obtd by contact of dibenzofuran & nitrogen dioxide ar RT (Ref 1)

Yamamiro (Ref 2) also prep the 1,8-; 2,8-; 3,8-; 4,8-; 4,6-; and 3,7-dinitro derivs


Trinitrodibenzo[a]fur an,
O₂N·C₆H₅

; mw 303.18,

N 13.86%. Several isomers are known:

2,3,7-Trinitrodibenzo[a]fur an, (called 2,3,6-Trinitro-diphenylen oxyl in Ger), prisms (from benz), mp 223-228°C and when pure 236-37°C; sl sol in alc, benz & glc acet ac; almost insol in petr eth; obtd with other nitroderivs by nitrating 2-nitrodibenzo[a]fur an with fuming HNO₃ (Refs 2 & 4)

2,7,8-Trinitrodibenzo[a]fur an, (called 2,6,7-Trinitro-diphenylen oxyl in Ger), yel ndls, mp 142-43°C; prep'd from 2,6-dinitrodibenzo[a]fur an by heating with fuming HNO₃ (Refs 1 & 3)

Other props are given in the Refs. Yamamiro (Ref 5) also prep 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)


2,3,7,8-Tetranitrodibenzo[a]fur an (called 2,3,6,7-Tetranitro-diphenylen oxyl in Ger),
(O₂N)₂C₆H₂\rightarrow C₆H₂(NO₂)₂

N 16.09%; plts (from xylene), mp 283° & when pure 285-86°; sol in hot glacial ac, xylene & acet; sl sol in alc; diff sol in benz; prep by nitratng 2,6-dinitrodibenzoferan with nitrosoyl hydrogen sulfate (Refs 2, 4, 6 & 7)

1,3,7,9-Tetranitrodibenzoferan (called 1,3,6,8-Tetranitro-diphenylenoxyd in Ger), dk-brown, mp 252.5° & 255°; prep by heating 3,5,3',5',1'-tetrinitro-2,2'-dihydroxybiphenyl with p-toluenesulfonyl chloride & 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 prep 2,3,5,7-; 2,4,6,8-; and 1,3,7,8-tetranitro derivs

9) H. Zahn & H. Zuber, ChemBer 86, 172-80 (1953) & CA 48, 1297 (1954)(Report a tetraniitro 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 nitratd derivs of Dibenzoferan were not found in Beil or in CA thru 1961

\[\text{Dibenzolsulfonyl Peroxide}\] See Benzenesulfonyl Peroxide in Vol 2, p B62-R

\[\text{1,1'-Di(benzoperoxy)-cyclohexane}\] See 1,1'-Bis(benzoilperoxycyclohexane in Vol 2 of Encycl, p B135-L

\[\text{9,9'-Di(benzoperoxy)-fluorene}\] See 9,9'-Bis(benzoilperoxylfluorene in Vol 2, p B135-R

\[\text{9,9'-Di(benzoperoxy)9,9'-fluorene-peroxide}\] See 9,9'-Bis(benzoilperoxylfluorenyl)-peroxide in Vol 2, p B136-L

\[\text{Dibenzopenone-peroxide Dimer}\]

\[\text{(C₆H₅)}₂C\overset{\text{O-O}}{\text{O--O}}\text{C(C₆H₅)}₂\]

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 & CCl₄; diff sol in eth, acet, eth acet & acet ac; insol in alc; was obtd by ozonolysis of 1,1-diphenyl-1-alkenes in CCl₄ 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 decomp completely to benzophenone

Ref(s): 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 H₂O₂ & concd H₂SO₄); JChemSoc-Japan (Pure Chem Sect) 70, 31(1949) & CA 45, 2916(1951); ChemHighPolymers 5, 201 (1948) & CA 46, 1795(1951)(Compd appears to be Dithiobenzopenone-peroxide, mp 70°)
4) Tobolsky & Mesrobian (1954), 172
5) J. 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°]

\[\text{Dibenzotetrazapentalene and Derivatives}\]

\[\text{Dibenzotetrazapentalene, Cl₂H₄N₄}\] may be considered as the parent compd of its tetranitro deriv:

\[\text{Tetranitrodibenzo-1,3a,4,6α-tetrazapentalene (TACOT)}\]
Dibenzoyl and Derivatives

Dibenzoyl, Bibenzoyl, Diphenylglyoxal or Diphenyl diketone. See Benzil in Vol 2 of Encycl, p B64-L

Nitration 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-diaza-3,6-dioxane (called Diphenyl dinitrosacetyl in Ger by Holleman (Ref 2) and Dibenzyglyoximeperoxyde or Diphenyl-dinitrosacyle in Fr by Böseken (Ref 2a))

\[
\begin{align*}
\text{C}_6\text{H}_5\text{.CO} & \quad \text{C} \quad \text{C} \quad \text{C}_6\text{H}_5\quad \text{N} \quad \text{O} \quad \text{O} \quad \text{N} \\
\text{C}_6\text{H}_5\text{.CO} & \quad \text{C} \quad \text{C} \quad \text{C}_6\text{H}_5
\end{align*}
\]

mw 294.26, N, 9.52%; Crystals (from chl), mp 188°, explodes at higher temp with a luminous flame; was obtd when the oil which separates in the prep of \(\text{C}_6\text{H}_5\text{CH}:\text{N}:\text{OH}\) was treated with ether and the ppt purified from hot acet. Its Azine, orn-yel plts, mp - dec 207°, was also prepd

Ref's: 1) Beil-not found 2) A.F. Holleman, Ber 20, 3361(1887); 21, 2840(1888); and Rec 11, 258(1892) 2a) J. Böseken, 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. Scarpatic & M. Rippa, Gazz 88, 804(1958) & CA 53, 18893(1959)

NOTE: Compare with Dibenzoylefuroxan

3,4-Dibenzoylefuroxan (called 3,4-Dibenzoylefuroxan or 3,4-Dibenzoylexidiazol in Ger),

Dibenzoyl Peroxide. See as separate entry in this Volume
D 1186

\[ \text{N} \quad \text{O} \quad \text{N} \]

C\(_6\)H\(_5\),CO,C –– C.CO.C\(_6\)H\(_5\), mw 278.26, N 10.07%; col crys (from alc), mp 118\(^\circ\); diffc sol in alc; was prepd by reducing the low-melt-
ing form of Dibenzoylfuroxan with ZnCl\(_2\) & concd HCl in glacial acetic acid below 40-50\(^\circ\)

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-Dibenzoylfuroxan-2-oxide; or 3,4-Bis (benzoyl)-1,2-dioxa-2,5-
diazene (called 3,4-Dibenzoylfurazan-oxyd or Dibenzoylfuroxan in Ger),

\[ \text{N} \quad \text{O} \quad \text{N}:\text{O} \]

C\(_6\)H\(_5\),CO,C –– C.CO.C\(_6\)H\(_5\), mw 294.26, N 9.32%; crys; exists as a high-melting form, mp 188\(^\circ\) and as a low-melting form (from eth or glac acet ac), mp 86-87\(^\circ\); deflgd 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,

\[ \text{N} \quad \text{O} \quad \text{N}:\text{O} \]

O\(_2\)N.C\(_6\)H\(_4\).CO,C –– C.CO.C\(_6\)H\(_4\).NO\(_2\); mw 384.26, N 14.58%; wh crys (from 2-nitro-propane), mp 154\(^\circ\); was prepd by reacting p-nitroacetophenone with concd HNO\(_3\) and warming cautiously to 55\(^\circ\) (Ref 3)

By a similar procedure, Snyder & Boyer (Ref 3) prepd 3,4-Bis(m-nitrobenzoyl)-furoxan, wh crys, mp 150\(^\circ\). They also prepd the respective Azines of the p-nitro deriv, orn-yel crys, mp 252\(^\circ\); and of the m-nitro deriv - bright yel crys, mp 251\(^\circ\).

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.
6) T.O. Kuda, Yakugaku-Zaushi 78, 808-09 (1958) & CA 52, 18382 (1958)

NOTE: See also 4,5-Dibenzoyl-1,2-diaxa-
3,6-diazine

Dibenzoyl-hydrazine. See Bis(benzoyl)-hydrazine in Vol 2 of Encycl, p B134-R

DIBENZOYL PEROXIDE
AND DERIVATIVES

Dibenzyol Peroxide or Bis(benzoyl)-peroxide
(called Dibenzoylperoxyd, Benzoylsuperoxyd or Benzoylperoxyd in Ger),
C\(_6\)H\(_5\),CO.O.O.CO.C\(_6\)H\(_5\), mw 242.22, O 26.42%; wh thomb crys (from eth); odorless & taste-
less; mp 104-06\(^\circ\)(dec), bp - explodes; Q\(_\text{comb}\) 1552kcal/mol (Ref 6) & 6417cal/g (Ref 8);
\( n_\text{D} \) 1.545 at 20\(^\circ\), sol in acet, eth, gluc acet ac, benz, pyridine, CS\(_2\) & chlor; 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)

Dibenzyol peroxide is flammable, expli-
oxidizing substance. It is dangerous to handle dry; has been known to explode during prepn, purification & storage. A sample exploded mildly at 100\(^\circ\) even under water. The crude product has also exploded at RT over H\(_2\)SO\(_4\) in a desiccator. A mixt of Dibenzyol peroxide & KMnO\(_4\) will deflg, and a mixt
with Na\(_2\)O\(_2\) 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 re-
ported stable after storage for at least 20 days at 45-50\(^\circ\).

The explosion temp value of a sample
contg 10% H\(_2\)O\(_2\) is 125\(^\circ\)(smoke); impact test
with PA App & 2kg wt 4 inches for a 16 mg sample; and Bristane 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 thio-
sulfate & KCIO3.

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 prodct 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 con-
tainers. Max shipping wt by rail express is
25-lbs per shipping case (Ref 23).

For addtl info on the props, reactions
and nature of Dibenzoyl peroxide, see
the following Refs

Ref s: 1) Beil 9, 179, (93) & [157] 2) C.
Brodie, JCS 17, 268(1864) 3) H. von Pech-
mann & L. Vanio, Ber 27, 1511 (1894); 30,
2003 (1897); 33, 1046 (1900)(Prepn from benzyol chloride, H2O2 & NaOH 4) A. Jaques, BritP
2350 (1912); JSCI 32, 674 (1913) & CA 8,
1347 (1914) 5) R.C. Foster, JSCI 40, 84T-
861(1921) & CA 15, 2356 (1921)(Prepn & props)
6) Anon, Jahresber CTR 4, 72 (1924-25)
7) J. Schwyzer, “Die Fabrikation Pharmazeu-
tischer und Chemisch-Technischer Produkte”,
J. Springer, Berlin (1931)(Reproduced by
Edwards Bros, Ann Arbor, Mich) 8) A. Schmidt,
SS 29, 263(1934) 9) K. Nokazi & P.D. Bart-
lett, JACS 68, 1686-92 (1946) and W.E. Cass.
JACS 68, 1976-82 (1946) (Kinetics of decompn in solvs)
10) Kirk & Otherm 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 Ben-
zoyle) 12) G.R. Lappin & D.L. Davidson,
C & EN 26, 3518(1948)(An expln is describ-
ed with suggested explanation & precautionary
measures; in handling of Dewar flasks wrap-
ing 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,
9895 (1951)(Exp ln attempted reduction with LiAlH4) 16) K.S. Bagdasar’yan & R.I.
Milyutinskaya, ZhurFizKhim 27, 420-32
(1953) & CA 49, 5393 (1955)(Decomp in
various solvs) 17) L. Horner & C. Betzel,
ChemBer 86, 1071-72 (1953) & CA 49, 10671
(1954)(Exp decomp by dimethylaniline)
17a) Tobolsky & Mesrobian (1954), 169
18) D.F. DelTar & L.A. Carpio, 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, IndChem-
ist 32, 271-76 (1956)(Props & industrial uses)
21) T. Sumimoto, JapP 5667 (1959) & CA 53,
21818-19 (1959)(Manuf) 22) Hawkins, Org-
Peroxides (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 chl.
The soln is cooled to -5° and 25 ml of 0.1N
ice-cold soln of Na methyldate is added at
once with cooling & shaking. After 4 to 5
mins at -5°, 100 ml of ice water, 5 ml of 10%
H2SO4 and 2g KI in 20 ml of 10% H2SO4 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 perox-
ide (Ref 1). See also Refs 2 & 3
Ref s: 1) G. Braun, OrgSyn 13 (1933), 87-88
2) P.A. Giguere & D. Lamontagne, CanJChem
29, 54-59 (1951)(Polarographic detn of Diben-
zoyl peroxide) 3) A.T. Blomquist & A.J.
Buselli, JACS 73, 3887 (1951)
Dinitrobenzoyl Peroxide or Bis(nitrobenzoyl)-
peroxide, [O2N.C6H4.CO2]2O2; mw 332.22, N
8.43%. The following isomers are known:
2,2'-Dinitrobenzoyl Peroxide or Bis(o-nitro-
benzoyl)-peroxide, crysts (from acetophenone
at 50°), mp 145° dec (Ref 5) or col plates (from
chI), mp 147° expl decomp (Ref 4); was prep
by shaking o-nitrobenzoyl chloride in chlf at 2\(^{\circ}\) with H\(_2\)O \& aq NaOH (Ref 4)

3,3',4'-Dinitro dibenzoyl Peroxide or Bis(m-nitrobenzoyl)-peroxide, ndls (from ethyl acet), mp 134\(^{\circ}\), 137\(^{\circ}\) \& 139-40\(^{\circ}\) (dec); readily sol in eth, acet \& benz; decomp explosively in contact with concd HNO\(_3\) or H\(_2\)SO\(_4\); prep'd as above (Refs 1, 4 & 5)

4,4'-Dinitro dibenzoyl Peroxide or Bis(p-nitrobenzoyl)-peroxide, yel ndls (from toluene), mp 151\(^{\circ}\) (dec) \& 156\(^{\circ}\) (dec); prep'd by the method of Price \& Krebs (Ref 2, 3, 4 \& 5)


Tetranitrodibenzoyle Peroxide or Bis(dinitrobenzoyl)-peroxide, 
\[ (\text{O}_2\text{N})_2\text{C}_6\text{H}_4\text{C}_6\text{H}_5\text{O}_2\text{O}_2; \text{mw} 422.22, \text{N} 13.27\%. \]

Only one isomer is known:

3,5,3',5'-Tetranitrobenezoyl Peroxide or Bis (3,5-dinitrobenzoyl)-peroxide, crystals (from boiling chlf, cooled and MeOH added), mp 160-62\(^{\circ}\) (dec) (Ref 3), mp 158\(^{\circ}\) (Ref 2); prep'd 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(benzoylperoxyfluoranyl)-peroxide.** See Bis(benzoylperoxyfluoranyl)-peroxide in Vol 2, p B136-L

3,6-Dibenzoyl-1,2,4,5-tetra keto-4,5-dihydro pyridazine or 3,6-Dibenzoyl-4,5-pyridazine dione-1,2-dioxide

\[ \text{C}_6\text{H}_5\text{O}\text{C}\cdots \text{N} (\cdots) \text{N} (\cdots) \text{C}\cdots \text{C} \cdots \text{O} \cdots \text{C}\cdots \text{O} \]

\[ \text{mw} 350.28, \text{N} 8.00\%; \text{brick-red crysts; rhomb plates (from acet-eth) or ndls (on evapn from acet) + 2 mola solv, mp - explodes on heating in a capillary tube or on Pt block; sol in acet, benz \& anisole; soln in H}_2\text{SO}_4 \text{ with decmpn; sol} \text{ in benz, chlf \& AcOH; was prep'd by treating 1,4-dibenzoyl butane-2,3-dione (C}_6\text{H}_5\text{COCH}_2\text{CO}_2\text{H}, \text{with N}_2\text{O}_4 \text{ at 25}^{\circ} \text{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 Dibenzyl in Vol 2, p B110-L

**Dibenzylamine and Derivatives**

Dibenzylamine or Bis(benzyl)-amine (called Dibenzylamin in Ger), \((\text{C}_6\text{H}_5\text{CH}_2)_2\text{NH}\); mw 197.27, N 7.10%; col to lt-yel liq, fr p -25.6 to -26.6\(^{\circ}\), bp 215\(^{\circ}\) at 39 mm pressure \& 228-71\(^{\circ}\) at 250 mm pressure; d 1.028; \(\text{np} 1.5743 \text{ at 21.6}^{\circ}\), \(\text{Q}^\circ v\text{comb} 1851 \text{kcal/mol}; \text{readily sol in alc \& eth; insol in w}; \text{methods of prep} \& \text{other props are given in Beil}

Ref: Beil 12, 1035, (453) \& (553)

**Mononitrodibenzylamine,**

\[ \text{O}_2\text{N}\cdot \text{C}_6\text{H}_5\text{CH}_2\cdot \text{NH}\cdot \text{CH}_2\cdot \text{C}_6\text{H}_5; \text{mw} 242.27, \text{N} 11.56\%. \text{Only the 2-Nitro deriv is described in Beil 12, 1078} \]
Dinitrodibenzylamine, (O₂N₆C₆H₄.CH₂)₂NH; mw 287.27, N 14.63%; the following isomers are known:
2,2'-Dinitrodibenzylamine, crys (from alc), mp 99-102; forms a Hydrochloride salt, C₁₄H₁₂₂N₂O₄ + HCl; dec at 238°; and a Nitrato, C₁₄H₁₂₂N₂O₄ + HNO₃, Iy-yel ndls (from w), mp 200-030° dec (Ref 1)
3,3'-Dinitrodibenzylamine, citron-yel prisms (from alc or eth), mp 83.5°, diff sol in w; forms a Hydrochloride salt, yel prisms (from dil HCl), mp 253°; and a Nitrato, Iy-yel prisms (from dil HNO₃), 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 Nitrato, yel plts (from dil HNO₃), mp dec 210-11° (Ref 3)

Trinitrodibenzylamine, C₁₄H₁₂₂N₅O₆; not found in Beil or in CA thru 1961
2,6,2',6'-Tetranitrobenzylamine or Bis-(2,6-dinitrobenzyl)-amine,
[(O₂N₆C₆H₄.CH₂)₂NH; mw 377.27, N 18.56%;
dk-yel ndls (from alc), mp 194°; obtb by intro-
ducing NH₃ into a benzene soln of 2,6-dinitro-
benzbromide; 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, Bull Fr 21, 118 (1917) & CA 11, 3028-29 (1917)

Note: Higher nitrated derivs of Dibenzyl-
amine were not found in Beil or in CA thru 1961

ω,ω'-Dibenzyldiene-diaminoguanidine. See Bis(benzyldieneamino)-guanidine in Vol 2 of Encycl, p B136-L

Dibenzylidene Diperoxide. See Dibenzal Diperoxide

Dibenzylmethane and Derivatives
a,y-Dibenzylmethane or 1,3-Diphenylpropane (called 1,3-Diphenylpropan or Dibenzylmethan in Ger), C₆H₅.CH₂.CH₂.CH₂.C₆H₅; mw 196.28, H 8.22%; liq, fr p 6°, bp 301-03°, 160° at 18 mm press, d 0.9855, b 1.5634; other props & many methods of prep are given in Beil
Ref: Beil 5, 613, (288), [1517] & [1864]

Nitro-nitroso-dibenzylmethane (called 2-Nitroso-2-nitro-1,3-diphenyl-propan in Ger), C₆H₅.CH₂.C(NO)NO₂.C₆H₅; mw 270.28, N 10.37%; blue rhmb crysts (from cold eth & alc), mp 85° (dec) & 72-73°, readily sol in eth, chl & benz; less sol in glc acet ac; diff sol in alc & petr eth; insol in w; was prep by treating dibenzylketoxime with ethyl nitrate & N₂O₅ in ether under cooling, or with N₂O₅ in eth at 0° (Refs 1 & 2)

Dinitro-dibenzylmethane,
C₆H₅.CH₂.(C(NO₂)₂).CH₂.C₆H₅; mw 286.28, N 9.79%; 127.5° & 132°; readily sol in chl; less sol in Nitrobenz; sl sol in eth, glc acet ac & benz; v sl sol in alc & petr eth; insol in w; was prep from the nitroso nitro deriv by oxidizing in glc ac proces with fuming HNO₃ (Refs 1 & 2)
Ref: 1) Beil 5, [1517] & [1865] 2) W. Charlot et al, JCS 1932, 36 (Dibenzyldinitromethane)

X,X-Dinitrodibenzylmethane, C₁₅H₁₄(NO₂)₂; ndls (from chl), mp 139°; obtb by treating dibenzylmethane in glacial acet acid with fuming HNO₃ (Refs 1 & 2)
Ref: 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,
O₃N₆C₆H₄.CH₂.CH₂(NO₂).CH₂.C₆H₄.NO₂; mw 331.28, N 12.69%; dk-yel crystals (from chl & eth), mp 140-41.5°; readily sol in chl; sl sol in alc, eth, w & glc acet ac; was prep from nitromethane and o-nitrobenzylcholoride in alc soln in the presence of Na ethoxide (Refs 1 & 2)
Ref: 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_3)_4; mw 376.28, N 14.89%; ndls
(from chlf + alc), mp 162-69°, obtb by treating
dibenzylmethane with fuming HNO_3 (Refs 1 & 2)
Refs: 1) Beil 5, 613 & 11866; 2) A. Michaelis & A. Flemming, Ber 34, 1293 (1901) &
JCS 80 I, 438 (1901)

NOTE: Higher nitrated derivs of Dibenzyl-
methane were not found in Beil or in CA thru
1961

Di(p-biphenyl)-amine or 4,4'-Di-(phenyl-di-
phenyl)amine. See Bis(p-biphenyl)-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 expn of Diboran-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 Di-
borane) 4) A.T. Whatley & R.N. Pease,
(Thermal expn 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) (Explosion reaction of Diborane
in dry & water-saturated air) 7) P.E. Sample
& H.P. Simons, IEC 50, 1699-1702 (1958)
& CA 53, 9795 (1959) (Explosion reactions of Di-
borane in benzene-saturated air) 8) P.
Breisacher et al, "Flame Front Structures of
Lean Diborane-Air and Diborane-Hydro-
carbon-Air Mixtures", pp 894-902 in 7th Symp
Deton (1959) 9) F.J. Martin et al, "Condensa-
tion of Products in Diborane-Air Detona-
tions", pp 633-44 in 8th Symp Combustn
1962

Di(bornylamino)diazido-copper,
(N_3)_2Cu(H_2N.C_10H_17)2; mw 454.10, N
24.68%; c Chrts, bp - explodes 207-08°, burns
quietly in a flame; insensitive to impact; ins-
sol in w, but slowly decomps; prepd by dis-
solving 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 Dibromomethyene (called
Dibromoacetylen or Dibromathiin in Ger),
Br.C.C.Br; mw 230.74; oily lq having a
penetrating odor, fr p -25 to -25° 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 tri-
bromoethylene & alc KOH
Refs: 1) Beil 1, 246, [222] & [191] 2) Cond-
ChemDict (1961), 356 3) Sax (1963), 675

Dibromomino-benzene Nitro Derivs. See
under Dibromophenylamine

Dibromoaniline. Its Mononitro & Dinitro derivs
are not expd. 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_{6}H_4Br_2; mw 235.92. All isomers are known:
1,2( or ortho)-Dibromobenzene, lq having a char-
acteristic odor, fr p 6.4°, bp 223-24° at 760 mm,
d 1.9759 at 25 °C, np 1.6086 at 25 °C; other props & methods of prepn in Ref 1
1,3(or m)-Dibromobenzene, liq, fr p -6.7 °C, bp 218-18.6 °C; d 1.957 at 25 °C, np 1.5914 at 55 °C; other props & methods of prepn are given in Ref 2
1,4(or p)-Dibromobenzene, sublimable plts (from alc, petr eth or acet) or monoclinc prisms, mp 87.5 °C, bp 220.4 °C; d 2.2836 at 24.2 °C, d (liq) 0.9641 at 99.6 °C; other props & methods of prepn are given in Ref 3
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), HC—C(N3)==C.Br
||
HC—C(Br)==CH ; mw 276.95, N 15.18%; yel ndls (from acet, alc or benz), mp 62 °C, probably explodes when heated rapidly to higher temps; v sol in eth; sol in hot alc; sl sol in w; prep’d by introducing NH3 into 2,4-Dibromobenzene-1-diazonium tetrachloroiodide in the cold (Re fs 1 & 2). The 2,4-Dichlorophenyl Azide explodes when heated rapidly or when sharply struck (See Vol 2 of Encycl, p B44-R)

Mononitrodibromobenzene, C6H5Br2(NO2); mw 280.92, N 5.000%. All possible isomers are known:
1-Nitro-2,3-dibromobenzene, monoclinc prisms, mp 85 °C, d 2.358, d 1.9764 at 109.5 °C; readily sol in acet & chl; sol in eth, eth acet & 3 pts of hot acet (Ref 1)
1-Nitro-2,4-dibromobenzene, yel plts or triclinic prisms (from alc), mp 61-62 °C, vol on steam bath, sublimes w/o decomp, d 1.9581 at 111 °C, sol in hot alc; diffc sol in cold (Ref 2)
1-Nitro-2,5-dibromobenzene, i-tol plts (from acet, acet + petr eth, or eth-alc), mp 84-85 °C, d 1.9146 at 110 °C (Ref 3)
1-Nitro-2,6-dibromobenzene, monoclinc prisms (from alc), mp 82-84 °C, sublimes on steam bath, d 1.9211 at 111 °C (Ref 4)
1-Nitro-3,4-dibromobenzene, monoclinc prisms (from alc or glac acet ac), mp 58-59 °C, bp 296 °C, volatile on steam bath, d 1.9835 at 111 °C; readily sol in gluc acet ac (Ref 4)

1-Nitro-3,5-dibromobenzene, ndls (from alc), prisms or plts (from eth), mp 106 °C, volatile on steam bath, d 1.9341 at 111 °C, insol in KOH (Ref 3)
Other props & methods of prepn the Mono-nitro derivs are found in Beil
Dinitrodibromobenzene, C6H2Br2(NO2); mw 323.93, N 8.60%. All possible isomers are known:
1,2-Dinitro-3,4-dibromobenzene, greenish crysts (from CS2), mp 109 °C, 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 °C, trimorphic: stable form, monoclinc prisms (from concd soln of eth-alc or alc, d 2.274; metastable form, monoclinc prisms (from cooling a saturated soln in eth acet), d 2.317; and labile form, rhmb bipyramidal crysts (from seeding an alc-eth soln); prep’d by nitrating 1-nitro-3,5-dibromobenzene with mixed acid on a water bath (Ref 1)
1,2-Dinitro-3,6-dibromobenzene, monoclinc prisms (from CS2 or glac acet ac), mp 159-60 °C; readily sol in hot abs alc; insol in w; prep’d 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 gl-ac acet ac) or crysts (from CS2), readily sublimes by heating on a w bath, mp 114-15 °C, d 2.313; mod sol in eth, chl & CS2; diffc sol in cold alc, petr eth & gl-ac 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 °C, prep’d by nitrating either m-dibromobenzene or 1-nitro-2,6-dibromobenzene (Ref 3)
1,3-Dinitro-2,5-dibromobenzene, ndls (from CS2, gl-ac acet ac or alc), mp 119-20 °C; obtd with other isomers by nitrating p-dibromobenzene with mixed acid (Ref 2)
1,3-Dinitro-4,5-dibromobenzene, monoclinc prisms (from CS2), mp 71 °C, d 2.373; readily sol in alc & gl-ac 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, rhomb crystals, 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 CS₂), mp 156.5°; diff sol in alc & eth; sl sol in CS₂; 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, chlor & CS₂; 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-dibromobenzene diazonium nitrate & NaNO₂ soln at 0° (Ref 7)

Other props of the dinitro derivs are given in Beil
1,3,5-Trinitro-2,4-dibromobenzene,
C₆H₅Br₂(NO₂)₃; mw 370.92, N 11.33%; it-yel prisms having a greenish shimmer, mp 135°; readily sol in eth, chlor & alc; 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 Na₂SO₃ in benzene contg alc (Refs 1 & 2). Its expl props were not reported

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 Dibromodiamino-anthracinon in Ger), C₁₄H₉N₂O₂Br₂; mw 398.05, N 7.04%. Several isomers are described in Beil:
2,3-Dibromo-1,4-diaminoanthraquinone,
C₆H₄(OCO)₂C₆Br₂(NH)₂ (Ref, p 203)
2,6-Dibromo-1,5-diaminoanthraquinone,
H₂N.C₆H₅Br(OCO)₂C₆H₂Br.NH₂, 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, bm crys (from aniline); sol in common org solvs; insol in w & alkalies [Ref, p (473)]
Refs: Beil 14, 203, 208 & (470), 472, 473

4,8-Dinitro-2,6-dibromo-1,5-diaminoanthraquinone, H₂N.C₆H₅Br(OCO)₂C₆H₂Br(NO₂).
NH₂, It-red crys having a metallic luster (from boiling phenol or Nitrobenz), mp - above 360°; was prepd by treating the nitramino deriv with phenol & H₂SO₄ at RT (Refs 1 & 4)

4,8-Dinitro-2,6-dibromo-1,5-dinitroanthraquinone, O₃N.HN.C₆H₅Br(NO₂)(CO)₂.
C₆HBr(NO₂).NH.NO₂; mw 576.05, N 14.59%; diamond-shaped crys, mp - deflgtr 142-43°; diff sol in boiling acet & glut acet ac; almost insol in other solvs; sol in conc H₂SO₄ giving a yel soln; was prepd by adding 2,6-dibromo-1,5-diaminoanthraquinone to colorless HNO₃ (d 1.52) and cooling

It forms an Ammonium salt,
(NH₄)₂C₁₄H₉N₂O₁₀Br₂-red-brown ndls (from w), deflgtr at 187°; Sodium salt,
Na₂C₁₄H₉N₂O₁₀Br₂, red-brown crys (from hot w), mp - deflgtr on rapid heating; and other salts (Refs 2, 3 & 4)
Dibromodiazenophenol (called Dibrom-diaze-phenol or Dibrom-chinon-diazid in Ger), C,H$_2$N$_2$Br$_2$O; mw 277.93, N 10.08%. Three isomers are found in Beil: 3,5-Dibromo-2-diazenophenol, 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 & glaic ac et; v sl sol in cold w & petr eth (Ref 1, p 523)
2,6-Dibromo-4-diazenophenol, 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-diazenophenol, 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 prep are given in Beil. The expl props of Dibromodiazenophenols were not reported

Refs: 1) Beil 15, 523, 530, 531 & [288]
2) See original papers by authors as given in Beil

2-Nitro-3,5-Dibromo-4-diazenophenol,

\[
\begin{align*}
\text{HC} & \rightarrow \text{C(O)} \rightarrow \text{C.NO}_2 \\
\text{BrC} & \rightarrow \text{C(:N$_2$)} \rightarrow \text{C.Br or} \\
\text{HC} & \rightarrow \text{C(O)} \rightarrow \text{C.NO}_2 \\
\text{BrC} & \rightarrow \text{C(N$_2$N)} \rightarrow \text{C.Br} ; \text{mw 322.92, N 13.01%}
\end{align*}
\]

golden-yel prts (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],

\[
\begin{align*}
\text{C} & \rightarrow \text{C(CH$_3$)} \rightarrow \text{CH} \\
\text{Br.C} & \rightarrow \text{C(N$_2$OH)} \rightarrow \text{C.Br} ; \text{known in the form of its salts:}
\end{align*}
\]

Bromide, C$_7$H$_5$Br$_2$.N$_2$.Br; obtd as yel crystals 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 crystals; in dry state both salts are very expl, detonating at 97-98° (Refs 1 & 2)
Thiocyanate, C$_7$H$_5$Br$_2$.N$_2$.SCN, orn-colored crystals, 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 1, 19 (1898) 3) B. Hirsch, Ber 31, 1261 (1898) & JCS 74 1, 474 (1898)

3,5-Dibromo-2-diazotoluene-4-sulfonic Acid. See under Diazotoluencesulfonic 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 Dibromofumar-säure in Ger), HOOC.CBr:CCBr.COOH; mw 273.89, O 23.37%; crystals (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$_2$C$_4$O$_4$Br$_2$ + ½H$_2$O, 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(bromiomino)-p-quinone. See Benzquinone-1,4-di(bromomine) in Vol 2 of Encycl, p B83-L
Dibromopentaerythritol, \((\text{BrCH}_2)_2\text{C(CH}_2\text{OH})_2\), and its Dinitrato, \((\text{BrCH}_2)_2\text{C(CH}_2\text{ONO}_2)_2\), crystals, mp 70°, were prepared by Koubta et al. (Ref 3). They also reported the prepn of Monobromopentaerythritol, \(\text{BrCH}_2\text{C(CH}_2\text{OH})_3\), and its Trinitrate, \(\text{BrCH}_2\text{C(CH}_2\text{ONO}_2)_3\), crystals, mp 91°. These derivs were prepn 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 prepared Monobromopentaerythritol trinitrate, \(\text{mp 89-90}°\), by nitrating the dinitro deriv with 100% white HNO\(_3\).

Refs: 1) Beil, not found 2) R. Huisgen & I. Ugi, Chem Ber 90, 2915, 2925 (1957) & CA 52, 15485 (1958)

NOTE: See also Dibromodiazophenol

Nitroderivatives of Dibromophenol

4-Nitroso-2,6-dibromophenol (called 4-Nitroso-2,6-dibromophenol or 2,6-Dibrom-chinon-oxim-4 (in Ger).

\[
\text{BrC\text{-C(OH)\text{-C.Br}}} \quad \text{Br.C:O} \quad \text{C.Br}
\]

\[
\text{HC\text{-C(Br)\text{-C(N.OH)\text{-CH}}} \quad \text{mw 280.92, N 4.99%; brn ndls (from tol, alc or glac acet ac or pils (from dil alc), mp - darkens at 160° & decomposes at 168-75° (Ref 1); Henry (Ref 2) obtd tan ndls (from MeOH), mp 168-69°(dec); readily sol in alc, eth & ethyl acetate; sol in NaOH & concd H\text{SO}_4; sl sol in chlf, grac acet ac & w; v sl sol in benz & pett eth; recently prepd by reaction of NaNO}_2 \text{ on 3,5-dibromo-4-hydroxybenzoic acid in ag alc (Ref 2).}
\]

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 addl info and under alternate name with parent compd Dibromophenol


Mononitromidobromophenol, \(\text{HO.C}_6\text{H}_2\text{Br}_2\text{NO}_2\); \(\text{mw 296.92, N 4.72%}. \text{Most possible isomers are known:}

2-Nitro-3,6-dibromophenol, goldn-yel ndls, mp 77° (Ref 1)

2-Nitro-4,6-dibromophenol, yel pils (from alc, eth or gluc 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)

DIBROMOPHENOL

AND DERIVATIVES

Dibromophenol (called Dibrom-phenol or Dibrom-1-hydroxy-benzol in Ger), C\(_6\)H\(_5\)Br\(_2\)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 decomp) (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 CC\(_4\) 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

3-Nitro-4,6-dibromophenol, yel ndls (from petr eth), mp 77-78°; forms a Hydrate, C₆H₅Br₂NO₃.H₂O, 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 acid acet; 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 3)

4-Nitro-2,6-dibromophenol, prisms or plts (from alc, petr eth or glac acid ac), mp 141-46°, volatile on steam bath, dec on heating above its mp (Ref 6)

Other props & methods of prep of the mono-nitro derivs are found in Beil.


Dinitrodibromophenol, HOC₆H₅Br₂(NO₂)₂; mw 341.93, N 8.20%. Several isomers are known: 2,4(ar or 6)-Dinitro-3,5-dibromophenol, 1-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 6)-dinitro-3,5-dibromoisocyanate with concd H₂SO₄ at 180-90°, or by heating 1,3-dinitro-2,4,6-tri bromobenzene 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°; prep 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 prep by reacting 3-nitro-2,4,6 tri bromophenol with dil HNO₃ at 12°, or 3,4-dinitrophenol with Br (Ref 3)

X,X-Dinitro-2,4-dibromophenol, ndls (from alc), mp 146-46.5°; prep by reacting propanolic acid-2,4-dibromophenyl ester with 2/1 HNO₃/H₂SO₄; forms Ba & K salts (Ref 2)


2,4,6-Trinitro-3,5-dibromophenol or Dibromopicric Acid, HOC₆H₅Br₃(NO₂)₃; mw 386.92, N 10.86%; prisms (from chlf, CCl₄ or benz), mp 165°, 173°, explosive; gives a yel color in alc or w; can be prep by nitrating 3,5-dibromophenol with HNO₃ in glac ac ac, or with mixed acid


3,5-Dibromophenol-4-diazoniumhydroxide [called also 3,5-Dibrom-1-oxy-benzol-diazoniumhydroxyd-(4) in Ger], HOC₆H₅Br₂.N(NO₃).OH; known as its Nitratrice, which deflgr on heating; separated as an amorphous ppt when 2,4,6-tri bromobenzene-1-diazonium-nitrate was heated with benz on a w bath (Revs 1 & 2)

Revs: 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, C₆H₅NH₂Br₂, may be considered as the parent compd of its nitro derivs.

The Mononitro and Dinitro deris of Dibromoaniline are not expl. The following nitro diris of Dibromophenyltrimethane are of interest:

2,6-Dibromo-4-nitrophenyltrimethane, BrC ≡ CNH(NO₂)C ≡ Br

HC ≡ C(NO₂)C ≡ CH; mw 340.94, N 12.35%; almost col ndls or yel-bm prisms (from benz or chlf), mp - explodes at 122°; readily sol in benz, chlf, eth & glac acid ac; less sol in alc & w; almost insol in petr eth & HCl; was prep 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-nitrophenyltrimethane, BrC ≡ C(NH.NO₂)C ≡ NO₂

HC ≡ C(Br)C ≡ CH; orn prisms (from chlf + petr eth), mp 91-92°; was obtd by treating an acetic acid soln of 4,6-dibromo-2-nitroaniline with HNO₃, followed by addn of acetic anhydride in the cold. A Barium salt, Ba(C₆H₅N₂O₄Br₂)₂, yel prts, was also prep'd.
Their expl props were not investigated (Ref 2)

Dibromophenyl Azide. See under Dibromobenzene

**2,6-Dibromoquinononechlorimide** [called 2,6-Dibrom-p-chinon-chlorimid-(4) in Ger],
\[ \text{BrC} \sim \text{C(=O)} \sim \equiv \text{CBr} \]
\[ \text{HC} \sim \text{C(NCl)} \sim \equiv \text{CH} ; \text{mw 209.38; yel prisms (from glc acet ac or alc), mp 80-83°, dec at 121°; was prepd by adding dropwise a 10% aq chloride of lime soln to a st acidic soln of the Sn double salt of 2,6-dibromo-4-amino-phenol (Ref 1)} \]

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.


Dibromoxyline. See item q in Vol 2, p C169-R, under CHEMICAL AGENTS

**Di(butylamino)diazo- and triazo-copper Compounds.**

**Di(butylamino)diazo-copper,**
\[ ([\text{N}_3]_2\text{Cu(C}_4\text{H}_9\text{NH}_2)_2] \], green ndls, mp 113-14°,
\[ \text{dfgr at 196°, does not expl under impact; was prepd by addn of NaN}_3 to an amine soln of n-butyl Cu salt (Ref 2) } \]

**Di(iso-butyramino)diazo-copper,**
\[ (\text{N}_3)_2\text{Cu(CH}_3)_2\text{CH}_2\text{CH}_2\text{NH}_2)_2] \], grn crstns, 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-butyrammonium)-triazolo-copper,**
\[ ([\text{CH}_3])_2\text{CH}_2\text{NH}[\text{Cu(N}_3)_3] \], grn-brown crstns, 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-buty)-benzidine** (called Di-isobutyrbenzidin in Ger),
\[ -\text{C}_6\text{H}_4\text{-NH-CH}_2\text{CH(CH}_3)_2] \]; may be considered the parent compd of its nitro derivs:

**3,5,3',5'-Tetranitro-N,N'-diisobutylbenzidine,**
\[ -\text{C}_6\text{H}_2(\text{NO}_2)_2\text{-NH-CH}_2\text{CH(CH}_3)_2] \]; mw 476.44, N 17.64%; dk red crstns (from ethyl ester of benzoic acid), mp 194°; obt 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'-diisobutyl benzidine,**
\[ -\text{C}_6\text{H}_2(\text{NO}_2)_2\text{-N(NO}_2)_2\text{-CH}_2\text{CH(CH}_3)_2] \]; mw 566.44, N 19.78%; ndls (from concd HNO}_3), mp-dec at 205°; obt 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'-Di-butylicarbamidilide. See Centralite, Butyl in Vol 2 of Encycl, p C140-L

Dibutylidiphenylurea. See Butyl Centralite under Centralites in Vol 2, p C140-L
**Dibutyl Magnesium, Mg(CH₂₇₂₇₂₇₂₇₂₇₂)**; mw 138.54; clear, dk-amber liq; a 20% soln in toluene ignites on contact with red fuming HNO₃; was prep'd by ppt of Mg chloride from butyl Mg chloride in ether soln with dioxane. This compd was prep'd for evaluation as a fuel, or fuel additive in ram jets & rockets. 


**Di-t-butyl Peroxide or t-Butyl Peroxide,** (CH₃)₃C.O.O.C(CH₃)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°, nₑ 1.3872 at 20°; stable in the presence of concd HCl & strong bases; can be prep'd from t-butyl alcohol & H₂O₂; t-butyl hydrogen sulfate & t-butyl hydroperoxide; and by reaction, of t-butyl alcohol or isobutylene, sulfuric acid & H₂O₂ (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.


---

**Dibutylperoxy-butane.** See 2,2-Bis(t-buty1peroxy)-butane in Vol 2 of Encycl, p B137-R

**Dibutylperoxy-propene.** See 2,2-Bis(t-buty1peroxy)-propene in Vol 2, p B137-R

**Dibutyl-o-phthalate (DBP or BuPh)** [called Phthalsäure-dibutylerster or Dibutylphthalat in Ger], C₆H₄(COO(CH₂)₃.CH₃)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°, nₑ 1.4900 at 20°, viscosity 0.203 poise at 20°; miscible with common org solvs; v sl sol in w; prep'd by treating n-butyl alcohol with phthalic anhydride in the presence of H₂SO₄ (Ref 1)

Dibutylphthalate is used as a solv for aromatic nitrocomps, such as DNT & Di-nitroethybenzene. 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 Di-
butylphthalate content of propellants is re-
ported by a number of investigators (Ref 5,
6a, 7, 8, 9, 10, 11, 12, 13, 14, 15 & 16a).
Following are two methods used by US mil-
itary installarions in analysis of propellants:
A. Zinc Reduction-Volumetric Procedure
(Ref 16a, Method 203.5). This method is
used for determining the DBuPh and DEPh
content of propolnts that do not contain inter-
ferring esters such as other phthalates, di-
methyl sebacate, triacetin, or sucrose octa-
acetate
Procedure. a) Prepare a specimen of propo-
lnt 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 diethy1
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 di-
solve the dried residue. Add 15 ml of distilled
water 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 ether 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 (ex-
traction) 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 Erlen-
meyer by blowing hot air and add 10 ml of
95% ethyl alcohol (MIL-E-463, Grade 1) di-
recting it around the sides, with swirling
p) Add 3-4 drops of phenolphthalein indicator
Prepare it by dissolving 0.1 g of phpt (re-
agent 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) drop-
wise with a medicine pipet until the soln turns
just pink, and then add 0.5N HCl soln drop-
wise until the pink color just disappears.
Prepare alcoholic KOH soln by dissolving
30 g of ACS grade KOH (85%) pellets in a
mixture of 600 ml ethyl alc and 50 ml w, filtering
thu a Whatman No 41 filter paper, diluting
1 to 1 with alc in a Pyrex bottle and cover-
ing it with a tight fitting rubber stopper
t) By means of a delivery pipet, add a volume
of KOH soln calcld to be at least twice that
required to saponify the DBuPh present
a) Reflux for 30 mins under water condenser and, if a ppt forms during heating, add a small amount of water thru the top of condenser.

c) Stop refluxing, add 50 ml of water 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 phpt 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, u, and v. Take the reading of blank titration (A).

w) Calculate as follows:

\[
\% \text{DBuPh} = \frac{13.92(A-B)N}{W}
\]

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-259. Propellant components sol in methylene chloride include triacetin, succrose octaacetate, DNT, TNT, nitrate esters (such as NG, TEGN, PETN, BTTN, PETRIN), DPA, 2-NDPA, ethyl cinnamate, 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 2355779 (1943) & CA 38, 3130 (1944) 3) All & En Exps 1946, 41-42 4) S. Berman et al, IEC 40, 1312-19 (1948) (Reaction rate of catalytic esterification of DBP)

5) S. Wachtell, PACheM Lab Rept 129161 (17 Jan 1950) (Determination of the DBP content of Solventless Powder, Composition 17: NC (12.6%) 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, J Appi Chem (London) 3, 421-25 (1953) (Detn of DBP in Cordite)

J. Tranchant, MP 35, 308-16 (1953) & CA 50, 2173 (1956) (Detection, distinction & detn of phthalic esters in propoints)


12) J. Sourd MP 38, 371-82 (1956) & CA 51, 11719 (1957) (Detn of DBP in propoints)

13) G. Norwitz, Anal Chim Acta 19, 216-23 (1958) (Detn of diethyl & DBP's in propoints)

14) L. Marvillet & J. Tranchant, MP 41, 179-82 (1959) (Analysis of phthalic esters in propoints)


**Dibutylsebacate** (called Sebacinsäure-dibutyl-ester or Dibutylsebacinat in Ger),

\(C_{6}H_{5}OOC(CH_{2})_{2}COOC_{6}H_{5}\); mw 314.45, O 20.35%; clear col, odorless liq, fr p - 11°, bp 349°, d 0.936, np 1.4399 at 25°; obd by reaction of sebacic or sebacic acid with butyl alcohol & HCl in benz (Refs 1, 2 & 3)

Hopper (Ref 2) dert the props of Dibutylosebacate and found it to be equal to DBP as a
gelatinizing agent for NC. It is currently used in some proplnts (Ref 4).


Dibutyltartrate (called 4- or d-Weinsäure-di- butylester or Dibutyl L-tartrat in Ger),
(CHOH.COO.C4H9)2; mw 230.30, O 27.79%;
liq, fr p 21.8°, bp 320° at 765 mm of Hg, flash
p 132°, d 1.0997 at 20°, nD 1.4451 at 20°;
viscosity at 18° 1.0587, at 40° 0.2532, at
80° 0.455 g/cm sec; may be prep’d by dis-
tillling tartratic 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 pro-
Plnts which produce no flash from the gun
barrel. An example of such a compn is: NC,
DNT & ester of an org acid, such as diaxyl
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),
C9H20N2O, mw 172.27, N 16.26%. Several
isomers are described in Beil:
N,N1-Dibutylurea, C4H9.NH.CO.NH.C4H9;
crysts (from eth & petr eth), mp 75°, bp 115°
at 0.03 mm of Hg (Ref 1)
N,N1-Dibutylurea, (C4H9)2N.CO.NH2; crysts,
hygro, solidification point 22-25°, bp 118°
at 2.3 mm; forms a Plicrate salt, C9H20N2O +
C2H5N3O7, mp 82-83° (Ref 2)
N,N1-Di-isobutylurea, [(CH3)2CH.CH2.NH2]CO;
ndls (from dil alc), mp 134-36°; insol in cold w;
readily sol in hot w, alc or eth (Ref 3)
N,N1-Di-isobutylurea, [(CH3)2CH.CH2.]N;
CO.NH2; crysts, mp 72-74°, bp 180° at 25 mm;
readily sol in common org solvs; forms an
Oxalate salt, 2C9H20N2O + C2H4O4, prisms
(from w), mp - dec 115° (Ref 4, p 170)
N,N1-Di-(d-sec-butyl) urea,
[CH3.CH2.CH(CH3).NH2]CO; ndls; readily sol
in alc; v sl sol in w (Ref 4, p 160)
N,N1-Di-(al-sec-butyl)urea,
[CH3.CH2.CH(CH3).NH2]CO; ndls (from dil alc),
mp 137-38° (Ref 5)
N-Butyl-N1-(d-sec-butyl)urea,
CH3.CH2.CH(CH3).NH.CO.NH.C4H9; ndls, mp.
47° (Ref 4, p 160)
N-(d-sec-Butyl)-N1-(d-sec-butyl)urea,
[CH3.CH2.CH(CH3).NH2]CO; crysts, mp 132°
(Ref 4, p 162)
N-isoo-Butyl-N1-tert-butyl-urea,
(CH3)2C.NH.CO.NH.CH2.CH(CH3)2; crysts,
mp 163° (Ref 4, p 174)
N,N1-Di-tert-butylurea,
(CH3)2C.NH.CO.NH.C(CH3)3; crysts (from
alc), mp 243°, sublimes at 250°; sol in alc &
eth; insol in w (Ref 6)
Refs: 1) Beil 4, [634] & [2991 2) Beil 4,
(372), [635] & [301] 3) Beil 4, 160 & [319]
4) Beil 4, 160, 162, 170 & 174 5) Beil 4,
162 & (372) 6) Beil 4, 175 & [321]

Di(1,1,1-trinitro-iso-butyl)-urea or Bis(1,1,1-
trinitro-iso-butyl)-urea,
C2H5.CH.NH.CO.NH.CH.C2H5
C(NO2)3
C(NO2)3 ; mw 424.26, N 25.34%; wh solld, 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 & propionalde-
hyde, and the reaction mixt heated for 20 mins and
diluted with water
Refs: US Rubber Co Quarterly Progress Repr
14, "Synthesis of Explosives and Propellants
or昱 Res & Develop Dept, Gen Labs,
Passaic, NJ (16 May 1951) (Contract NORD
10129)

NOTE: No other nitrated derivs of Dibuty-
urea were found in Beil or CA thru 1961

Di-n-butyl Peroxide (called Dibutylper-
oxyd or "Butylperoxyd" in Ger),
C4H7.CO.OO.CO.C4H7; mw 174.19, O 36.74%;
gas, crysts at -80°; Qform in liq state at 25°
-161.0 & in gaseous state at 25°-60 kcal/mol
(Ref 3); Qcomb -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 Na2O2 & butyryl chloride in eth at 0°, or butyric anhydride in petr eth at -15° (Ref 1)


Di-iso-butyryl Peroxide (called Disobutrylperoxyd in Ger), (CH3)2CH.CO.OO.CO.CH(CH3)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 decomp


Dicadmium-acetato-perchlorate-acetate,
[Cd2(CH3COO)2](CH3COO).ClO4.3H2O; mw 555.4, ClO4 17.9%; col ndls, mp - deflgr violently on heating; was obtd when a soln of Cd acetate in HClO4 was allowed to evap slowly in the presence of CH3COOH

Refs: 1) Beil-not found 2) R. Weinland & H. Schlaich, ZAnorgChem 150, 41 (1925) & CA 20, 720 (1926)

Dicamphoryl Peroxide,

\[
\begin{align*}
\text{CO}_2\text{CO} & \quad \text{C}_8\text{H}_{15} \\
\text{CO}_2\text{CO} & \quad \text{C}_8\text{H}_{15}
\end{align*}
\]

mw 398.44, O 32.13%; cryssts (from acet-petr eth 4/1), mp 142° with expl decomp, 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 & Na2O2 to an ethereal soln of d-camphoric anhydride at 0°. Compare with d-Camphoric Acid Peroacid 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),

\[
\text{CH}_3\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{CO} \rightarrow \text{O}_2
\]

CH3.CH2.CH2.CH2.CH2.CH2.CO; 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 BaO2 in the presence of a little water


Dicarbamylamine. See Biuret in Vol 2, p B164-L

\[\text{N,N'}-\text{Dicarboethoxy-1,3-diamino-isopropanol or N,N'}-\text{Isopropanol Diurethane}\]

(C2H5OOC)NH.CH2.CH(OH)-CH2NH(COOC2H5)
and its nitrated derivative,

(C2H5OOC)(O2N)NH.CH2.CH(OONO2)-CH2
N(NO2)(COOC2H5), serve as intermediates in the prep of Dinitramino-isopropanol Nitrate, described under Diaminohydroxypropane and Derivatives

\[\text{Dicarboethoxyethyleneediamine and Derivatives}\]

\[\text{N,N'}-\text{Dicarboethoxyethyleneediamine; 1,4-Dicarboethoxy-1,4-diazaabutane or Ethylenediurethane}\]

(called N,N'-Dicarbàthoxyáthylendiamin; Äthylene-bis-carbamidsäureäthylester; or Äthylendiurethan in Ger),

C2H5.OOC.NH.CH2.CH2.NH.COOC2H5; mw 204.22, N 13.72%; ndls (from w), mp 112° or
prisms (from alc), mp 113-8* readily sol in alc
or eth; sol sol in w; obd by heating ethylene-
diisocyanate with abs alc, or by reaction of
ethylenediamine & chloroformic acid ethyl
ester at 0
Ref: Beil 4, 254, [693] & [1532]

N,N'-Dinitro-N,N'-dicarboxethylenediami-
none; 1,4-Dinitro-1,4-dicarboxy-1,4-diaza-
bute (called N,N'-Dinitro-N,N'-dicarbóxóxy-
äthylendiamin or N,N'-Dinitro-äthylenedi-
turan in Ger.),
C2H5OOC.N(NO2).CH2.CH2.N(NO2).COOC2H5;
mp 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
HNO3
Ref: Beil 4, 257 & [1547]

4,7-Dicarbethoxy-1,4,7,10-tetrazadecane. See
Bis(aminomethyl)-ethanediurethane in Vol 2,
p B129-R

Dicarboxamide-acetylene. See Acetylene-
dicarboxamide in Vol 1, p A65-L

Dicarboxanilide-acetylene; Di(N-phenyl-
carboxamide)-acetylene or Bis(carboxanilide)-
acetylene, C5H5NOC.C=CC.HOC5H5;
mw 264.27, N 10.60%; pinkish-white crys-
ts (from MeOH-water), mp 194-96°; was prepd
from acetylenedicarboxylic acid by a series of re-
actions to give bromofumaramide which was
dehydrohalogenated by a dil soln of methanolic
KOH.

Its hexanitro deriv, N,N'-Di(2,4,6-trinitro-
phenyl)acetylene-dicarboxamide,
(NO2)2C6H2HOC.C=CC.HOC6H2(NO2)2; 5
mw 534.27, N 20.97%; wh crys, 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 iso-
lated some expl products. For example,
N,N,N',N'-Tetrachlorotriamine or N,N,N',N-
Tetrachlorobesane-diamine,
Cl2NOC(CH2)4CONC12, obd by reacting adi-
pic amide & aq NaHCO3 at 0° with Cl gas for
2hrs, dissolves in boiling water with degra-
dation, giving a highly explo oil (di- or trichloro-
amine). The Sodium salt of adipic acid dibromo-
amide decompl at 30-32°
Ref: P. Eckert et al, Reyna, SyntheticaZell-
wolle 29, 2-8, 89-99(1951) & CA 45, 7527,
8761(1951)

Dicarboxy-phenazine-N-oxide, C14H4N5O3,
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')
C14H4N6O13; mw 464.22, N 18.11%;
\[
\begin{align*}
\text{COOH} & \quad \text{NO} & \quad \text{COOH} \\
\text{O}_2\text{N.C} & \equiv \text{C} & \equiv \text{C} & \equiv \text{C} & \equiv \text{C} & \equiv \text{C}.\text{NO}_2 \\
\text{HC} & \equiv \text{C} & \equiv \text{N} & \equiv \text{C} & \equiv \text{CH} & \equiv \text{NO}_2 \\
& & & & & \equiv \text{NO}_2
\end{align*}
\]

assumed structure, since it was not definitely established; why expl compd; obcd 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

\[\alpha,\alpha'-\text{Dicarboxypimelyl Tetrazide} \text{ (called } \alpha,\alpha'-\text{Dicarboxypimelinsäure Tetrazid in Ger.),} \]

\[(\text{N}_2\text{O.C})_{2}\text{CH}.(\text{CH}_2)_3.\text{CH(CO.N}_3)_2; \text{ mw 348.25, N 48.27%; wh powd (from eth), explodes by friction, impact or rapid heating; melts under benz at 50-55°C; stable in dry state; readily sol in abs alc; less sol in eth, benz or chl; insol in water; was prepd by reaction of the tetrahydrazide with HCl} \]


\[\text{Dicarboxypropionyl-peroxide. See } \text{Bis(succinyl)-peroxide in Vol 2 of Encycl, p B156-R} \]

\[\text{Dichloralperoxyhydrat. A Ger name for the compd } \text{Bis(α-hydroxy-β,β,β-trichloroethyl)-peroxide described in Vol 2, p B148-R} \]

1,1-Dichloramino-5-(α-chlorophenyl)-α-tetrazole

or 1,1-Dichloramino-5-(2'-chlorophenyl)-1H-tetrazole,

\[\text{o-ClC}_6\text{H}_4.\text{C} \equiv \text{N(NCl}_2\equiv \text{N} \equiv \text{N} \equiv \text{N} \equiv \text{N} \equiv \text{N}; \text{ mw 264.52, N 26.47%; granular solid (from alc), exploded violently when dried by spreading and rubbing on a porous porcelain plate: was prepd by treating 1-amino-5α-chlorophenyl-α-tetrazole (qv) with hypochlorous acid} \]

Refs: 1) Beil not found  2) R. Stolle et al, JPraktChem 138, 2 & 10 (1933) & CA 27, 4798 (1933)

1,1-Dichloramino-5-phenyl-α-tetrazole or 1,1-Dichloramino-5-phenyl-1H-tetrazole,

\[\text{(C}_6\text{H}_5).\text{C} \equiv \text{N(NCl}_2\equiv \text{N} \equiv \text{N} \equiv \text{N} \equiv \text{N}; \text{ mw 230.1, N 30.44%; OB to CO}_2 \text{-147.3%; solid, extremely violent and sensitive expl; was prepd by treating 1-}
\]

1-amino-5-phenyl-α-tetrazole with hypochlorous acid

Ref: 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)-α-tetrazole.

See in Vol 1, p A266-R

\[\text{Dichloroacetylene or Dichloroethylene (called Dichloroacetylen or Dichloroethin in Ger),} \]

\[\text{Cl.CCl.Cl; mw 94.93; oil, ft p -64 to -68°C, bp 32-33°C at 748 mm press, forms ndls at temps below its fr p; explodes on contact with air; d 1.261 at 20°C; nD 1.4279 at 20°C; miscible with many org solvs; can be stored at RT in ether soln, with which it forms an addn compd.} \]

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 trichloroethylene or tetrachloroethane with KOH; by heating Ba trichloroacrylate or Ba chloropropionate; 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

Dichloroacetyl Peroxide or Chloroacetyl Peroxide (called Bis-chloroacetyl-peroxyc in German), ClCH₂-CO.OO.CO.CH₂Cl; mw 186.98; small hygroscopic ndls, mp 55-36⁰C; dec > 70⁰C; explodes violently on impact, friction, or when mixed with Al, Pb peroxide, S or KClO₃; readily sol in most solvs, except petr ether; can be prep'd from chloroacetyl chloride & 3% H₂O₂ in the presence of Na acetate under cooling and shaking of the mix, and from chloroacetyl chloride & Na₂O₂ in water at 0⁰C, or 3% H₂O₂ in pyridine in the cold
Refs: 1) Beil 2, 199 & (449) 2) L. Vaino & E. Uhfelder, Ber 33, 1043 (1900) & JCS 78 I, 371 (1900) 3) Tobolsky & Mesrobian (1954), 169

N,N-Dichloroalkylamines or Alkyl dichloroamines RNCl₂ were patented by Riedel (Ref) for use as liq expts. 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-Dichloroamino-5-(o-chlorophenyl)-α-tetrazole

1,1-Dichloroamino-5-phenyl-α-tetrazole. See 1,1-Dichloroamino-5-phenyl-α-tetrazole

1,1-(N-Dichloroamino)-5-(p-tolyl)-α (or 1H)-tetrazole. See 1,1-(N-Dichloroamino)-5-(p-tolyl)-α (or 1H)-tetrazole in Vol 1, p A266-R

Dichloroanilino-ethane or Dichlorophenylaminomethane. 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 Dichloroazobenzene

Dichloroazobenzene and Derivatives

Dichloroazobenzene (called Dichloroazobenzol in German), Cl₄C₆H₄N₂·N₄Cl₄H₄Cl, mw 251.11, N 11.16%. Its 2,2'-Dichloro-o-tetra-colns (from petr ether or alc), mp 136-37⁰C, 3,3'-Dichloro-orn-red ndls (from alc,) mp 10¹⁰, 4,4'-Dichloro-derivs, l-ylnd ndls (from acet), mp 188° (Ref 1); and 2,4-Dichloroazobenzene, Cl₂C₆H₄N₂·N₂C₆H₅, orn crys (from alc), mp 105° (Ref 2) are described in the literature

Mononitrodichloroazobenzene, C₆H₄Cl₂N₃O₂; mw 266.10, N 15.79%. Two isomers are found in Beil
2'-Nitro-2,4-dichloroazobenzene,
Cl₂C₆H₃N₂·N₂C₆H₄(NO₂)₂; dk-salmon colored ndls (from alc), mp 155.5° (Ref, p 51); and 2 (or 3)-Nitro-4,4'-dichloroazobenzene,
(O₂N)₂Cl₆C₆H₃N₂·N₂C₆H₄Cl; pale-ylnd crys, mp 210° (Ref, p 58)
Ref: Beil 16, 51, 58

Dinitrodichloroazobenzene,
(O₂N)₂Cl₆C₆H₃N₂·N₂C₆H₄Cl(NO₂); 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 gluc acid acet; s1 sol in acet; insol in alc; was prep'd by oxidation 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°; obt 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°, prep'd 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. Bartin 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, AustralianChem 11, 491
(1958) & CA 53, 7147-48 (1953)
Tetranitrodichloroazobenzene,
(O₂N)₄Cl₂C₆H₄N₃C₆H₂Cl(NO₂)₂; mw 431.11,
N 19.50%. Two isomers are known:
2,4,2',4'-Tetranitro-5,5'-dichloroazobenzene,
yel crys (from 50% acetic acid) turning greenish
gradually on exposure to light, mp 93°,
explodes on further heating; readily sol in alc,
eth, petr eth, glacial acetic acid & benz; was obtd
on warming a-nitrosao-5-chloro-2,4-dinitro
phenylhydrazine with 50% acetic acid (Refs 2 &
4)
2,4,2',4'-Tetranitro-6,6'-dichloroazobenzene,
onndls (from boiling chlorobenz + alc), mp
244-45°; was obtd when 2,4,6,2',4',6'-hexa
nitroazobenzene 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 compn with Nitrostarch as a gelatinizer
(Refs 1 & 2)

Mercury salt, flocculent red ppt; was prep'd
by adding while stirring an aq soln of Hg acetate
(1 mol) to a hot aq soln of N,N'-dichloro-
azodicarbamidine with NH₃ added from
time to time to keep the mix just sl acid at the
start of the reaction & sl alkaline towards the
end
Silver salt, black ppt; was prep'd by adding
while stirring an aq soln of ammoniacal AgNO₃
(2 mol) to a hot aq soln of N,N'-dichloro-
azodicarbamidine. 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 co-
precipitated with materials such as dextrin
Refs: 1) L.A. Burow, USP 2086533 (1937)
& CA 31, 6467 (1937) 2) Blatt, OSRD 2014
(1944)

Dichloroazoxybenzene
and Derivatives

Dichloroazoxybenzene (called Dichlor-azoxy-
benzol in Ger), Cl,C₆H₄,N(O):N,C₆H₃Cl;
mw 267.11, N 10.49%. Its 2,2'-Dichloro-, t-
yel ndls (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,C₆H₄,N(O):N,C₆H₃(N,O₂)Cl; mw 312.10,
N 13.45%. 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 prod-
ucts (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.
Warers, JChemSoc 1950, 1018 & CA 44,
7256 (1950)
Di nitrodichloroazoxybenzene, 
\( \text{Cl(O}_2\text{N)C}_6\text{H}_3\text{N}:\text{O):N.C}_6\text{H}_3\text{(NO}_2\text{)Cl; mw 357.11, N 15.69%} \). Only two isomers are known: 
5,5'-Dinitro-2,2'-dichloro-, reddish-yellow needles (from ace), mp 163-64° (Refs 1 & 3); and 
3,4-Dinitro-3,3'-dichloro-azoxybenzene, mp 157° (dec) (Ref 2). Other props & methods of prep are given in the Refs 

Trinitrodichloroazoxybenzene, 
\( \text{Cl(O}_2\text{N)C}_6\text{H}_3\text{N}:\text{O):N.C}_6\text{H}_3\text{(NO}_2\text{)Cl; mw 402.11, N 17.42%} \). Three isomers are known: 
Trinitro-3,3'-dichloroazoxybenzene, crystals, mp 182° (Refs 1 & 2) 
2,4,6-Trinitro-3,3'-dichloroazoxybenzene, crystals, mp 165° (Refs 1 & 2) 
Trinitro-4,4'-dichloroazoxybenzene, yellow crystals (from glacial acetic acid), 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 
3,5,5',5'-Tetranitro-4,4'-dichloroazoxybenzene, 
\( \text{Cl(O}_2\text{N)C}_6\text{H}_3\text{N}:\text{O):N.C}_6\text{H}_3\text{(NO}_2\text{)Cl; mw 447.11, N 18.80%} \). Crystals, mp 226-28°; was obtd by treating 3,3',5,5'-tetranitro-4,4'-dihydroxyazoxybenzene (mp 186-87°) with p-toluene-sulfonfyl chloride (Refs 1 & 2). The expl props of this compd were not reported 

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

Dichlorobenzene 

Dichlorobenzene (calleld Dichlor-benzol in Ger), 
\( \text{C}_6\text{H}_4\text{Cl}_2; \text{mw 147.01}. \) This compd exists as - ortho, meta & para derivs. A mixt of these isomers is obt'd when benzene vapor & chlorine gas, preheated to 400-500°, are passed thru a reaction chamber at 650-700° (Ref 4); 
\( \alpha(\text{or} 1,2)-\text{Dichlorobenzene, col liq, fr p -17°, bp 180.5°, flash p 155°F (Ref 5); d 1.3003 \) at 25°, \( \text{nD}_2 \) 1.5501 at 20° (Ref 1) 
\( \beta(\text{or} 1,3)-\text{Dichlorobenzene, liq, fr p -24.8°, bp 173°, d 1.2828 at 25°, nD}_2 \) 1.5464 at 20° (Ref 2) 

\( \beta(\text{or} 1,4)-\text{Dichlorobenzene, white crystals, exists in two modifications: α-monoclinic, mp 53.5° and β-triclinic, mp 54° which is interchangeable 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 concentra-tions of Dichlorobenzenes, can produce para-lysis 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 5ppm by vol. Long exposure to the p-deriv is reported capable of producing cataracts (Ref 6) 

Other props & methods of prep are given in Beil 

Azido & Diazio Derivatives 

Dichlorobenzene 

1-Azido-2,4-dichlorobenzene. See Vol 2, p B44-R 
1-Azido-2,5-dichlorobenzene, \( \text{N}_3\text{C}_6\text{H}_3\text{Cl}_2; \text{mw 188.01, N 22.34%} \). Cycl crystals, mp 30°; readily sol in common org solvs; was prepd by reaction of aq ammonia with a paste of 5,5-dichlorobenzenediazonium tetrachloro-iodide & crushed ice (Ref 1, p [208] & Ref 3) 

1-Diazo-2,5-dichlorobenzene or 1-Diazonium-hydroxide-2,5-dichlorobenzene, 
\( \text{Cl}_2\text{C}_6\text{H}_3\text{N}:\text{O):OH; known only in the form of its salts: Tribromide, Cl}_2\text{C}_6\text{H}_3\text{N}_2\text{Br}_3, \text{yel} \)
Nitroso & Nitro Derivatives of Dichlorobenzene

1-Nitroso-2,4-dichlorobenzene, C<sub>7</sub>H<sub>4</sub>Cl<sub>2</sub>N<sub>2</sub>O; mw 192.01, N 7.30%. Several isomers are described in Beil: 1-Nitroso-2,4-dichloro-, nor obtd pure; 1-Nitroso-2,5-dichloro-, col crystals, mp 101°; and 1-Nitroso-3,4-dichlorobenzene, mp 89°, dec on heating above it mp (Ref)

Mononitrodichlorobenzene, C<sub>7</sub>H<sub>4</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>2</sub>; 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] & 1615, 616, 617

Dinitrodichlorobenzene, C<sub>7</sub>H<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>; 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<sub>2</sub> 2-nitro-5,6-dichloroaniline (Ref 1)
1,2-Dinitro-3,5-dichlorobenzene, ndls (from alc + benz), mp 95-96°; obtd by nitrating with mixed acids 1-nitro-3,5-dichlorobenzene (Ref 2)
1,2-Dinitro-6,6-dichlorobenzene, ndls (from alc), mp 101-03°, bp 318°(dec), d 1.6915 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, crystals (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<sub>4</sub>), 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 crys (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 Fli 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<sub>3</sub> & concd H<sub>2</sub>SO<sub>4</sub> 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 Fli test)
1,4-Dinitro-2,3-dichlorobenzene, no props given (Ref 8)
1,4-Dinitro-2,5-dichlorobenzene, citron-yel crystals (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<sub>2</sub> soln at 50° (Ref 10)

Commercial Dinitrodichlorobenzene or Parazal. A mix 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 sensitivty - 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°C Heat test, 60 min; by 135°C German test, 120 min.

Its toxicity is similar to that of TNT but the comt 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 expil 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 expin contain phosphene (COCl₂)

and are very poisonous. Babcock (Ref 12) proposed for military use an expil filler composed of a dinitro-p- & dinitro-o-dichlorobenzenz with or w/o Feric Acid, thus producing a mixt castable below 100°C.


Trinitrodichlorobenzenz, C₁₂H₁₁(NCl₂)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°C; was prepd by heating 2,4,6-trinitro-3-chlorophenol with p-toluene-sulfonyl chloride & N,N-diethylenilin and by other methods (Refs 1 & 2)

This expl compd has not found any practical military application.


(5,7-Dichlorobenzonitrobenzotriazol-1-ol. See Vol 2, p B88-L, under Benzotriazol and Derivatives

5,6-Dichlorobenzotriazol-1-ol. See Vol 2, p B88-L, under Benzotriazol

Di(chlorobenzoyl) Peroxide, Bis(chlorobenzoyl)
Peroxide or Dichlorobenzoyl Peroxide (called Bis(chlor-benzoyl)-peroxyd or Dichlor-benzoyl-peroxyd in Ger),

CIC₆H₄CO.OOC₂C₂H₄Cl; mw 311.11, O20.57%. Three derivs are known:

Diochlorobenzoyl) Peroxide, crys (from chl + MeOH), mp 110-02°C dec; was prepd by oxidizing 2-chlorobenzoyl chloride with H₂O₂ in alkaline soln (Ref 3)

Di(m-chlorobenzoyl) Peroxide, crys (from hydrocarbon solvs), mp 123°C dec; was prepd by reaction 3-chlorobenzoyl chloride in dry toluene with an excess of ag Na₂O₂ (Ref 4)

Di(p-chlorobenzoyl) Peroxide, crys (from C₂H₅), mp 142°C dec; explodes in a steel bomb when heated to 180° giving 4-4′-dichlorodiphenyl, 4-chlorobenzoic acid, CO₂ & other products; more sensitive to impact & friction than

Dibenzoyl Peroxide; was prepd by reacting 4-chlorobenzoyl chloride in acetone with either 5% H₂O₂ or 25% NaOH below 6°C or a soln of Na₂O₂ in ice water (Refs 1 & 2)


Dichlorodiaminoethane, Cl₄NH.CH₂.CH₂.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-Holeite;

(O₂N)Cl₂.NH.CH₂.CH₂.NCl(O₂)₂; mw 218.99, N 25.58%; crys (from alc), mp 41-41.4°C; detonates violently when heated to 100°C; it decoup with gas formation in 10% NaOH soln;
reduced by NaHSO₃ soln to Ethylene Nitramine (O₂N.NH.CH₂.CH₂NH.NO₂); was prep'd when a 5% excess of N,N,N',N'-tetracloro-1,2-diaminoethane (Cl₂N.CH₂.CH₂N.Cl₂) and 99% HNO₃ were added in 30 mins to stirred acetic anhydride at 35°C, distilling the reaction mixt at 15 mm pressure, and chilling the oily residue at -75°C.

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 Difluorodichlomethan in Ger), CCIF₂; mw 120.93; liq, fr p -158°C, bp -29.8°C, liq d 1.311 at 25°C, liq nD 1.287 at 25°C; crit temp 112°C, crit press 40.6 atm, crit vol 1.792 ml/g; heat capacity 0.240 cal/g (°C) at 25°C; viscosity 0.26 cp at 25°C; dielectric constant 2.13 at 29°C; sol in water at 1 atm 0.028% by wt (Ref 2). Other props & methods of prep 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


Dichlorodimethyl ether. See item 0 in Vol 2, p C169-R

N,N'-Dichloro-N,N'-dinitro-1,2-diaminoethane. See under Dichlorodiethylenedioctane in this vol.

Dichloro-EDNA; Dichloro-Haleite. See N,N'-Dichloro-N,N'-dinitro-1,2-diaminoethane under Dichlorodiaminoethane

DICHLOOROETHANE AND DERIVATIVES

Dichloroethane, Ethylene Dichloride or Ethyldene Dichloride, C₂H₄Cl₂; mw 98.97. Two isomers are possible:
1,1-Dichloroethane, as-Dichloroethane, or Ethyldene Dichloride (called 1,1-Dichloroethane or Ethyldenechlorid in Ger), CH₂-CHCl₂, col liq having a smell & taste resembling chloroform, fr p -97.6°C, bp 57.3°C, flash p -8.5°C, d 1.1601 at 30°C, np 1.41658 at 20°C, Qcomb 267.4 kcal/mol at C₈; 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 A1, 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-Dichloroethane or Athyldenechlorid in Ger), CH₂Cl-CH₂Cl, col liq with an odor resembling that of chloroform, turns yellow on long exposure to sunlight, fr p -35.4°C, bp 83.5°C, flash p 17°C (CC) & 21°C (OC), d 1.2383 at 30°C, np 1.4449 at 20°C, Qcomb 296.4 kcal/mol at C₈; expl limits in air 6.2-16.9% by vol; its toxicity over short periods is less than that of chloroform, but the fatal dose is less than that of chloroform; 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 0 ppm; can be manufd by reacting Cl₂ 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)
Reps: 1) Beil 1, 83, (23), [52] & [139]
2) Beil 1, 84, (24), [52] & [141] 3) Kirk & Othmer 5, 2nd edit (1964), 148-54

Azido, C2H5Cl2N3, and Diazido, C2H2Cl2N6, derivs of Dichloroethane were not found in Beil or in CA thru 1961

Nitro- & Nitrosoderivatives of Dichloroethane

1-Nitroso-1,1-dichloroethane, CH3.CCl2(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, C2H3Cl2NO2; mw 143.96, N 9.73%. The following isomers are known:

1-Nitro-1,1-dichloroethane, CH3.CCl2(NO2), liq, bp 120-21° at 756 mm, flash p 57.8°, d 1.4250-1.4271 at 20° (Ref)
1-Nitro-1,2-dichloroethane, CH2Cl.CHCl(NO2), 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°, nD 1.4765 at 15° (Ref)

Other props & methods of prepn are given in Beil
Ref: Beil 1, [202]

Dinitrodichloroethane, C2H2Cl2N2O4; and Trinitrochloroethane, CH2Cl2N3O6; 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-åthan in Ger), (O2N)2Cl.CCl(NO2)2; mw 278.96, N 20.09%; snow-wh amor powd, sublimes at RT to form star-shaped crysfts, mp - begins to decompt at 60-65° with evolution of N oxides, melts sharply at 107°; volatile in steam with partial decompt; very sol in org solvs with decompt; slowly converted by boiling w into CO2, oxides of N, HCl & dichloro-dinitromethane; forms with KOH or KI, Dipotassium tetranitroethane, a yel crys 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 (Reps 1 & 2)

Expl props of parent compd were not reported
Reps: 1) Beil 1, [70] 2) L. Hunter, JChemSoc 125, 1482(1924) & CA 18, 3168(1924)

N,N-Dichloroethylamine or Ethylidichloroamine, C2H5.NCl2, 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 monoalkylamino hydrochloride with CaOCl2. 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)
Reps: 1) Beilstein 4, 127, (358), [616] & [1230] 2) J. Tscherniak, Ber 9, 143-150 (1876) & 32, 3582(1900) 3) J.D. Riedel, GerP 301799 (1914) & CA 15, 1996(1921)

Dichloroethylene, C2H2Cl2; mw 96.95. Three isomers exist:

1,1-Dichloroethylene, Vinylidene Chloride or 2,2-Dichloroethylene (called 1,1-Dichloroäthylene or Vinylidenchlorid in Ger), CH2CCl2; 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°, np 1.4249 at 20°; explosive limits in air 5.6-13% by vol; can be produced by dehydrochlorination of 1,1,2-trichloroethylene by agitating with an aq suspension of Ca(OH)2 (Reps 1 & 4, p 178). It is used in the manuf of vinyl compds & for the production of 1,1,1-trichloroethane (Reps 4)

1,2-Dichloroethylene; Acetylene Dichloride; Dioform or s-Dichloroethylene (called 1,2-Dichlor-äthylene or Acetylenedichlorid in Ger), CHCl:CHCl; known in trans & cis forms which are colorless, mobile liquids with a sweetish, sl irritant odor resembling that of chlf (Reps 2 & 4, p 180)
Trans form: CHCl

\[ \text{ClHC} \text{H, fr p -49.4}^\circ, \text{bp 47.7}^\circ, \]
d 1.2631 at 10\(^\circ\), \(n_\rho \) 1.4662 at 20\(^\circ\); forms a
ternary azeotrope contg ethanol 1.4 & water
1.1% boiling at 44.4\(^\circ\)C

Cis form: CHCl

\[ \text{CHCl, fr p -81.5}^\circ, \text{bp 60.2}^\circ, \]
d 1.2917, \(n_\rho \) 1.44900 at 20\(^\circ\); forms a ternary
azeotrope contg ethanol 6.65 & water 2.85%
which boils at 53.8\(^\circ\)

The tech product contains varying pro-
portions of trans & cis isomers depending
upon the conditions of manuf. A typical tech
product boils in the range 45-60\(^\circ\), has a flash,
p of 6\(^\circ\), and expln limits in air 9.7-12.8%
by vol. This compd burns with diffc but it
forms expn 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] \& 16471
2) Beil, 1, 187, (78), [159] \& 16511 3) Naoum,
NG (1928), 447 4) Kirk \& Othmer, 2nd edit
5(1964), 178, 180 5) ChemikerZtg 88, No 10
(1964), 175 Cited by Dr A. Langhans in
Explosivst 1964, 246 (Reports Dichloroethy-
lene to be combustible)

Dichloroethylsulfide. See item H (or HS) in
Vol 2, p C168-L

Dichloro-1-Halo. See N,N'-Dichloro-N,N'-
Dinitro-1,2-dicarbamoethane under Dichloro-
diaminoethane in this Vol

**DICHLOROHYDRIN**

**AND DERIVATIVES**

Dichlorohydin or Glycerol Dichlorohydin,
C\(_3\)H\(_5\)Cl\(_2\)O; mw 128.99; exists in two isomers,
both of which are present in the commercial
product:

a,\(\beta\)-(or 2,3)Dichlorohydin or 2,3-Dichloro-
1-propanol [called 2,3 Dichloro-propanol (1);
2,3-Dichloro-1-propylalkohol; Glycerin-1,2-
dichlorhydrin; or \(\beta\)-Dichlorhydrin in Ger],
C\(_2\)H\(_2\)O:CHCl.CH\(_2\)OH; col sl viscous liq, bp
182-85\(^\circ\), d 1.3607-1.3616 at 20\(^\circ\), \(n_\rho \) 1.4819-
1.4849 at 20\(^\circ\); Qcomb 3184 cal/g at C\(_V\), flash
p 195\(^\circ\)F (OC); miscible with org solvs;
also forms azeotropes (Refs 1 & 4). For
prep, toxicity \& uses, see below under
Commercial Dichlorohydin

a,\(\gamma\)-(or 1,3)Dichlorohydin or 1,3-Dichloro-
2-propanol [called 1,3-Dichloro-propanol (2);
\(\beta,\beta\)-Dichloro-isopropylalkohol; Glycerin-1,3-
dichlorhydrin; or \(\alpha\)-Dichlorhydrin],
C\(_2\)H\(_2\)Cl.CH(OH).CH\(_2\)Cl; col sl viscous liq
having a sweet taste, bp 175\(^\circ\), d 1.3645 at 20\(^\circ\),
\(n_\rho \) 1.48375 at 20\(^\circ\), Qcomb 3151 cal/g; solubili-
ity in water 15.6% by wt; miscible with al-
cohols, acet, benz \& eth; not miscible with
petr eth; forms azeotropes with \& some
org solvs (Refs 2 \& 4). For prep, toxicity
\& uses, see below under Commercial Di-
chlorohydin

**Commercial Dichlorohydin** consists of the
above two isomers, the proportions of which
depend on method of prep; d 1.36-1.39,
bp 175-80\(^\circ\), flash p 74\(^\circ\). Glycerol was the
main source for the prep of glycerol chloro-
hydrins until the process for direct substitu-
tive chlorination of propylene to allyl
chlordie paved the way for synthesis by
chlorohydration 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\(^\circ\) to yield 90% of prod-
uct which is mainly the a,\(\gamma\)-form. Synth-
esis from propylene yields a mixt of approx
70% a,\(\beta\)-form \& 30% a,\(\gamma\)-form. Addn of HCl
to epichlorohydin, CH\(_2\)OH\(_2\)Cl, at

\[ -10^\circ \text{ yields 99.6\% a,\(\gamma\)-dichlorohydin \& 0.4\% a,\(\beta\)-form. The a,\(\beta\)-form may be obtd in
92.5\% yield by chlorination of acrolein,
followed by reduction, or by chlorination of
allyl alcohol (Ref 5). Naoum (Ref 4)
states that a mixture of two dichlorohydins,
both bp 174\(^\circ\) \& asyn bp 182-183\(^\circ\), has been
used to prep commercial dichlorohydin 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 exps, since it is less volatile than some other solv (such as ether, acetone, ethyl or anyl 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).


Nitrite- & Nitrate Derivatives of Dichlorohydrin

2,3-Dichlorohydrin-1-nitrite (called 2,3-Dichloro-propyl-nitrat in Ger),
$\text{CII}_2\text{C.CII}\text{Cl.CH}_2\text{O.NO}_2$; mw 141.99, N 9.86%;
lyr lachrymatory liq, bp 55° at 14 mm press, d 1.323 at 22°, np 1.456 at 16°, was prep'd by reacting 2,3-dichloro-1-propanol with $\text{NaNO}_2$ & dil HCl.

Ref: Beil 1, [1427]

2,3-Dichlorohydrin-1-nitrate (called $\beta_\gamma$-Dichloropropyl-nitrat in Ger),
$\text{CII}_2\text{C.CII}\text{Cl.CH}_2\text{O.NO}_3$; mw 173.99, N 8.05%;
lyr, bp 180°, d 1.3 at7°; was prep'd by nitratmg 2,3-dichlorohydrin with $\text{HNO}_3$.

Ref: Beil 1, 356

1,3-Dichlorohydrin-2-nitrate (called ($\beta_\beta$-Dichloro-isopropyl)-nitrat in Ger.),
$\text{CII}_2\text{C.CII}\text{CH(O.NO}_2$).$\text{CH}_2\text{Cl}$; mw 173.99, N 8.05%;
col very mobile liq, having an aromatic odor, fp p below -20°, bp 180-90° (dec), d 1.459 at 15°; was prep'd by nitratmg 1,3-dichlorohydrin with mixed acids (Ref 1). Naoum (Ref 2) reports some addnl props & 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 spcicy 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 Chlorogydin Dinitrate. Naoum (Ref) recommends nitration by introducing, with cooling, dichlorohydrin into a mixt of $\text{HNO}_3$ (60%) & $\text{H}_2\text{SO}_4$ (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 decmps according to one of the following equations:

$2\text{C}_2\text{H}_5\text{Cl}_2(\text{ONO}_2) \rightarrow 6\text{CO} + 4\text{HCl} + 3\text{H}_2\text{O} + 2\text{N}_2$

$\rightarrow 4\text{CO} + 2\text{H}_2\text{O} + 4\text{HCl} + 2\text{C} + \text{H}_2\text{O} + 2\text{N}_2$

The heat of combustion for first equation is 259 cal/g and for second 440 cal/g at $C_0$ with $\text{H}_2\text{O}$ vapor. Heat of formation is 81.9 kcal/mole.

It has been recommended that coml Dichlorohydrin Nitrate be used as an antifreeze agent to dynamites as a substitute for Chlorohydrin Dinitrate. According to Naoum (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 gelatized expls.

Ref: Naoum, NG (1928), 191-93

NOTE: No higher nitratd 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 Stolle & Dietrich];

(CIN)$_2$C$\equiv$N$\equiv$N

HN——C(NCl); mw 165.98, N 42.20%; yel amorphous powd; mp detonates violently
1,4-Di(chloroimino)-p-quinone. See Benzo-quinone-1,4-di(chloroimine) in Vol 2, p BB3-L

4,5-Dichloroindazole-6,7-quinone [called 4,5-Dichlor-indazol-6,7] in Ger],

\[ \text{Cl}_2\text{C} \equiv \text{C}(-\text{Cl}) \equiv -\text{C}(-\text{CH}) \equiv \text{N} \]

or

\[ \text{Cl}_2\text{C} \equiv \text{C}(-\text{Cl}) \equiv -\text{C}(-\text{CH}) \equiv \text{N} \]

\[ \text{O}(-\text{C}) \equiv -\text{C}(-\text{NH}) \equiv \text{N} \]

\[ \text{O}(-\text{C}) \equiv -\text{C}(-\text{NH}) \equiv \text{N} \]

mw 217.02, N 12.94%; red lfts (from gluc acet ac + HNO₃); mp - defagr above 350°; sol in cold dil caustic soda soln; sl sol in acet & alc; v sl sol in benz & benzene; was prepd by heating 4,5,7-trichloro-6-hydroxyindazole with concd HNO₃ in gluc 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, [I92] 2) K. Fries, Ann 454, 315, 319 (1927) & CA 21, 2693 (1927)

Dichloroisonicotinyl 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), CH₂Cl₂, colorless, mobile liq having a pleasant ethereal odor, fr p -96.7°, bp 40.4°, d 1.326 at 20°, nD 1.4246 at 20° (liq), Qcomb 144.5 kcal/mol; expl limits in O₂ 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 CCl₄. 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 ed 5 (1964), 111ff 3) US Specification MIL-D-6998B (June 1967) Mononitrodichloromethane, CHCl₂NO₂; 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 (O₂N.CH₂.COOK) (Refs 1 & 2)

Refs: 1) Beil 1, [I13] 2) W. Steinkopf & M. Kühnel, Ber 75B, 1939 (1942) & CA 37, 4687 (1943)

Dinitrodichloromethane, CCl₂(NO₂)₂; mw 174.94, N 16.02%; colorless oil, bp 46° at 20 mm, d 1.6124 at 20°, nD 1.4575 at 20°; sol in alc, eth, chlf, benz & CS₂; insol in w; can be prepd by heating 2,4,6-trichloroaniline or trichloroethylene with concd HNO₃, and by other methods (Ref 1). See also Refts 2 & 3

Its expl props were not investigated


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

Dichloroisonicotinyl Azide. See 2,6-Dichloropyridine-4-carboxylic Acid Azide in this vol

DICHLOOROPHENOL AND DERIVATIVES

Dichlorophenol or Hydroxydichlorobenzene (called Dichlor-phenol or Dichlor-hydroxy-
benzol in Ger), C₆H₅Cl₂-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, crystals (from benz), mp 59°, bp 209-11°, volatile with steam; of no coml importance (Refs 3 & 6)

2,6-Dichlorophenol, crystals (from petr eth), mp 66-68°, bp 220°, volatile with steam; no coml applicarion (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, crystals (from petr eth), mp 68°, bp 233° at 757 mm press; can be nitrated & brominated to produce interesting derivs, but its high starting cost has precluded coml develop (Refs 5 & 6)

Other props & methods of prep are given in the Refs


Nitroderivatives of Dichlorophenol

Mononitrodichlorophenol, O₂N.C₆H₅Cl₂.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), prisms (from eth), mp 121-24°, sublimes below 100°, deflg on rapid heating, d 1.822 at 19°; sol in alc, eth, chlf & benz; sl sol in w; its Ammonium salt, H₄N.C₆H₅Cl₂O₂N₃, on-red ndls, sublimes at 100° & deflg 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 glc acet ac), plts (from eth) & crys (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 prep are given in the Refs


Dinitrodichlorophenol, (O₂N)₂C₆H₅Cl₂.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 HNO₃ in the cold (Refs 3 & 5) and by other methods (Ref 6)


2,4,6-Trinitro-3,5-dichlorophenol (called 3,5-Dichloro-2,4,6-trinitro-phenol or Dichloropikrinsäure in Ger),
O₂N.C—C(OH) = C.NO₂

Cl.C—C(NO₂) = [Cl.C; mw 298.00, N 14.10%;
lt.-yel prisms (from glc acet ac), mp 139-40°;
or col crys (from chl or CCl₄), mp 135°, bp -
explodes; readily sol in alc & eth; sol in hot
w; v sl sol in petr eth; obtbd when 3,5-dichloro-
phenol in glc acet ac was treated with red
fuming HNO₃ 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. Will-
stätter & 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 Azidodichloro-
benzene. See under Azido & Diazo Deriva-
tives of Dichlorobenzene in this vol

Dichlorophenyl-azetetrazole. See Bis(chloro-
phenyl)-azetetrazole in Vol 2, p B139-L

Dichloropicolinyl Azide. See 4,6-Dichloro-
pyridine-4-carboxylic Acid Azide

2,6-Dichloropyridine-4-carboxylic Acid Azide
or 2,6-Dichloro-isonicotinyl Azide (called
2,6-Dichloro-isonicotinsäure-azid or 2,6-
Dichloropyridin-carbonsäure-azid-(4), in Ger),
Cl.C — N — C.C1

HC—C(COON₂) = CH ; mw 217.02, N 27.04%;
crys, mp 89°; defgr very weakly on heating
in a free flame; was prepbd by treating its hydra-
zide, NC₅H₂Cl₂.CO.NH.NH₂ (mp 184°) with
NaNO₂ in sq 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-Dichloropicolinyl Azide,

Cl.C — N — C.COON₃

HC—C(C1) = CH ; mw 217.02, N 27.04%;
crys, mp 74°; was prepbd in a manner simi-
lar to that of the 2,6-Dichloro deriv (Ref 2)
Refs: 1) Beil-not found 2) R. Graf, JPrakt-
Chem 133, 36 (1932) & CA 26, 1933 (1932)

2,6-Dichlorquinonechlorimide [called 2,6-
Dichloro-chinon-chlorimid-(4) in Ger],
Cl.C — C(O) — C.C1

HC—C(NCl) = CH ; mw 210.46; yel ndls
(from alc), mp 67-68°; was prepbd from the
HCl salt of 2,6-dichloro-4-aminophenol by treat-
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.CH₂.N(NO₂) CH₂.N(NO₂).CH.N(NO₂).CH₂Cl;
mw 314.02, N 31.23%; fine crys (from benz),
mp 144.5-45.5°, bp - explodes; was prepbd by
saturating with anhyd HCl at 10° a soln of
1-acetoxy-7-trifluoroacetoxy-2,4,6-trinitro-
2,4,6-triazaheptane. GSX also was prepbd from
HCl & the diacetate of 1,7-diacetoxy-2,4,6-
trinitro-2,4,6-triazaheptane; 1,7-bis-trifluoro-
acetoxy-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 prepns. 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),
(C₅H₅.CH:CH.CO.O-)_2; mw 294.29, O 21.75%;
col ndls (from acet or alc-acet), mp 133°, 144°,
deflg on heating in a flame; was prep'd by
slowly addg cinnamoyl chloride in acet to a
sl excess of alkaine H₂O₂ (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. D.-
korsch, Monatsch 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_combsta)
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, 6835 (1956)
10) I.V. Sukmansky & A.I. Yurzenko,
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
HC=CH—C—CH  HC—C—CH=CH
HC=CH—C—CO.OO.CO—C—CH=CH

Dicuminoxyo Peroxide (called Dicuminoxyperoxyd in Ger),
[[CH₃₂CH]C₆H₄.C.O₂H₂O₂; mw 326.38,
O 19.61%; ndls (from eth), mp - explodes on
heating; was prep'd 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(a,a-dimethylbenzyl)
Peroxide, [C₆H₅.C(CH₃)₂O]-; mw 270.36,
O 11.84% ; wh crysts (from MeOH), mp 39°,
bp - decomp rapidly at 120°, np 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 decomp of acetyl peroxide in a soln of the hydroperoxide & cumene (Ref 2)

Its mononitro deriv, Bis(a,a-dimethyl-p-nitrobenzyl Peroxide, [P-O3N.C6H4.C(NO3)2O]-2, crysts(from alc), mp 158°(decomp), was prep'd by Hock & Kropf (Ref 4)


Dicuprocetaldehyde. See Vol 1, p A73-L

Dicy. One of the abbreviations for Dicyanamide

Dicyanamide (called Dicyanamid, Dicyanimid or lmino-dicarbonsäure-dinitril in Ger), NC.NH.CN; mw 67.05, N 62.67%; ndls + H2O; obtd by reacting chloramine with an aq soln of KCN and by other methods; forms some unstable salts such as:

Ammonium salt, NH4C2N3, ndls (from alc + eth), mp 126°, decomp on heating above 126°

Copper salt, Cu(C2N3)2, blue-grn ndls, deflgr on heating

Lead salt, cryvst, decomp on rapid heating

Mercury salt, Hg(C2N3)2, cryvpt, deflgr on heating to 110° and increases in vol like a Pharaoh's serpent, emitting a squeaky noise

Other props are given in Beil

Ref: Beil 3, 82, [68] & [154]

Dicyanomidaeoxide. See Cyanofomimidine 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, Armour Res

- Foundation Rept 3200-6(1963)

Dicyandiamide. See Cyanoguanidine in Vol 3, p C587-L


DICYANDIAMIDINE

AND DERIVATIVES

Dicyandiamidine, Guany lurea or Amidinourea

( CA name ) (called Dicyandiamidin, Guanyl-
harnstoff, Carbamoylguanidin or Guadinid-
carbonsäure-amid in Ger),

H2N.C( :NH).NH.CO.NH2; mw 102.10, N 54.88%;

crysts (from alc), mp 104-105.5°, bp dec 160°;

v soln in w, warm alc & pyridine; insol in benz,
chlf, eth & CS2; can be prep’d by heating di-
cyandiamide with aq NH3 at 150°, and by other

methods (Ref 1). Its admixtures with oxidiz-
ing agents were recommended as powerful
expl (Ref 3)

Dicyandiamidine forms numerous cryvst

salts, addn compds & a nitrocompd.

Its Perchlorate salt, C2H6N4.O + HClO4,

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 im-

pact. It has been proposed for use in military

& commercial expls (Ref 2): a) Perchlorate

69, NaN03 29 & woodmeal 2% b) Perchlor-

ate 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

Ref's: 1) Beil 3, 89, (42), [74] & [164]

2) W. Rintoul & E. Beckett, BritP 14706(1915)

& CA 13, 1640 (1919) ; USP 129879(1915) &

CA 13, 1766 (1919) 3) C. Manelli & B. Luigi,

ItalP 49536(1919) & CA 14, 2555 (1920)

4) Blatt, ORSD 2014(1944)

Nitro dicicyandiamidine (called N1-Nitro-N-
guanyl-harnstoff or Nitro dicicyandiamidin in Ger)

H2N.C( :NH).NH.CO.NHNO2; mw 147.10, N
47.61%; fine crystals, mp - deflgr on heating; Q^v_{comb} 285.82 kcal/mol & Q^v_{form} 71.96 kcal/mol (Ref 4); sol in NH_3 & alcalies; 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 exps (Ref 2). Its Silver salt, AgC_4H_6N_5O_3, amor ppt, deflgr on heating


Dicyandiamidine Perchlorate. See under Dicyandiamide in this vol

Dicyandiaze (called Dimers Cyanazid; "Diazido-cyanimino-methan"; or "Dicyandiaze" in Ger), NC.N:C(N)_2,O or (NC.N):_2; mw 136.08, N 82.35%; col ndls, mp - decmp at 127° and explodes violently at 143°; also explodes violently on sl friction or shock; readily sol in alc, eth & acet; sol in sl; v sl sol in benz; insol in petr eth; was obtd when 3 mols NaN_3 & 1 mol dibrominalonitride reacted in cold water & ether, and the so in acidified. Ag nitrate ppts from the soin 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'-Dicyandicyclohexyl in Ger), NC.C_6H_{10}.C_6H_{10}.CN; mw 216.32, N 12.95%; wh crystals (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 moire) may result in an expl


1,2-Dicyanoethene or Succinonitrile (called Bernstein-säure-dinitril; Succinonitril; Äthylen cyanid; 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 brissant than PA


Dicyanomethane or Malononitrile (called Malonsäure-dinitril, Malonitril or Methylen cyanid in Ger), NC.CH_2.CN; mw 66.06, N 42.41%; col cryts, mp 31.6-32.4°, bp 108-109° at 17 mm press, cryst d 1.191 at 20°, liq d 1.049 at 35°, nD 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 phthalochlor, gives Dicyanomethane (Ref 1).

Blatt (Ref 2) reports this compd (under NITRILES) to be an expl less powerful & less brissant than PA


Dicyano(methyl)amine (called Bis-cyanmethylanin, Iminodiessigsäure-dinitril, Iminodi acetonitril or Diglykolamidsäure-dinitril in Ger), HN(CH_2.CN)_2; mw 95.10, N 44.19%;
crystals (from w), plts (from alc or eth), mp 75-77°; can be nitrated to form an expl deriv (Ref 1)

**Di(cyanomethyl)nitramine**, O₃N.N(CH₂-CN)₂; mw 140.10, N 39.99%; crystals (from acet + w), ndls (from anhyd bena), mp 96-97°; sl sol in benz, alc, eth & ethyl acet; v sl sol in w & chl; insol in petr eth; prepd by nitrating Di(cyanomethyl)amine with 99% HNO₃ & acetic anhydride at 10-15°(Ref 2)

This exp 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, ORSD 2014(1944); 4) ADL. PureExplComps, Part 1 (1947), p 64

**4,5-Dicyano-1,2,3-triazole** (called 4,5-Dicyan-1,2,3-triazol in Ger),
NC.C—N==N || NC.C—N==NH
|| NC.C—NH or NC.C==N

mw 119.09, N 58.81%. Exists in two modifications;
- Yellow form, yell crystalls (from w), mp 145-50° (dec); readily sol in alc & eth; insol in ligroin & petr eth; was prepd by reacting H₂N.CH(CN).C(CN):NH or H₂N.C(CN):C(CN).NH₂ (diamo-maleonitrile or tetramer hydrocyanic acid) with HNO₂
- Colorless form, crystalls (from w), mp 145-50° (dec); readily sol in alc & eth; insol in ligroin; obtd on sublimation of the yell form at 140°.

This comp forms several crystall salts; its Silver salt, Ag₄C₄N₅, defigr on heating (Refs 1 & 2).

Refs: 1) Beil 26, [160]; 2) E. Griszkiewicz-Trochimowski, RocznChem 1, 458-78(1921) & CA 17, 1425 (1923)

**Di(cyclohexonecarbonyl)-peroxide.** See Bis(hexahydrobenzoyl)-peroxide in Vol 2, p B143-L

**1,8-Dicyclohexylamino-3,6-diazaooctane**, C₁₈H₄₂N₄C₁₂; crystalls (from w), mp 299.4-7°; isolated as the tetrahydrochloride, is the parent comp of its nitro deriv:

**1,8-Dicyclohexylamino-3,6-dinitro-3,6-diazo-octane**, C₂₁H₁₁[N(NO₂).CH₂.CH₂.N(NO₂)]C₆H₁₁; mw 484.47, N 23.13%; crystalls (from boiling HNO₃, 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% HNO₃, 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,
C₆H₁₀.NH.CH₂.CH₂.NH.C₆H₁₀, is the parent compd of its nitrated derivs:

**N-Nitroso-N'-nitroso-N,N'-dicyclohexyl-ethylenediamine or N,N'-Dicyclohexyl-1,2-dinitroso-o-methane,** C₆H₁₁.N(NO₂).CH₂.CH₂.N(NO₂).C₆H₁₁; mw 282.38, N 19.84%; crystalls (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% HNO₃ & NaNO₂ at 25° (Ref 2)

**N-Nitro-N'-nitroso-N,N'-dicyclohexyl-ethylenediamine or N,N'-Dicyclohexyl-N-nitro-N'-nitroso-1,2-diaminoethane,** C₆H₁₁.N(NO₂).CH₂.CH₂.N(NO₂).C₆H₁₁; mw 298.38, N 18.78%; crystalls (from benz or alc), mp 148-149.9°; was prepd by treating N,N'-dicyclohexyl-N-nitro-1,2-diaminoethane-mononitrate suspended in aq NaNO₂ with chloroform & acetic acid, or by treating a slurry of dicyclohexyl-imidazolide dinitrate with abs HNO₃ & acetic anhydride (Ref 3)

**N,N'-Dinitro-N,N'-dicyclohexyl-ethylenediamine or N,N'-Dicyclohexyl-1,2-dinitroso-o-methane,** C₆H₁₁(NO₂).CH₂.CH₂.N(NO₂).C₆H₁₁; mw 314.38, N 17.82%; crystalls (from 70% HNO₃.
then isopropy ether, and pyridine or chlf, chlf-petp eth or benz-petr eth), mp 212-13°; was obtd by nitrating N,N'-dicyclohexyl-1,2-diaminoethane dihydrochloride with 99% HNO₃ at 0° & acetic anhydride in acetic acid
(Refs 2 & 3)

The expl props of the nitrated deriv of Dicyclohexyl-ethylenediamine were not investigated

1,3-Di(cyclohexyl)-imidazolidine,

\[
\begin{align*}
\text{C}_6\text{H}_{11}.\text{N}.\text{CH}_2.\text{CH}_2.\text{N}.\text{C}_6\text{H}_{11} & \quad \text{mw 236.39,} \\
\text{N} & \quad 11.85\%; \text{ liq, fr p 13-15°, b p 125-26° at} \\
0.5 \text{ mm} & \quad 137-38° \text{ at 0.7 mm press; was obtd by evaporating to dryness under reduced} \\
\text{press a soln of N,N'-dicyclohexyl-1,2} & \quad \text{diaminoethane, its dihydrochloride & formaldehyde in abs alc; forms a dinitrate;} \\
1,3-Di(cyclohexyl)-imidazolidine Dinitrate, & \quad \text{O}_3\text{NH}-\text{C}_6\text{H}_{11}.\text{N}.\text{CH}_2.\text{CH}_2.\text{N}.\text{C}_6\text{H}_{11}\text{HNO}_3 \\
& \quad \text{CH}_2 & \quad \text{mw 362.42, N} \\
15.46\%; \text{ wh ppt, mp 143-44° & after 2 hrs} & \quad 147-48°; \text{ was obtd by chilling its parent} \\
\text{compd in abs alc at -60° & adding cold abs} & \quad \text{HNO}_3 \text{ (Ref 2); decom in presence of moisture} \\
to the dinitrate salt of N,N'-Dicyclohexyl- & \quad \text{to the dinitrate salt of N,N'-Dicyclohexyl-} \\
1,2-diaminoethane & \quad 1,2-diaminoethane} \\

Its expl props were not reported

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, [(N₃)₂Cu(C₇H₉N₂)₂]; mw 361.88, N 30.96%; explodes at 202-203° but not by impact; can be prepd by dissolving Cu(N₃)₂ in the appropriate amine; by addg Na₃N to an amine soln of the appropriate Cu salt; and by rearranging of the cuprate of the appropriate amine
(Ref 2)

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 sub stricted 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. Montijo, 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 PicArsn (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-ohmmeter 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

<table>
<thead>
<tr>
<th>EXPLOSIVE</th>
<th>RESISTIVITY&lt;sup&gt;a&lt;/sup&gt; ohm-cm</th>
<th>PELLET&lt;sup&gt;g&lt;/sup&gt; THICKNESS INCHES</th>
<th>DIELECTRIC STRENGTH BREAKDOWN VOLTAGE, KV</th>
<th>VOLTS/ mill</th>
<th>DIELECTRIC CONSTANT 100C</th>
<th>100KC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Batatol</td>
<td>1.79 x 10&lt;sup&gt;11&lt;/sup&gt;</td>
<td>0.380</td>
<td>5.5</td>
<td>145</td>
<td>3.543</td>
<td>3.471</td>
</tr>
<tr>
<td>Composition B&lt;sup&gt;c&lt;/sup&gt;</td>
<td>3.59 x 10&lt;sup&gt;10&lt;/sup&gt;</td>
<td>0.125</td>
<td>23</td>
<td>182</td>
<td>3.562&lt;sup&gt;d&lt;/sup&gt;</td>
<td>3.297&lt;sup&gt;d&lt;/sup&gt;</td>
</tr>
<tr>
<td>Octol(75/25-HMX/TNT)</td>
<td>2.99 x 10&lt;sup&gt;10&lt;/sup&gt;</td>
<td>0.040</td>
<td>5.5</td>
<td>139</td>
<td>3.121</td>
<td>3.013</td>
</tr>
<tr>
<td>PBX&lt;sup&gt;e&lt;/sup&gt;</td>
<td>∞</td>
<td>0.0385</td>
<td>32</td>
<td>831</td>
<td>2.523</td>
<td>2.517</td>
</tr>
<tr>
<td>PBX-9404&lt;sup&gt;f&lt;/sup&gt;</td>
<td>4.19 x 10&lt;sup&gt;-10&lt;/sup&gt;</td>
<td>0.0385</td>
<td>22</td>
<td>560</td>
<td>3.679</td>
<td>3.414</td>
</tr>
<tr>
<td>TNT</td>
<td>8.97 x 10&lt;sup&gt;-10&lt;/sup&gt;</td>
<td>0.040</td>
<td>3.8</td>
<td>94</td>
<td>5.167</td>
<td>2.849</td>
</tr>
</tbody>
</table>

<sup>a</sup> Measurements made on machined pellets 3/4 x 3/4 inch
<sup>b</sup> 76/24/0.06/0.1 - Ba(NO<sub>3</sub>)<sub>2</sub>/TNT/stearoyiacetic acid/NC
<sup>c</sup> 59.5/39.5/1 - RDX/TNT/wax
<sup>d</sup> Measurements made on pellets 0.125 inch
<sup>e</sup> 91/8.5/1.5 - RDX/dioctylphthalate/poly styrene
<sup>f</sup> 94/3/3 - HMX/NC/tris β-chloroethyl phosphate
<sup>g</sup> 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 0.3", 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 kilicycle, and a Hewlett Packard Model 650A test oscillator plus a HP Model decade amplifier for higher frequencies. For the Composition B samples, a Keithley 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)

**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 10⁻⁵ 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
2. Kirk & Othmer 11, 2nd ed (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.
Dienite. 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 Azo-phenol in Vol 1, pA656-R
Diethoxyazoxybenzene and Its Trinitro-Derivative. See under Azoxyphenetole in Vol 1, p A670-R

1,9-Diethoxypentamethylen-2,4,6,8-tetranitramine; 3,13-Di-o x-o-5,7,9,11-tetranitro-5,7,9,11-tetrazazapentadecane (CA nomenclature); or 1,9-Diethoxy-2,4,6,8-tetranitro-2,4,6,8-tetrazazanonane (called by McKay et al) (Refs 1 & 2)

C₂H₅.O.CH₂.N(NO₂).CH₂.N(NO₂).CH₂.N(NO₂).CH₂.OCH₂.H₃; mw 400.31, N 27.98%; wh crs (fractionally crsld), mp 160-167°; was obtd by refluxing for 10 hrs 1,9-dinitroxy pentamethylen-2,4,6,8-tetranitramine (qv) with 95% alc, then cooling, filtering & fractionally crystallizing the product


1,7-Diethoxy-2,4,6-trinitro-2,4,6-triazahexe or 3,11-Dio xo-5,7,9-trinitro-5,7,9-triazatridecane (CA nomenclature), C₂H₅.O.CH₂.N(NO₂).CH₂.N(NO₂).CH₂.N(NO₂).CH₂.OCH₂.H₃; mw 338.28, N 24.85%; crs (from alc), mp 79-80.5° & 82° (Ref 2); insol in w; sol in alc & eth; obd by refluxing 1,7-dinitroxy-2,4,6-trinitro-2,4,6-triazahexe (qv) with 99% ethanol for 25 min and diluting with water. Its expl props were not reported


Nitroso- and Nitroderivatives of Diethyamine

N-Nitroso-diethyamine, (C₂H₅)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°, p₃ 1.4379 at 20°; other props & methods of prep are given in Beil

Ref: Beil 4, 129, (358), 6171 & 1233

N-Nitro-diethyamine, (C₂H₅)₂N.NO₂; 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° diethyamine nitrate with 100% HNO₃ in acetic anhydride in the presence of ZnCl₂

Ref: Beil 4, 130 & 1233

Di(2,2,2-trinitroethyl)amine; Bis(2,2,2-trinitroethyl)amine or 2,2,2,2',2',2'-Hexanitrodiethyamine (HeNDEt₂A), [(O₂N)₃C.CH₂]₂NH; mw 343.14, N 28.58%; nls (from chl), mp 107° & 116° (Ref 3); can be prepd by treating 2 mol 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 NH₃ (Refs 2 & 3);

2HCHO + 2(O₂N)₃CH + NH₃ = 2H₂O + [(O₂N)₃C.CH₂]₂NH This compd might be suitable as an ingredient of expl or propelnt compns. Ville (Ref 3) reported it to be sensitive to impact and capable of violent & spontaneous expln


Di(2,2,2-trinitroethyl)-nitramine; Bis(2,2,2-trinitroethyl)nitramine (BTNEN); or N₂N₂N₂, 2,2',2'-Heptanitrodiethyamine, Code name HOX, [(O₂N)₃C.CH₂]₂N.NO₂; mw 388.14,
N 28.85%; wh crstys (from chl), mp 94-96°; decomposes rapidly above its mp, d 1.91 at 20°; was first synthesized by German scientists (Ref 2); can be prepared by reacting tetranitromethane with NH₃ in the presence of H₂O₂; the resulting ammonia nitroform is then treated with formaldehyde (Ref 3).

This expl has an impact sensitivity value of 12-15 cm for 50% explosion with 2 kg wet; heat of combustion 1355 kcal/g; ignition temp 200° for 5 sec value; and rate detonation 7180 m/sec at d 1.5 & 8520 m/sec at d 1.9 g/cc (Ref 3). BNTEK is reported to be more powerful & more brisant than TNT. It was recommended for use in mixtures with AI (Ref 2).

Refs: 1) Beilnot found 2) G. Römer, "Report on Explosives", PBL Rep 85160 (1945) 3) D.L. Koub & R.H. Saunders, "Summary Report, Task A, High Explosives", Hercules Powder Co, Navy Contract NORD-11290 (Dec 1951) 4) J.A. Young et al, IEC 48, 1375-78 (1956) & CA 50, 16274 (1956), the melt is intrinsically explosive when most of the HNO₃ is evaporated. They proposed an alternate method: after adding the reagents during 2 hrs, the melt is diluted with MeOH, pptd with eth, allowed to stand overnight, the sols are decanted, and the ppt is washed with solv.


D(ethylamino)-diazido Copper Salt, [N₃]²Cu(C₂H₅NO₂)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 elect conductivity; can be prepared by dissolving Cu(N₃)₂ in the amine soln and addg acid, MeOH or ether to ppt the product; or by addg NaOH to an amine soln of the Cu²⁺ salt (Ref 2).

Refs: 1) Beilnot found 2) M. Straumanis & A. Cirulis, ZAnorgChem 251, 345-46 (1943) & CA 37, 6574 (1943)

Diethylaminoethylol Dinitrate, (O₂NCH₂CH₂N(C₂H₅)₂HNO₃; mw 225.20, N 18.66%; crysts, mp 60°; was prepared by Barbieri (Ref 2) by stirring diethylanino-ethanol, HOCH₂CH₂N(C₂H₅)₂, into fuming HNO₃ in an ice bath, and evaporating the excess HNO₃ in vacuo at a temp less than 40°. Acdg to Fakstrop & Christiansen (Ref 3), the melt is intrinsically explosive when most of the HNO₃ is evaporated. They proposed an alternate method: after adding the reagents during 2 hrs, the melt is diluted with MeOH, pptd with eth, allowed to stand overnight, the solns are decanted, and the ppt is washed with solv.


Diethylaminoethylguanidine, C₅H₁₈N₄, may be considered as the parent compd of its nitro deriv, although not used to prepare it: 1,1-Diethyl-3(2-nitraminoethyl)-2-nitroguanidine or N-(β-Nitraminoethyl)N'-diethyl-N nitroguanidine, NH₂CH₂CH₂N₂⁺NO₂

C (N₂NO₂)

N(C₂H₅)₂; mw 248.25, N 33.86%; crysts (from 95% alc), mp 152.5° dec; was obtd when 1-nitro-2-nitriminoimidazolidine was covered with diethylamine and allowed to stand at RT for 3 days,

Diethylaminoethylguanidine, C₅H₁₈N₄, may be considered as the parent cmpd of its nitro deriv, although not used to prepare it: 1,1-Diethyl-3(2-nitraminoethyl)-2-nitroguanidine or N-(β-Nitraminoethyl)-N'-diethyl-N nitroguanidine, NH₂CH₂CH₂N₂⁺NO₂

C (N₂NO₂)

N(C₂H₅)₂; mw 248.25, N 33.86%; crysts (from 95% alc), mp 152.5° dec; was obtd when 1-nitro-2-nitriminoimidazolidine 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 Dipivrate. See Vol 1, p A209-L

Diethylammonium Diazenitratocuprate,
(C2H5)2NH2[Cu(N3)2(NO3)]; mw 283.74, N 39.49%; crysts, explodes weakly at 214°
expl in contac with concd H2SO4, but not by impact; can be best prepd by reaction of the
ammonium azide & Cu(NO3)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(N3)2, called Cuprates]

DIETHYLANILINE
AND DERIVATIVES

Diethylaniline, Diethylphenylamine or Diethylaminobenzene, C10H15N, mw 149.23, N 9.39%.
The following isomers are known:
N,N-Diethylaniline (called Diethylanilin in Ger), C6H5.N(C2H5)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°,
nD 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 phosphite
in the presence of a catalyst. Diethylaniline is used in org synthesis and in the
prep of dyes. It forms numerous cryst salts & addn compds (Refs 1 & 5)
C-Diethylaniline (called Amino-diethylbenzol or Diathylanilin in Ger), H2N.C6H5(C2H5)2.
Three derivs are known: 2,4-Diethylaniline, liq, volatile with steam, bp 141° at 30mm press,
d 0.9345 at 25°, nD 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 & prepns are given in Ref
(Ref 2); and 2,6-Diethylaniline, liq, bp

235-36°, nD 1.5461 at 20°; obtd by alkyla-
tion of aniline with ethylene in the presence of
A1 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

Monoazidotdiethylaniline, C10H14N4, not found
in Beil or in CA thru 1961

Diazenidoethylamine or N,N-bis(2-azidoethyl)-aniline, C6H5.N(CH2.CH2.N3)2, mw
231.26, N 42.40%; liq, bp 132-34° at 0.2mm
press, nD 1.5748 at 25°; was prepd by reflux-
ing for 24hrs a mixt of N,N-bis(2-chloroethyl)
aniline & NaN3 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)

Triaizodietylaniline, C10H12N10 not found
in Beil or in CA thru 1961

Nitroso- and Nitroderivatives
of Diethylaniline

4-Nitroso-N,N-diethylaniline (called N,N-
Diethyl-4-nitroso-anilin, 4-Nitroso-diethylanilin or p-Nitroso-diethylanilin in Ger),
ON.C6H5.N(C2H5)2; mw 178.23, N 15.72%;
green prisms (from eth), grn lfts (from acet), or
green 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 diethyl-
aniline in HCl with NaNO2 soln and by other
methods. It forms cryst salts & addn compds
(Ref 1). No expl props were reported
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,
O2N.C6H4.N(C2H5)2; mw 194.23, N 14.42%.
The three possible isomers are described in Beil:
7-Nitro-N,N-diethylaniline, red-colored oil, bp 37° forms some unstable salts, such as Hydrobromide, C16H14N2O2 HBr, hygr. fts (from alc), dec 160°; Hydrochloride, C16H14N2O2 HCl, prisms (from alc), dec 156°; Hydroiodide, C10H14N2O2 HI, hygro ndls (from alc), dec 112°, and Picrate, C10H14N2O2 C6H5N3O7, golden fts, mp 119-20°, deflg on rapid heating (Ref 1).

3-Nitro-N,N-diethylaniline, dk-yel oil, bp 288-90°; forms a Picrate, crysls (from alc), mp 138° (Ref 2).

4-Nitro-N,N-diethylaniline, lt-grn crysls, mp 59° (Ref 3).

Other props & methods of prepn are given in the Refs.

Ref's: 1) Beil 12, (341) 2) Beil 12, 702 & (346) 3) Beil 12, 715, (351) & [386].

Dinitro-N,N-diethylaniline,

(O2N)2C6H2N(C2H5)2; mw 239.23, N 17.57%.

Three isomers are found in Beil:

2,4-Dinitro-N,N-diethylaniline, ornl prisms or prts (from acet) or yel crysls, mp 80°, d 1.374 at 15°; readily sol in hot alc; sol in acet, chlf, CS2, benz & hot eth; sl sol in petr eth (Ref 1).

2,5-Dinitro-N,N-diethylaniline, dk-red monoclinic crysls (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, ornl-colored crysls (from acet), mp 95°; and a β-modification or labile form, lt-yel fts (from MeOH), mp - stable in dry state at RT but goes to α-form at 90° or above; forms an addn cmpd with 2,4-Dinitro-N,N-diethylaniline, C10H13N3O4 + C10H13N3O4, mp 57-59°, which, on crysln from chlf + petr, separates into its components (Ref 3).

Other props & methods of prepn are given in the Refs.


Trinitro-N,N-diethylaniline,

(O2N)3C6H2N(C2H5)2; mw 284.23, N 19.71%.

Three isomers are known:

2,3,4-Trinitro-N,N-diethylaniline, crysls (from CC14), mp 132-33°; decomp to 2,4-dinitrophenylene-1,3-diamine on heating with alcohols NH3 to 125°; and to 2,4-dinitro-N1,N2 diethylphenylene-1,3-diamine on heating with methylamine in alc at 100°; was obt with the 2,4,5-trinitro deriv on nitratng 3,4-dinitrodiethylaniline with HNO3 (Ref 1).

2,4,5-Trinitro-N,N-diethylaniline, yel crysls, mp 158°; for prep 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° & 166-67°, d 1.476 at 16°; sol in eth acet, acet, 96% alc, abs alc, MeOH, benz, chlf, eth pyridine, CS2, CC14 & tolune; 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 given in the Refs.


NOTE: See also 1,2-Di(trinitroethylamino)-benzene under 1,2-Di(ethylamino)-benzene in this Vol.

N,N-Diethylaniline Pentaazidodicuprate,

(C10H16N6)[Cu2(N3)5], mw 685.28, N 33.47%; bnm lustrous ndls, burns in a flame with a crackling noise (1 mg), larger amt detonate (0.1 g), mp - explodes with a loud report at 198-99°; readily decomp by dissolving in water; was prepd by reaction of CuCl2*2H2O in MeOH, diethylaniline and a hotaq soln of NaN3.

Ref's: 1) Beil-not found 2) A. Cirulis & M. Straumanis, Ber 76B, 829 (1943) & CA 38, 1972 (1944).

DIETHYL BENZENE

And Derivatives

Diethylbenzene (called Diethyl benzol in Ger), C6H4(C2H5)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°, nD 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°, nD 1.4955 at 20° (Ref 2)

1,4-Diethylbenzene, liq, fr p -42.8°, bp 183.8°, d 0.8620 at 20°, nD 1.4947 at 20° (Ref 3)

Other props & methods of prepn are given in the Refs:

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.

Mononitrodioethylbenzene, O_2N.C_{6}H_{5}(C_{2}H_{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°, nD 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°, nD 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 the Refs:

Dinitrodioethylbenzene, (O_2N)_{2}C_{6}H_{2}(C_{2}H_{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 aic), mp 89-90°, was obtg, 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. Volcavi, Gazz 87, 332, 340 (1957) & CA 52, 14550 (1958)

2,4,6-Trinitro-1,3-diethylbenzene,
(O_{2}N)_{3}C_{6}H(C_{2}H_{5})_2; mw 269.21, N 15.61%; prisms (from pettr), lfts (from aic), mp 62-63°, was prepd by nitrating 1,3-diethylbenzene with mixed acid in the cld for 4 hrs and then heating to 135° (Refs 1, 2 & 3). No expl props of this compd were reported.


2,3,6-Trinitro-1,3-di(β-nitroxyethyl)-benzene, (O_{3}N)_{2}C_{6}H(CH_{2}C_{2}H_{5}ONO)_{2}; mw 391.21, N 17.90%; exp 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'-Diethylenilene or 4,4'-Dietlyldiamino-biphenyl. (called N,N'-Dithylbenzidin in Ger), [-C_{6}H_{4}.N.C_{2}H_{5}]; mw 240.34, N 11.65%; plts or ndls (from aic), 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_{6}H_{4}.N(NO).C_{2}H_{5}]; mw 298.34, N 18.78%; yel lifts, mp 162.5-163.5°; mod sol in hot glc acr ac; less sol in hot petr eth; was prepd by nitrosating N,N'-diethylbenzidene with NaNO_{2} in dill HCl (Refs 1 & 2)

Refs: 1) Beil 13, 233 2) E. Bamberger & M. Tchivinsky, Ber 35, 4184 (1902)

N,N'-Di(2-nitroethyl)-benzidine or N,N'-Bis (2-nitroethyl)-benzidine,
[-C_{6}H_{4}.NH.C_{2}H_{5}.ONO]_{2}; mw 330.34, N 16.96%; crystals from EtOAc, MeOH & xylene, mp 177°; was obtg by addg dropwise an excess of nitroethyrene (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.


3,5,3',5'-Tetranitro-N,N'-diethylbenzidine,
[-(O_{2}N)_{2}C_{6}H_{2}.N.C_{2}H_{5}]; mw 420.34, N 20.00%; crystals (from methyl salicylate), mp 248°; was obtg 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 Romburg,
Rec 41, 41 (1922) & CA 16, 1238 (1922)
N,N,3,5,3′,5′-Hexanitro-N,N′-diethylenizidine,
[(O\_2N)\_2C\_6H\_2N(NO\_2)]\_2; mw 510.33;
N 21.97%; It-yel ndls (from HNO\_3), mp 230°
de) was prep by treating the tetranitro
deriv with concd HNO\_3 (Ref 2). No expl
props were detd
Refs: 1) Beil 13, [109] 2) G. van Romburg,
Rec 41, 41 (1922) & CA 16, 1238 (1922)
NOTE: See also Diethylphenyldiamine and
its Tetrannitro Derivatives in this Vol

Diethylbenzylamine and Derivatives
Diethylbenzylamine (called Diethylbenzylamin
in Gen), C\_6H\_5CH\_2.N(C\_2H\_5)\_2; mw 163.25, N
8.58%; cl r oil, bp 211-12°; diffe sol in w;
can be prep by reacting diethylbenzylcinnamyl-
ammonium chloride with Na amalgam in water
and by other methods; forms some cryst, un-
stable salts (Ref)
Ref: Beil 12, 1021, (448) & [546]
Azido, C\_11H\_16N\_4, and Diazido, C\_11H\_15N\_2O\_2,
derivatives were not found in Beil or in CA
thru 1961

Nitroderivatives
Diethylnitrobenzylamine, O\_2N.C\_6H\_4.CH\_2.-
N(C\_2H\_5)\_2; mw 208.25, N 13.45%. Three iso-
mers are known:
Diethyl-2-nitrobenzylamine, yel liq, having a
herring-like odor, bp 144° at 13 mm & 177-77°
at 42 mm press; readily sol in common org
solvs; v sl sol in w; forms a Picrate,
C\_11H\_16N\_2O\_2 + C\_3H\_3N\_3O\_7, mp 122° (Ref 1)
Diethyl-3-nitrobenzylamine, yel liq, having a
herring-like odor, bp 158° at 13 mm & 206-208°
at 42 mm 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 13 mm & 219-21°
at 42 mm press; readily sol in common org
solvs; v sl sol in w; forms a Picrate, mp 131°
(Ref 3)

Other props & methods of prep 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%; cryst (from petr eth + eth, below
10°, 7 times), mp 66°; was obt by adding
dropwise an excess of nitroethylene
(CH\_2.CHNO\_2) in an inert solv to benzyl-
amine at -20° for 30 mins, isolating by treat-
ing 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 Diethyl-
benzylamine were found in Beil or in CA thru
1961

Diethylbisphenyldiamine or 3,3′-Diethyldi-
aminobiphenyl, C\_16H\_20N\_2, may be considered
as the parent compd of its tetranitro deriv al-
though not used to prep it:
4,6,4′,6′-Tetranitro-N,N′-diethyl-3,3′-biphenyl-
diamine (called 4,6,4′,6′-Tetranitro-3,3′-di
(ethylamino)-diphenyl by van Alphen),
[(O\_2N)\_2C\_6H\_2NH.C\_2H\_5]\_3; mw 420.34, N
20.00%; mp yel powd which turns quickly
red on heating at 270-80°, mp 315-20°(dec);
was prep 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 Diethyl-
benzylamine were found in Beil or in CA
thru 1961. See also N,N′-Diethylbenzylidine
and Derivatives in this Vol

N,N′-Diethylcarbonilide. See Centralite 1
in Vol 2, p C127-L

Diethyl Carbonate. See Ethyl Carbonate in Vol 6

Di(ethylcarboxamide)-piperazine, C\_10H\_20N\_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,
A piperazine hexahydrate (QH2·6H2O), where QH2 represents 1,4-piperazine & Q the 1,4-piperazine ring group) in water was neutralized with conc'd
HCl, heated 1 hr at 50° with KCNO in w, and chilled to give Q(CONH2)2, mp 288-90° (dec). Formalin was added to Q(CONH2)2 suspended in saturated aq Ba(OH)2. CO2 was passed into the soln.; BaCO3 removed, the filtrate concd at RT, and acetone added to ppt
Q(CONHCH2OH)2, mp 190-210°. Solns of the latter compd & CH(NO3)2, both in water, were mixed for 1 hr at 45°, filtered, and the product collected. This compd was patented as an expn & propelnt (Ref 2).

Ref:s: 1) Beil-not found 2) P.O. Tawney, USP 3038002 (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 Diethylbiphenylidine in this Vol

4,4'-Diethyldiaminobiphenyl. See N,N'-Diethylbenzidine in this Vol

Diethyldiborane, B2H4(C2H5)2; mw 83.79; exists as sym-, vapor press 30mm at 0°, which does not decomp in 24 hrs at 25° & unsym-, vapor press 46mm at 0°, which 4.5% decomps in 0.5 hr, and 29.4% decomps in 16 hrs at 25°. Sym-diethyldiborane was separated by fractionating from the equilibrium mixt obt'd by heating ethyldiborane. Unsym-diethyldiborane was prep'd 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 B2H6 at 25-55°, there is produced B2H7(C2H5)2, mp 101°, nD 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


Diethylidydroxyamine. See Diethylolamine in this Vol

N,N'-Diethyl-N,N'-diphenylurea. See Centralite I in Vol 2, p C127-L

N,N'-Diethyl-N,N'-diphenylurea-Nitroguanidine Complex (Centralite-1NgU Complex), H2N.C(NH).NH.NO2+ [H2C2N6C6H5]2CO; Ir-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).

Ref:s: 1) Beil-not found 2) M. Parpaillon, MP 38, 233-41 (1956) & CA 51, 11717 (1957)

DIETHYLENEDIAMINE
AND DERIVATIVES

Diethylenediamine, Piperazine or Hexahydropyrazine (called Piperazine, Hexahydropyrazine or Diethylenediamine in Ger),
Di(ethylenediamino)-cadmium (II) Azide,
\( \text{Cd}[\text{C}_2\text{H}_4(\text{NH}_2)_2]\text{I}_2(\text{N}_3)_2; \) mw 325.37, N 26.55% 
white ppt, mp - melts & deflagr 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,
\( \text{Cu}[\text{C}_2\text{H}_4(\text{NH}_2)_2]\text{I}_2(\text{N}_3)_2; \) mw 267.79, N 31.39%; violet crystals, mp 180-82°, melts in a flame followed by expln, explodes in a pre-heated block at 212-16°; sol in w.; sl sol in alc.; insol in eth.; was prep'd 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- & trans-forms. Prepn and properties are given in Ref 2.

1) cis-Dinitro-bis(ethylenediamine)cobalt (III) nitrate cis-[Co en_2(NO_2)_2NO_3], N 29.44%.
Brown-yell cryst., may be prep'd by treating potassium hexanitrocobaltate (III) with dil aq soln of ethylenediamine (abbr en),
\( \text{C}_2\text{H}_4(\text{NH}_2)_2\text{I}_2, \) followed by heating and cooling. The resulting nitrite cis-[Co en_2(NO_2)_2NO_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)_2NO_3], N 29.44%. Yell cryst.; may be prep'd by adding ethylenediamine (70% aq soln), partially neutralized by HNO_3, to aq soln of 
\( \text{Co(NO}_3)_2\text{H}_2\text{O} \) 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

Ref: 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(CH₂-CH₂-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°, ng 1.4469 at 20°; can be obd 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)

Diethylene glycol is used in plasticizers, humectants, solvs, brake fluids, antifreeze
& stiffening agents. Its derivs are useful in
many other applications (Refs 5 & 7)

The conditions of acceptance & examina-
tion of Diethylene glycol intended for nitra-
tion 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 & Orthmer; 2nd edit 10 (1966),
646

Diethylene glycol Dinitrate (DEGDN) (called
Diäthylenglykol-dinitrat in Ger);
O(CH₂-CH₂-ONO₂)₂; mw 196.12, N 14.29 %
; clear, colorless liq, having no odor, fr p
-11.2 to -11.4°, bp 160-61°, volatile betw
15 & 55°, d 1.3909 at 15°, Q_combustn 548.7
kcal/mol; readily sol in chlf, MeOH, eth,
acet, benz, toluene & glc acetic acid; al-
most insol in alc, CCl₄ & CS₂; prepd in 85%
yield by nitrating pure diethylene glycol (50 gms)
with mixed acid (HNO₃ 50, H₂SO₄ 45 & H₂O 5%
- 18.4 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 cmpds to be useful as an expl.

Its props are tabulated in Ref 26 and de-
scribed in more detail by Urbanski (Ref 25)

Hough (Ref 3) patented its prepn and
of propants "based" on NG gelatinized with
20-25% DEGDN (Ref 4). Other uses in
propants are described in patents by Achil-
es (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 prop-
plnts are due to its props of being a good gelatin-
izer for NG, and its high chem stability
resulting in less hazards during processing of
propants than is possible with NG propants
Refs: 1) Beil 1, [521] & [2112] 2) W.H.
Rinklebach, IEC 19, 925-27 (1927)
3) A. Hough, BritP 341456 (1929) & CA
5033 (1931) 4) A. Hough, BritP 343107
(1929) & CA 27, 2303 (1933) 5) W.H.
Rinklebach & H.A. Aaronson, IEC 23, 160-
63 (1931) & CA 25, 1998 (1931) 6) O.W.
Stickland et al, "General Summary of Ex-
pls Plant", PB Rept 925 (1945), p 57
(Prepn as practiced at German Krümmel
Fabrik of D-A) 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)
Diethylenglycol Perchlorate,
HO.H₂C.H₂C.O.CH₂.CH₂.ClO₄⁻; mw 204.53,
O 46.94%; col oil, very hygro, explodes violently
on heating rapidly; was obtd from ethyl-
ene oxide & HClO₄ 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(aminomethyl)-amine in Vol 2, p 128-L

Diethylenetriamine Complexes of Cobalt.
See Cobalt in Vol 3, p C383-R

Diethyl ether and Derivatives
Diethyl ether, Ethyl Ether, commonly known as Ether
(called Diäthyläther, Äthylläther or Äther in Ger) O(C₂H₅)₂; mw 74.12, O 21.59%;
col, very volatile, highly flammable liq, hav-
ing a sweet pungent odor & burning taste,
fr p -116.3°, bp 34.3°, 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°, np 1.3527 at 20°, Qcombstn 651.7
kcal/mol at 20°; miscible with alc, benz,
chl, petr eth, oils & many fat solvs; very
sol in concd HCl, but may explode in contact
with anhyd HNO₃; 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 sub-
lethal 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, CO₂, CCl₄ & dry chemical fire
extinguishers may be used. Automatic sprink-
ers, deluge systems & high-pressure water
spray are also effective in extinguishing
Ether fires (Refs 1, 6 & 7)
Uses. Diethyl ether is probably the most im-
portant member of the ether family. It has
a wide range of uses in the chem industry:
- a good solv for fats, waxes, oils, resins, dyms,
guns & alkaloids; mixed with alc, it is an
excellent solv for Guncotton, Collodion Cot-
ton & Pyroxylin; a denaturant for alcohols;
a startling fuel for diesel engines; a general
anesthetic in surgery; and as an inert reaction
medium in Grignard & Wurtz-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 in-
org acid, Ether undergoes cleavage. It can
be oxidized to acetic acid. With halides, Ether
forms a variety of cryst addn compds.
Ether can be nitratd safely in the vapor
phase to give methyl nitrte, ethyl nitrte &
2-nitroethyl ethyl ether
For addnl info, see in Vol 6 of Encycl
under Ether
Refs: 1) Beil 1, 314, (158), [311] & 11289
2) P.W. Danckworts, ZAngChem 40, 1317(1927)
& CA 22, 587(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. Esters 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 sodium, should be added & the mix 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$_2$SO$_4$, 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.


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 & Lorriette (Ref 2) suggest comparing the behavior of a test sample with that of pure Ether, prep'd by washing ordinary Ether with ag H$_2$SO$_4$ 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$_2$CrO$_4$ + dil H$_2$SO$_4$) 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 propihnts 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, peroxydes, 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. Lorriette, MP 21, 277-80 (1924) 3) L. Brandt, ChemZtg 51,
981-83 (1927) & CA 22, 869 (1928) (Peroxide detection by KI - starch & Tl
solns)
4) A. Lalanne, MP 25, 421-35 (1932-33)
(Freezing points & densities of binary mixts
of Ether-Alcohol) 5) (7). Ploux & (7).
Parpaillon, MP 30, 255-61 (1948) 6) AB
Bofors, Nobelkultur, "Analytical Methods for
Powders and Explosives", Bofors, Sweden
(1960), 86 7 CondChemDiet (1961), 453
1-Azido-1,2-diamylether (called β-Azido-diethyl-
äther in Ger), N₃H₂C.H₂C.O.C₂H₅; 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 prep'd 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 97(11), 2579 (1910)
3) K.A. Kornev & S.B. Serebyrain, ZapiskiInstitKhim-
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. Sinnenma & J.F. Arens,
Rec 75, 1423-28 (1956) & CA 51, 8683 (1957)
(Describe prep of 1-azido-1-ethoxyethane
[H₂C.CH(N₃)OC₂H₅] and 1,1-diazido-1-etho-
ythene [H₂C.C(N₃)₂OC₂H₅]
Mononitrodiethylether (called Athyl(2-nitro-
äthyl)-äther or 2-Nitro-diäthylether in Ger),
O₃N.H₂C.H₂C.O.C₂H₅; 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); n₂ 1.4160 at 25°(Ref 4); was first
prep'd by heating β-iododiethylether with Ag
nitrate, and later by heating either 36 parts
of 1,2-dinitroethane or 8 parts 2-nitroethyl-
nitrate with ethanol (Ref 1); and by other
methods (Refs 2, 3 & 4)
Refs: 1) Beil 1, 359 & (1365) 2) A.E.
Wildier-Smith et al, USP 2416650 (1947) & CA
41, 3478 (1947)(Prepn of 2-Nitroethylethers)
3) A. Lambret a et al, JCS 1947, 1475 & CA 42,
4907 (1948) 4) H.B. Hass & D.E. Hudgins,
JACS 76, 2692 (1954) & CA 49, 8786 (1955)
Di(2-nitroethyl)ether or Bis(2-nitroethyl)
ether (called β,β-Dinitro-diäthyäther or
Athyl-β,β-Dinitroäthyäther in Ger),
(O₂N)₂HC.H₂C.O.C₂H₅; 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 acidifi-
cation if its Potassium salt, K₂C₄H₇N₂O₅;
le-yel ndls (from aic), prep'd by reacting tri-
nitroethane with K ethoxide; forms a Bromine
deriv, C₄H₇N₂O₅Br, col oil, bp 103-04° at
13mm press; and a Silver salt, Ag.C₄H₇N₂O₅,
which is probably expl (See Refs)
Refs: 1) Beil 1, 340 2) A. Hantsch &
A. Rinckenberger, Ber 32, 637 (1899) & JCS
761, 404 (1899) 3) J. Meinshaeimer, Ber 36,
434 (1903) & JCS 84(1), 223 (1903)
4) J. Meinshaeimer & M. Schwartz, Ber 39,
2546 (1906) & JCS 90I, 618-19 (1906)
5) J.A. Chenike, USP 2818453 (1957) & CA 52,
8539 (1958)
(Solv useful to separate aromatic hydrocar-
bons from their mixts)

**Diethyl-ethylenediamine and Derivatives**

_N,N'-Diethyl-ethylenediamine_ (called _N,N'-
Diethyläthyläthyldiamin_ or _α,β-Bis(äthylamin)-
häthan in Ger), H₃C₂.H.N.CH₂.CH₂.N.H.C₂H₅;
mw 116.20, N 24.11%; liq, bp 151-52° &
65.5° at 35mm press; was prep'd by reaction
of ethylene dichloride & ethylamine; forms
some salts which are unstable on heating:
[Cu(C₅H₁₆N₂)₂](ClO₄)₂, blue-violet crts,
deflg on heating; and [Ni(C₅H₁₆N₂)₂(H₂O)₂] -
(ClO₄)², reddish-blue plts (from MeOH),
deflg 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_;
(O₂N)₃C.H₂.C.H₂.C.H₂.N.CH₂.Ch₂.C(NO₂)₃;
mw 386.20, N 29.02%; yel solid, mp 97°;
was obtd by treating 1 mol of ethylenediamine,
(H₂N.C.H₂.C.H₂.N.H₂), with 2 moles of tri-
nitroethanol [(O₂N)₃C.H₂.OH], 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
propelnts
Refs: 1) Beil-not found 2) F.R. Schenck
Diethylguanidine and Derivatives

Diethylguanidine (called Diethyl-guanidin in Ger), C₅H₁₁N₃; mw 115.18, N 36.49%. Two isomers are known:
N,N'-Diethylguanidine, (C₂H₅)₂N.C(NH).NH₂; hygr ndls (from eth), mp 88-89°C; readily sol in w, alc & benz; sl sol in eth; forms cryst salts with acids (Ref 2)
N,N'-Diethylguanidine, C₂H₅NH.C(NH).NH⁻; C₂H₅; It-yel oil; forms cryst salts (Ref 1)

Other props & methods of prep are found in Beil

N,N-Diethyl-3-nitroguanidine or 1,1-Diethyl-3-nitroguanidine, (C₂H₅)₂N.C(NH).NO₂; mw 160.18, N 34.98%; crys (from alc), mp 92-94°C; was prep by reacting 2-methyl-1-(or 3)-nitro-2-thiophenecourea

[CH₃SC(NH).NH.NO₂], with diethylamine at RT (Ref 3)

N,N'-Diethyl-2-nitroguanidine or 1,3-Diethyl-2-nitroguanidine,
C₂H₅HN.C(NO₂),NH.C₂H₅;crysts (from 95% alc), mp 115-116°C; was obtd by reacting 1-nitroso-2-nitramino-2-imidazoline,

O₂N⁻→C(NHNO₂)==N

H₂C ———— CH₂, with ethylamine in water below 30°C (Ref 2)

Their expl props were not reported
Refs: 1) Beil-not found 2) A.F. McKay, JOC 16, 1849 (1951) & CA 46, 9907 (1952)
3) L. Fishbein & J.A. Gallagher, 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) C₄H₁₀.COOC₂H₅.CH(C₂H₅)₂C₄H₅; mw 370.56, O 17.27%; lt-colored oil, frp -70°C, bp 417°C, 214°C at 5mm press, flash p 405°C, d 0.9268 at 20°C, nD 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 485-L). US military requirements & tests are described in Ref 2.
Di(2-ethylhexyl) azelate or Dioctyl Azelate (DOZ) (CH₂)₇[COOC₂H₅(C₂H₅)C₄H₉]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°, nD 1.4472 (Refs 2 & 3). DOZ is used as a plasticizer in some composite rocket propellants (Ref 3)

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 Dioctyl Sebacate (DOS), (CH₂)₆(COOC₂H₅)₁₇; 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°, nD 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)

Diethylhydroxylamine and Derivatives

Diethylhydroxylamine (called N,N-Diethylhydroxylamin in Ger) C₄H₉N₂O; mw 88.15, N 31.78%. Two isomers are known:
N,N-Diethylhydroxylamine, (C₂H₅)₂N.H₂; hygr liq, having an etherish ammonia-like odor, bp 96-99°; readily sol in w, alc, eth, chlf & benz (Ref 1)
N,N¹-Diethylhydroxylamine, C₂H₅.HN.NH.C₂H₅; liq, bp 84-86° at 758mm press; forms a Hydrochloride salt, C₄H₁₂N₂ + HCl, plts (from aq HCl), mp 160°(dec)(Ref 2)
Other props & methods of prepn are given in Beil
Diethylhyponitrite or Ethylhyponitrite (called Diäthylhyponitrit, Äthylhyponitrit or Diazoveroxan in Ger), C₂H₅.O:N:O:N₂.C₂H₅; mw 118.14, N 23.71%; oil, bp - cannot be distilled even under reduced pressure, explodes on heating at 80-82°, on impact, or by friction; d 1.0326 at 20°, n₁ 1.4047 at 20°; readily sol in alc, eth & benz; insol in w, HCl & NaOH; was prep'd by treating ethyliodide in ether with Ag hyponitrite in a freezing mixt (Refs)


Diethyldiene Diperoxide; Diacetaldehyde Peroxide; Acetaldehyde Superoxide; or Ethyldiene Peroxide (called 3,6-Dimethyl-1,2,4,5-tetroxan or Diäthyliden-diperoxyd in Ger),

\[
\begin{align*}
\text{CH}_3\text{CH} & \quad \begin{array}{c} \text{O} \\ \text{O} \end{array} \quad \text{CH.CH}_3 \\
\text{H}_2\text{C.CH} & \quad \begin{array}{c} \text{O} \\ \text{O} \end{array} \quad \text{C.CH}_3
\end{align*}
\]

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, (CH₃CHOO)₂, crysts, mp 22.5-23°, nD 1.4160, a highly expl compd, has been prep'd by several methods; a) allowing an ethereal soln of a-hydroxyalkylhydroperoxide to stand over P₂O₅ b) vacuum treatment of 1-hydroperoxyethanol at 70-75° c) dehydration of di-(1-hydroxyethyl) peroxide with P₂O₅ and d) from butene ozonide


Diethylketene Peroxide (called Diäthylketen Peroxyd in Ger), C₆H₁₂O₃; mw 130.14,- O 36.88%; oil; was prep'd by treating an ethereal soln of diäthylketene, (C₂H₅)₂C:C:O, with H₂O₂ at -20°, an extremely expl compd; decmp into CO₂ & diäthylketone if allowed to stand in ether for several days (Refs 1 & 2)

Refs: 1) Beil I, (384) 2) H. Staudeiner & J. Maier, Ann 401, 287 (1913) & CA 8, 496 (1914)

Diethyl Lead Dioxide (called Diäthylbleidiazid in Ger), (C₂H₅)₂Pb(N₃)₂; mw 349.39, N 24.10%; crysts, mp - detonates with a bright flame when heated; was prep'd by adding drop-wise aq Na azide to an aq soln of diethyl lead chloride at 50°(Refs 1 & 4). Diethyl lead chloride, (C₂H₅)₂PbCl₂, can be prep'd by the action of tetraethyl lead on chlorine, followed by crysta 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 parented 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. Grütner & E. Krause, Ber 49, 1426

**Diethyl Lead Dibenzoate,**

(C$_2$H$_5$)$_2$Pb(OOC.C$_6$H$_5$)$_2$; mw 507.55, O 12.61%; col ndis (from benz), mp 188°, explodes mildly when heated rapidly; sol in hot alc & benz; insol in w; can be prep by re-
action 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 sul-
fite (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$_2$H$_5$)$_2$Hg; 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 1012cal/mol, I$_2$ 1.5487 at 20°; almost insol in w; sl sol in alc; sol in eth; was prep 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 expts in most cases

2) H. Gilman & R.E. Brown, JACS 52, 3314-
17 (1930) & CA 24, 4758(1930) 3) M.M.
Konton, ZhObshchKhim 19, 730-33 (1949) & CA 44, 1044(1950) 4) Sax (1963), 717 (Compd is highly toxic)

**Diethyl-methylenediamine**

and Derivatives

N$_2$N'-Diethyl-methylenediamine,

H$_2$C$_2$.HN.CH$_2$.NH.C$_2$.H$_5$, 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(ethyl nitramine)-methane; or 3,5-

Dinitro-3,5-diazahentane,

H$_5$C$_2$.N(NO$_2$).CH$_2$.N(NO$_2$).C$_2$.H$_5$; mw 192.18, N 29.16%; crystals (from hexane), mp 75.7-77.2°; was prep by addg in small portions ethyl nit-
tramine to a chilled soln of paraformaldehyde in 90% H$_2$SO$_4$ 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-nitroguani-
dine. See under Diethylnitaminoethylguanidine in this Vol

**Di(ethylnitrosamine)-azoxybenzene.** See Bis(ethylnitrosamine)-azoxybenzene in Vol 2, p B142-R

**DIETHYLOLAMINE AND DERIVATIVES**

*Diethylolelamine; Diethanolamine; 2-Aminobutane-
1,4-diol; or Diethyl-2,2'-dihydroxyamine; or Bis (2-hydroxyethyl)-amine [called Bis-(2-hydroxy-
ethyl)-amin; 2,2'-Dihydroxy-diethylammon (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 crystd salts & addn compds, some of which are unstable on heating. Other props & methods of prep are found in Ref 4


*Diethylole-nitroamine 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
obt'd when NaNO₂ in water was slowly added to diethylolamine in w, previously neutralized with 2N HCl (Refs 1 & 2).


Diethylolamine Dinitrate; Di(nitroxyethyl)amine or 2,2'-iminodiethanol Dinitrate (CA nomenclature), HN(CH₂CH₂ONO₂)₂; known in the form of its salts: Hydrochloride, C₄H₉N₃O₆.HCl, plaids (from acet ac), mp 162.5-630°C (dec); Nitrate [called Di(nitroxyethyl) ammonium nitrate] C₄H₉N₃O₆.HNO₃, wh hygr crystals (from acet ac or acet), mp 120.5 & 127°C, can be ignited and burns leaving a deposit of C; can be detonated by impact; readily sol in ac, MeOH, hot alc, w & nitromethane; insol in benz, chloro, eth & petr eth; was obt'd by reaction of concd HNO₃ at low temp on diethylolamine;

Picrate. C₄H₉N₃O₆ + C₆H₅N₂O₃, crysts (from toluene + eth), mp 156-37°C.


Di(nitroxyethyl)-nitrosoamine or 2,2'-Nitroso(diethyl)amine Dinitrate (CA nomenclature), ON.N(CH₂CH₂ONO₂)₂; mw 224.14, N 25.00%; crysts (from eth + petr eth), mp 46-47°C, slowly dec spontaneously at 25°C; readily ignites and burns, leaving a bn 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 decomposed rapidly on boiling; was prep'd by the action of NaNO₂ in w & dil HNO₃ on Di(nitroxyethyl)-ammonium Nitrate (qv) at 0°C.


Di(2-nitroxyethyl)-nitramine, N,N-Bis(2-nitroxyethyl)-nitramine; Diethanolnitramine Dinitrate (DINA) or 2,2'-Nitroso(diethyl)amine Dinitrate (CA nomenclature) [called Bis-(2-nitroxyethyl)-nitramine or Disal-petersäureester des Bis-(2-hydroxy-äthy)-nitramins in Ger], O₃N.N(CH₂CH₂ONO₂)₂; mw 240.14, N 23.33%; crysts fromaq acet, mp 52.5°C, dec at 165°C, d 1.67 at 25°C; heat capacity 0.38 cal/g, heat of fusion 23.5 cal/g, heat of combust 1260-1350 cal/g; sol in gla AcOH, MeOH, alc, benz & eth; insol in w, CCl₄ & petr eth; can be prep'd from diethylolamine by nitrating with 100% HNO₃ & acetic anhydride in the presence of HCl at 5-40°C (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°C 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 expl 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 expls in 5 trials vs no expls 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(nitroxyethyl)-nitroamine, which might be removed by heating.

Uses. DINA has been proposed as a non-volatile 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).

(Thermal expl of DNA under purely convective heat transfer conditions) (See also CA 56, 11873 (1962) 27) Urbanski 3 (1967), 36
Diethylolamine Trinitrate; Diethanolamine Trinitrate, Di(β-nitroxyethyl)-ammonium Nitrate; Bis(2-nitroxyethyl)-ammonium Nitrate; or Dinitroxyaminodiethane Nitrate [called in Fr Nitrate de Dinitroxy-diethylammonium or Nitrate de Bis(nitroxyethyl)-ammonium], O₂N₂NH(CH₂.CH₂ONO₂)₂; mw 258.16, N 21.71%; wh crystals, mp 120.5-23.8°, d 0.80 (bulk) 1.595 (pressed at 2800 kg/sq cm; Q_combust = 2260 cal/g; sol in acet, MeOH, hot alc & w; less sol in cold alc & cold w; was prep'd by treating diethylolamine with either 98% HNO₃ or mixed acid (Refs 2, 3 & 4).

Its expl props were det 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.


Di(2-nitroxyethyl)-chloramine; 2,2'-Chloroimino-diethanol Dinitrate (CA nomenclature) or β-β'-Dinitroxyethyl-chloramine (called by Meyers & Wright) [called Bis(2-nitroxyethyl)-chloramin or Dsalpersäureester des Bis(2-hydroxy-ethyl)-chloramins in Ger], C₂H₄(C₂H₂ONONO₂)₂; mw 229.58; N 18.30%; gr-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°, ng 1.4840 at 25°, can be prep'd 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 HNO₃ & acetic anhydride at 10° (Refs 1 & 2). This compd can be nitrated to give an 80% of DNA (qv).


Di(ethylolamino)-benzene and Derivatives
1,3-Di(ethylolamino)-benzene; 1,3-Bis(β-ethanolamino)-benzene; or 1,3-Bis(β-hydroxyethylamino)-benzene, C₆H₄(NH₂(CH₂.CH₂OH)₂; may be considered as the parent compd of its nitrated derivs although not used to prep them: Dinitro-1,3-di(ethylolamino)-benzene, (O₂N)₂C₆H₄(NH₂(CH₂.CH₂OH)₂; mw 286.24, N 19.58%
2,4-Dinitro-1,3-di(ethylolamino)-benzene or 2,4-Dinitro-di(4-hydroxyethyl)-m-phenylenediamine, yel crystals (from ethyl acet), mp 140-41°; was prep'd by reaction of an excess of hydroxyethylamine & 2,3,4-trinitromethylaniline 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 prep'd 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.

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, (O₂N)₂C₆H₄[N(NO₂)₂.CH₂.CH₂OH]₂; mw 421.27, N 23.28%; thick, sticky mass (no cryst product obtld), 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(oethyl)l-ethylene diamine and Derivatives
N,N'-Di(b-ethyl)-ethylene diamine; N,N'-Bis(2-hydroxyethyl)-ethylene diamine; or
2,2'-[Ethylenediamino]-diethanol (CA nomenclature) [called 1,2-Bis(2-hydroxy-ethylamino)-äthan or N,N'-Bis(2-hydroxy-ethyl)-äthylenediamin in Ger],
CH₂-NH.CH₂-CH₂-OH

CH₂-NH.CH₂.CH₂-OH; 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-ethyl)-ethylene diamine Dinitrate; 2,2'-[Ethylenediamino]-diethanol
Dinitrate (CA nomenclature) or N,N'-Bis
(2-nitroxyethyl)-ethylene diamine [called
N,N'-Bis(2-nitroxyethyl)-äthylenediamin or
Disalpetersäureester des 1,2-Bis-(2-hydroxy-
äthylamino)-äthans in Ger],
CH₂-NH.CH₂-CH₂-OONO₂

CH₂-NH.CH₂.CH₂-OONO₂; 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% HNO₃
as described in Refs 2, 3, 5, 6 & 7
Refs: 1) Beil 4, [718] 2) J.R. Johnson,
OSRD 915 (Oct 1942), 21-24 3) R.N.
Jones & G.D. Thorn, CanJRes 27B, 855
(1949) & CA 44, 2848 (1950) (UV absorption
spectrum of the Dinitrate) 5) A.T. Blom-
quist, 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)-ethylene dinitrimate;
N,N'-Bis(β-nitroxyethyl)-ethylene dinitrat-
mine; 2,2'-[Ethylene-bis(nitroimino)]-di-
ethanol Dinitrate (CA nomenclature); and
1,8-Dinitroxy-3,6-diazaoctane Dinitrate
(Jones & Thorne) [called N,N'-Dinitro-
N,N'-bis(2-nitroxy-äthy1)-äthylenediamin;
or Disalpetersäureester des N,N'-Nitro-
N,N'-bis(2-hydroxy-äthyl)-äthylenediamins
in Ger],
CH₂-N(NO₂).CH₂.CH₂.CH₂.ONO₂

CH₂-N(NO₂).CH₂.CH₂.CH₂.ONO₂; mw 328.11;
N 25.62%; wh crys (from MeOH), mp
60-62° & 65.5-67.5°; was prepd from the
tetranitrate deriv by heating it with acetic
anhydride & a small amt of a chloride cata-
lyst (ZnCl₂) (Refs 2, 3, 4 & 5), or by a two-
stage process from ethylene oxide & ethylene-
dinitrimate, (CH₂.NHNO₂)₂, in NaOH, and
treating the product, [CH₃N(NO₂)CH₂.CH₂OH]₂,
with HNO₃ 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
propins
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)

Diethyl-oxamide and Derivatives
N,N'-Diethyl-oxamide, Diethanol-oxamide,
Dibydroxy-diethyl-oxamide, or Bis(hydroxy-
ethyl)-oxamide [called N,N'-Bis(2-hydroxy-
äthyl)-oxamid or Oxalsaure-bis(2-hydroxy-
äthylamid) in Ger],
CO.NH.CH₂.CH₂-OH

CO.NH.CH₂.CH₂-OH; mw 176.17, N 15.90%;
crysts (from alc), mp 167-69.2°C; readily sol
in w, glc acet ac & hot alc; less sol in
chlf, benz & petr eth; insol in eth; was
prep'd by addg 1 equiv of dibutyl oxalate to
a well stirred soln of 2 equivs of monoethyl-
anolamine in dry butanol below 37°C, and
then heating the reaction mixt to 98°C for 2 hrs
(Refs 1 & 2)
Stuart & G.F. Wright, CanJ Res 26B, 402,
408 (1948) & CA 42, 6753 (1948)
N,N'-Di(nitroxyethyl)-oxamide; N,N'-Di
(ethylox)-oximide Dinitrate; N,N'-Bis(2-
nitroxyethyl)-oximide; or N,N'-Bis(2-hydroxy-
ethyl)-oximide Dinitrate (CA nomenclature),
CO.NH.CH₂.CH₂ONO₂
| CO.NH.CH₂.CH₂ONO₂; mw 266.17, N 21.05%;
crysts (from HNO₃), mp 148.2°C; was prep'd
by nitrating the parent compd with abs HNO₃
for 3 hrs at 0°C, or by nitroxylation mono-
ethanolamine, converting the nitroxyethyl-
ammonium nitrate to free base by cautiously
addg KOH, and treating with diamin 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, CanJ Res 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-ethylox)-oximide Di-
nitrate; N,N'-Dinitro-N,N'-bis(2-hydroxy-
ethylo)oximide Dinitrate (CA nomenclature);
or Bis-(nitroxyethylnitro)-oximide, abbreviated
as NENO [called N,N'-Dinitro-N,N'-bis
(2-nitroxy-athylox)amid or Disalpeter-
säureester des N,N'-Dinitro-N,N'-bis(2-
hydroxy-athylox)-oxamids in Ger; and Dinitrate
du Dioxabethdenitrooximide in Fr],
CO.N(NO₂).CH₂.CH₂ONO₂
| CO.N(NO₂).CH₂.CH₂ONO₂; mw 356.17,
N 23.60%; exists in two polymorphic forms:
alpha. ndls, stable at RT, obtd by slow
crystn from chlf below 38°C; and beta,
massive monoclinic, obtd by crystn from
chlf above 38°C & filtering to prevent forma-
tion of alpha form; mp 90-92°C, dec 105°C,
d 1.706 at 22°C (α-form), d 1.686 at 22°C &
1.562 at 92.6°C (β-form); Q_p_combstn 712
kcal/mol; sol in acet, hot alc, eth acet &
sis-dichloroethylene; sl sol in benz, chlf &
alc; insol in w (Refs 1, 17 & 18). This
compd was first prep'd by Von Herz (Ref 2)
by condensing ethanolamine with oxiacid,
and nitrating the product. Stuart &
Wright (Ref 17) prep'd it by nitrating N,N-
diethylox-amide with mixed acid at 40°C,
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
1.60-1.64 g/cc entirely free of cavitation.
It does not stain the hands or cause derma-
titis as does Tetryl. No health hazards have
been encountered in use after 4 yrs
(Ref 17)

NENO is an HE comparable in power &
brisance to Tetryl and 1.3 times more power-
ful than TNT. It forms an eutectic with
TNT (52% by wt of NENO), melting at 61°C
& d 1.678 at 22°C, and with 1,3-DNB (62%)
by wt of NENO), melting at 61.8°C(Ref 16)
The expl & other props of NENO recorded in
various Refs below are as follows:

Explosion Temp, °C, 260-70° deflugr; 210°C
for 25 mg sample in 5 sec (Ref 20)
Heat of Combustion, 2024 cal/g at Cᵥ
(Refs 7, 10 & 14)
Heat of Explosion at 25°C, 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°C
25-30 mins (Ref 17)
Vacuum Stability Test at 100°C 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°C, 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, dinitrodime
thylamine 10 & dimethyloxalate 5%, which freezes at 72-73° to a homogeneous, hard, nonhygro, powerful exp1 fairly insensitive to shock & friction. VDlHerz (Ref 2) patented several exp1 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 sen
tive to friction.

Other uses of NENO have included its substitution for Tetly in boosters & de
tonators, 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. Naoum, GerP 56800(1931) & CA 27, 2814(1933) 4) A. Foulon, SS 27, 399

Diethylol Peroxide, Diethanol Peroxide or Bis(1-hydroxyethyl)-peroxide [called a.a-
Diory-diathylyperyox or Bis-(1-hydroxyethyl)-peroxyd in Ger], CH3.OH.OO.CH(OH).CH3; mw 122.12, O 52.41%; col, easily flowing oil having a pungent odor, bp - explodes on heating in a tube; can be prepbd by treating freshly distilled acetalddehyde with a 3% dry ehtereal soln of H2O2 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. Wingerl, 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, ArchExptPathPharm 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(hydoxyethyl)-piperazine in Vol 2, p B146-R

Diethylol-propylenediamine and Derivatives

\[ \text{N,N'-Diethylol-propylenediamine; N,N'-} \]
\[ \text{Diethanol-propylenediamine; N,N'-} \]
\[ \text{(2-hydroxyethyl)-propylenediamine or 2,2'} \]
\[ \text{(Propylenediino)-diethanol (CA nomenclature), CH}_3\text{NH.CH}_2\text{CH}_2\text{OH} \]
\[ \text{CH}(_3)\text{NH.CH}_2\text{CH}_2\text{OH; mw} \]
162.23, N 17.27%; liq, bp 202-04° at 20 mm, d 1.0463 at 20°, nD 1.4895 at 20°; prep. described by Tkaczyński (Ref 2). It may be considered as the parent compd. of its dinitramines although not used to prep them. 


N,N'-Diethyl-propylene-dinitramine or N,N'-Bis(2-hydroxyethyl)-propylene-dinitramine, 

CH2.N(NO2).CH2.CH2OH

CH(CH3).N(NO2).CH2.CH2OH; mw 252.23, N 22.21%; oil - no props given; was obtd by passing ethylene oxide gas thru stirred propylene-dinitramine 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-nitroxyethl)-propylene-dinitramine, 

CH2.N(NO2).CH2.CH2.OONO2

CH(CH3).N(NO2).CH2.CH2.OONO2; mw 342.23, N 30.92%; cryst (from alc), mp 78-81°; obtd by nitrating the above compd with 98% HNO3 at 5-10° (Ref 2). No exp. 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, 

O.NH.CH2.CH2.OH

S

O.NH.CH2.CH2.OH, 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 

O.N(NO2).CH2.CH2.OONO2

S

O.N(NO2).CH2.CH2.OONO2; mw 364.17, N 23.08%; crysts, exp. prep. by condensing sulfuryl chloride, (SO2Cl2), with ethanol amine, HO.C2H4.NH, and nitrating the product. This exp. 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'-Diethyl-oximid in Gerl, CO.NH.C2H5 

CO.NH.C2H5; 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 exp. deriv 

Refs: 1) Beil 4, 112, (352), [605] & [213] 

N,N'-Dinitro-N,N'-diethyloxamide, 

CO.N(NO2).C2H5

CO.N(NO2).C2H5; mw 234.17, N 23.93%; wh crysts (from alc), mp 34-35°, bp - explodes; readily sol in eth; diff 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 exp. Was recommended in Germany for use in blasting & military exps (Ref 2) 

Other Dinitroalkyloxamides have been prep'd as indicated in Refs 1 & 2 and claimed to be powerful & insensitive exps 

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 Diethyl-oxamide were found in Beil or in CA thru 1961

Diethyl Peroxide (called Diäthylperoxyd in Ger), 

C2H5 - O0 - C2H5; 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°, ng. 1.3715 at 16.5°; miscible with alc & eth; diff sol in w; was prep'd by Baeyer & Villiger (Ref 2) from diethyl sulfate, H2O2 & aq KOH soln (Ref 1) 

Diethyl Peroxide easily inflames when touched with an object preheated to 250°; explodes in an atmosphere of CO2 when
touched with a hot Cu wire; forms expl mixts with air; could not be exploded by impact. Its thermal decomp 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)

Diethyl Peroxydicarbonate,
\[ \text{H}_5\text{C}_2\text{O}_2\text{CO} - \text{OO} - \text{OCO.C}_2\text{H}_5; \text{mw} 178.14, \text{O} 53.89\%; \text{expl oil or solid possessing the sharp ozone-like odor of volatile acyl peroxides, mp 28-35°(dec), decomps spontaneously at RT, defl gr on contact with a flame; } n_d 1.4017 \text{ at } 20°; \text{sol in a wide range of org solvs; sl sol in w; obt in an impure form by Wieland et al (Ref 2) by reaction of ethyl chlorofomate in chlf soln with powdered Na peroxide. Strain et al (Ref 3) prep the cmpd by the same procedure but pointed out the special care that was reqd in temp control (0-10°) in all operations of prep & 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 Peroxide (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 & Me sobrian (1954), 170

Diethylphenylenediamine and Derivatives
Diethylphenylenediamine, \( \text{C}_{10}\text{H}_{16}\text{N}_2; \text{mw} 164.24, \text{N} 17.06\%. \) Four isomers are found in Beil:

\[ \text{N,N-Diethyl-phenylenediamine or 2-Amino-diethylaniline, H}_2\text{N.C}_6\text{H}_4\text{N(C}_2\text{H}_5)_2; \text{a strong smelling oil, bp 312.5^° at 744 mm press} \]
(Ref 2)

\[ \text{N,N-Diethyl-m-phenylenediamine, liq, bp 276-78^°} \]
(Ref 3)

\[ \text{N,N-Diethyl-p-phenylenediamine, liq rapidly turning brown on exposure to light, bp 260-62^°} \]
(Ref 4)

\[ \text{N,N-Diethyl-1,3-phenylenediamine, H}_5\text{C}_2\text{HN.C}_6\text{H}_4\text{NH.C}_2\text{H}_5; \text{no description given} \]
(Ref 1)

Other props & methods of prep are found in the Refs


Nitroderivatives of
Diethylphenylenediamine

4-Nitro-N,N'-diethyl-1,3-phenylenediamine,
\[ \text{H}_2\text{N.C}_6\text{H}_3(\text{NO}_2)_2.N(C}_2\text{H}_5)_2; \text{mw} 209.24, \text{N} 20.08\%; \text{lt-yel crys}, \text{mp 139°; was prepd by heating 3,4-dinitrodiphenylamine with alcoholic NH}_3 \text{ in a tube at 120°} \]
(Ref)

Ref: Beil 13, [30]

Dinitrodiethylphenylenediamine, \( \text{C}_{10}\text{H}_{14}\text{N}_4\text{O}_4; \) not found in Beil or in CA

2,4,6-Trinitro-N,N'-diethyl-1,3-phenylenediamine,
\( (\text{O}_2\text{N})_2\text{C}_6\text{H}(\text{NH.C}_2\text{H}_5)_2; \text{mw} 299.24, \text{N} 23.41\%; \text{yel crys}, \text{mp 142-44^°; was prepd by heating N-nitro-N-ethyl-2,3,4,6-tetranitroaniline with ethyamine in a tube at 50-60°, or from 3-chloro-2,4,6-trinitrophenol & ethyamine in alc. Its exp1 props were not reported} \)

Refs: 1) Beil 13, 61 & (17) 2) J.J. Blanksma, Rec 21, 325(1902)

Tetranitrodiethylphenylenediamine, \( \text{C}_{10}\text{H}_{12}\text{N}_8\text{O}_8; \) not found in Beil or in CA

N,N',2,4,6-Pentanitro-N,N'-diethyl-1,3-phenylenediamine,
\( (\text{O}_2\text{N})_2\text{C}_6\text{H}(\text{N(NO}_2)_2)_2.C}_2\text{H}_5)_2; \text{mw} 389.24, \text{N} 25.19\%; \text{col crys}, \text{mp 165°; was obtd by nitrating the above trinitro deriv (Refs 1 & 2). Its exp1 props were not reported} \)

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-phenylene-diamine; bis(Trinitroethylenimine-o-phenylene or o-bis(2,2,2-Trinitroethylamino) benzene, 
C₆H₄[NH.CH₂.C(NO₂)₃]₂; mw 434.24, N 25.81%; expd compd, mp 70 °C; was prep'd by condensing o-phenylenediamine & 2,2,2-
trinitroethanol, (O₂N)₃C.CH₂.OH, both in methanol, chilling the mix and ppg the 
product by dropwise addn of H₂O (Ref 2). The exph 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 Diethylphthalat or Phthal-
säure-diäthylerster in Ger), C₁₂H₁₄(OOC₂H₅)₂; 
mw 222.23, O 28.80%; clear, colorless lqd, 
fr p -40.5 °C, bp 295-308 °C; d 1.1268 at 15 °C, d 
1.0872 at 55.7 °C, nD 1.5049 at 16 °C, viscosity 
0.1006 g/cm sec at 25 °C; flash p 243 °F (CC); 
Q⁰combstn 6404 cal/g or 1423 kcal/mol; Q⁰ 
form 829 cal/g or 184 kcal/mol (Refs 1 & 4).

Diethylphthalate can be prep'd by the action 
of alcohol on phthalic anhydride in the 
presence of anhyd HCl, CaCl₂ or CuSO₄.

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 in-
gredient 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 °C 

Ester Content as diethylphthalate - 99.0% min 
Acidity as phthalic acid - 0.03% min 
Ash - 0.01% max

Its den 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, An alChem 20, 1066-67 (1948) & CA 
43, 1568 (1949) (Detn of DEPh in proplnts)

3) S. WachteII, "Investigation of Chromato-

graphic and Spectrophotometric Methods of 
Powder Analysis. Development of Chromato-

graphic-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) (Chromato-

graphic analysis of powders contg DEPh)

8) G. Norwitz, "Determination of Diethyl 
and Dibutyl Phthalates in Propellants", 
AnalChemAcca 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) PropIntManual 
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 den 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 StdMethodsChem-

Analysis (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 CuSO₄, stopper the flask and swirl occasionally while addig 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 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% K₂CO₃ soln to the second separatory funnel, shake, test with indicator paper to assure an excess of K₂CO₃, and transfer the methylene chloride to a 250-ml iodine flask. Wash the K₂CO₃ 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 K₂CO₃, 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 K₂CO₃
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 & Diocylphthalate=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 glacial acetic acid) 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 prep 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 (Sokhlet, Roweg or equi-
valent) to be extracted by the Method 1 04.1.3 of Ref, which is also described in Vol 2 of Encycl, p Cl 31-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 Diagram]

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 mix of silicic acid (Malnikrode's or equivalent) and Celite (Johns-Manville's Hydro-Supercei, 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, 3 V 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 1.0 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-methyl-
ene 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 origi-
nal 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 pro-
cEDURE (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 test indicates 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
ML-E-463, Grade 1) into the beaker to
dissolve the residue
u) Transfer the soln into a 100-ml volu-
metric 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 Spectropho-
trometer (Beckman Instruments Inc, Model DU,
or equivalent), determine the adsorbenece of
the soln at a wave length of 275 millimicrons
x) Calculate the percentage of DETPh in the
propellant using the following formula:
\[
\% \text{ DETPh} = \frac{(29950A)}{CW}
\]
where: \(A = \) Corrected adsorbence of pro-
plnt 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, tri-
acetin, or sucrose octaacetate

The method is the same as described
under "Dibutylphthalate, Analytical Proce-
dures", except that the formula for calcn
is different, namely:
\[
\% \text{ DETPh} = \frac{(11.1(A-B)N)}{W}
\]
where: \(A = \) Ml of standard hydrochloric acid
soln for blank titration
\(B = \) Ml of hydrochloric acid for
titratin of sample
\(N = \) Normality of standard hydro-
chloric 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 oxi-
dation.

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 carbonrubd (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

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). Re-attach 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 stop-
cock when the bottom of benzene layer in (D) reaches the 5-ml mark

q) 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 prep'd from K dichromate (Burr-StdsSample No 136) in 40-perc. (by wt) H2SO4.

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 compt desribed in the detailed Spec

w) Calculate the percentage of DETPh in the proplnt on a volatile-free basis as follows:

\[ \text{% DETPh} = \frac{[13.89(A-B)N]}{W-C} \]

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
Ml = [13.89(V-v)N] / W

N = Normality of Na thiosulfate soln
W = Weight of sample corrected for total volatiles, grams
Wb = 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)


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-pentene. See under Acetylene Hydroperoxides and Peroxides

3,6-Diethyl-1,2,4,5-tetraketo-4,5-dihydropyridazine-3,6-dicarboxylate or 4,5-Dihydro-4,5-dioxo-3,6-pyridazino-dicarboxylic acid-1,2-dioxide diethylester,

\[ \text{C}_2\text{H}_5\text{OOC.C.} \rightarrow \text{N.}(\text{O}) \rightarrow \text{N.(O)} \rightarrow \text{C.COOC}_2\text{H}_5; \]

\[ \text{C.(O)} \rightarrow \text{C.(O)} \]

mw 286.20, N 9.79%; crysfts., mp 70°, dec on long standing at RT, explodes when heated with Pt black; readily sol in acet & eth acet; diff sol in alc, eth, eth acid & benz; was obtd by reacting diethyl ketopine in eth with N2O4 at -15° (Ref 2)

Refs: 1) Beil - not found 2) E.J. Virgin, Diss, UnivUppsalas, 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-triazole,
\[
\begin{array}{c}
\text{H}_5\text{C}_2\text{C} & \text{NH} & \text{C} & \text{NH} & \text{C} & \text{C}_2\text{H}_5 \\
\text{N} & \text{N} & \text{N} & \text{N} & \text{N} & \text{N}
\end{array}
\]
mw 192.22, N 43.72%; crystals (from alc); sol in acids; sl sol in hot w & NH_3; was prep'd by heating dihydrazozone oxamide or oxalhydrazide, (C_2H_8N_6H_2O), with propionic anhydride, (C_2H_5CO)O, to yield the intermediate propionyl diethyltriazole (wh crystals, mp 192º) which on heating in HCl and adding 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 exp pls for the compd or its salts were detd

Refs: 1) Beil - not found 2) G. Dedichen, AnhanddNorskeVidensksaps-AkadOso, 1, Mat-NaturvKlasse 1936, No 5, 42 pp & CA 31, 4987 (1937)

Diethyltriazolyl-formamidine. See N,N'-Diethyltriazolyl-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],

\[
\begin{array}{c}
\text{Bi}_3\text{C}_6\text{H}_2\text{N}:\text{N}(\text{COO.C}_2\text{H}_5)\text{NH} \text{COO.C}_2\text{H}_5 \\
\text{mw 517.00, N 10.84%; ndls (from eth acetate + petr eth), mp 111-15º; turns bm on exposure to light; sol in alc, eth & benz; insol in water & petr eth; decomp in aq NaOH; was obt'ed by reaction of 2,4,6-tribromobenzene-diazonium salt & diethyl hydrazinedicarboxylate in the presence of aq Na acetate & Na carbonate (Refs 1 & 2)}
\end{array}
\]

Refs: 1) Beil 16, (417) 2) O. Dimroth & G. deMontmollin, Ber 43, 2910 (1910) & CA 5, 494 (1911)

_{N^1,N^2}-Diethylenetriethylene Tetranitramitro or 1,10-Diethy-1,4,7,10-tetranitrotetriethylene-tetramine, \CH_2\text{N(NO_2)}.{\text{CH}_2\.\text{CH}_2\text{N(NO_2)}.{\text{CH}_2}\.\text{H}_5
\]

\[
\begin{array}{c}
\text{CH}_2\text{N(NO_2)}.{\text{CH}_2\.\text{CH}_2\text{N(NO_2)}.{\text{CH}_2}\.\text{H}_5 \\
\text{mw 382.34, N 29.51%; crystals (from alc), mp 96.5-96.8º; sol in alc; insol in alkalies; gives a positive Frankimont test for nitroamine; was prep'd from a suspension of triethylenetetramitro in eth soln contg diazoethane, which reaction evolved gas rapidly, and on evaporation of the ether left a sirup which gradually solidified during four days (Ref 2). This is an expl compd}
\end{array}
\]

Refs: 1) Beil - not found 2) G.N.R. Smart & G.F. Wright, JACS 70, 3142 (1948) & CA 43, 570 (1949)

Diethyurea and Derivatives

_{1,3-Diethyurea or N,N'-Bis-ethyurea (called N,N'-Di-ethyl-harnstoff or Kohlensäure-bis-äthylamid in Ger), NH.C_2.H_5
\]

\[
\begin{array}{c}
\text{CO} \\
\text{NH.C_2.H_5; mw 116.16, N 24.12%; anisotropic lfts (from petr eth), ndls (from alc), mp 112º, bp 263º, d 1.0415; readily sol in w, alc & eth; can be prep'd from ethylamine & CO}_2 at 180-200º, and by many other methods (Ref) It may be considered as the parent compd of its hexamitro deriv}
\end{array}
\]

Ref: Beil 4, 115, (353), [608] & [218]

Azido, C_2H_11N_3O, and Diazido, C_2H_10N_2O, derivs were not found in Beil or in CA thru 1961

Mononitrito, C_5H_11N_3O_3, Dinitrito, C_5H_10N_4O_5, Trinitrito, C_5H_9N_4O_7, Tetrinitrito, C_5H_8N_4O_9, and Pentanitrito, C_5H_7N_3O_{11}, derivs were not found in Beil or in CA thru 1961

_{1,3-Di(2,2,2-trinitroethyl) urea; Hexanitro-diethyurea; or N,N'-Bis(beta,beta, beta-trinitroethy) urea, Code name BTNEU, NH.CH_2.C(NO_2)_3
\]

\[
\begin{array}{c}
\text{C:O} \\
\text{NH.CH_2.C(NO_2)_3; mw 386.16, N 29.09%; crystals, mp 187-91º (dec); d 0.5-0.6 g/cc,}
\end{array}
\]
Q_{combustn} 1645 cal/g; Q_{form} 70 kcal/mol (Ref 3); was first reported in the open literature by Schencck & Wetterholm (Ref 2) who prepd the cmpd by stirring 2 mols of tetrinitroethanol with 1 mol of urea in aq soln for 2 hrs at 80°. This cmpd was also prepd by addg dimethyloleurea 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 expld cmpd may be used in expl mixts as rocket propltnts, in pyrotechnic comprs, as Diesel oil additives, and as synthesis intermediates. See also Ref 1a.

A new type of gelatinized O-balanced safety expl, known as "Securite", 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(NO₃)₂ 58.7% was prepd (called A) and compared with a 35% NG Ammonium Diamnltite (called B). Heat of explnd, 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. Expld 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 evnl of red gas starting at 195°; B exploded at 195° and evolved red gas starting at 155°.


Diferrocenyl, Biferrocene or Bicyclopentadieneylene-bis[cyclopentadienyl iron]

C₅H₄—C₅H₄

Fe Fe

C₅H₅  C₅H₅; mw 370.14, Fe 30.18; orn crysts (from alc), mp - darkens at 205° & melts with decmpn at 230°; readily sol in benz; mod sol in petr eth, toluene, dioxane, tetrahydrofuran & alc; was formed by the decmpn of diferrocenyl mercury, (C₅H₅FeC₅H₄)₂Hg, in the presence of Pd black (Reps)


Differential Pressure Measuring for Examination of Propellants. This subject is discussed in a paper by H. Rumpf, 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 constant rate. Since a test 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.


**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:

Diffusion Coefficients of Solventless Propellants is determined by J. Chosson, MP 32, 255-78 (1950) & 33, 357-69 (1951)
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)

Di-fflamy (Welsh for Flameless). A propellant manifold 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

Diffuoranino Compounds. Some compds of interest in the field of exps are described in the following classified repts:
Refs: 1) Thiokol Chemical Corp, Reaction Motors Div, "Diffuoranino Chemistry", RMD Rept 076-5-62 (1 Jan to 31 Dec 1962) [Contract N001 1878(00)] (Conf) 2) B.J. Levy, "A Study of the Explosion Limits of Simple Diffuoranino 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 N001 4065(00)] (Conf)

Diffuorenyl-diperoxide. See 9,9'-Bis(diffuorenyl)-diperoxide in Vol 2 of Encycl, p B143-L

Diffuorenylidenehydro(tetrazine. See 3,6-Bis(diffuorenylidenehydro)-sym-tetrazine in Vol 2, p B143-L

Diffuorobenzene and Derivatives
Diffuorobenzene (called Diffuor-benzol in Ger), C6H4F2; mw 114.09. Three possible isomers are known:
1,2-Diffuorobenzene, aromatic smelling liq, fr p -34°, bp 93° at 759 mm press, d 1.1496 at 25°, nD 1.4451 at 18°; decomp in a steam of air (Ref 1)
1,3-Diffuorobenzene, liq, fr p -59°, bp 83.1°, d 1.1572 at 20°, nD 1.4404 at 18° (Ref 2)
1,4-Diffuorobenzene, liq, fr p -26.7 to -3°, bp 82-83° at 752 mm & 88.95° at 763 mm press, d 1.1722 at 18.9°, nD 1.4422 at 18.9°; Qcomb 704.3 kcal/mol (Ref 3)

Other props & methods of prepn are given in Beil

Azido, C6H3F2N3, and Diazido, C6H3F2N2, derivs of Diffuorobenzene were not found in Beil or in CA thru 1961
Mononitrodifluorobenzene, C6H3F2.NO2; 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 14 mm press (Ref 2); bp 76-80° at 11 mm press, nD 1.5055 at 20° (Ref 5)
4-Nitro-1,3-difluorobenzene (called 2,4-Difluor-1-nitro-benzol in Ger), liq, fr p 9.7°, bp 206.9° at 13.9°, nD 1.5149 at 13.9° (Ref 1) & nD 1.5150 at 20° (Ref 5)
2-Nitro-1,4-difluorobenzene (called 2,5-Difluor-1-nitro-benzol in Ger), yel liq, fr p 11.0°, bp 206.5°, d 1.4671 at 17.2° & 1.4571 at 13.9°, nD 1.5149 at 13.9° (Ref 1)

Other props & methods of prepn are found in Beil
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)
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**, C₆H₂F₂(NO₂)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, Melliland-
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,
IIEC 45, 22869335) & CA 48, 307 (1954)
(Used to protect US Air Force fabrics against
fungus attack) 4) H.Zahn & A. Würz, Bio-
chemZ 325, 182 (1954) & CA 49, 3864 (1955)
(Reactions of amine, imine, phenol, thiol &
imidazole with DNFFB) 5) H. Zahn & A.
Würz, TextileRes J 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) (Janovsky
reaction) 7) Farbwerke Hoechst A-G, vorm
Meister Lucius & Brüning, BritP 75568 (1956)
& CA 51, 9686 (1957) (Use of DNFB as wood
preservative, pesticide & dye intermediate)
8) N.N. Vorozhtsov & G.G. Yakobson,
NauchDoklVysshShkoly, Khim i Khim-
TeckhnoL 1958, No 1, 122 & CA 53, 3110
(1959) (Prepn from corresponding dichloro
deriv) 9) K. Fukui et al, NipponKagakyo-
Zasshi 79, 1129 (1958) & CA 54, 5518 (1960)
(Prepn in 88% yield) 10) T.L. Fletcher et
al, JOC 25, 1346(1960) & CA 54, 24591 (1960)
(Prepn, mp 73-74°)

**Dinitrodifluorobenzene** Difluoroxide, C₆H₂F₂NO₂,
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 (?),
C₆HF₂N₂O₃; mw 201.09, N 20.90%;

\[
\begin{align*}
\text{F} & - \text{C} \equiv \text{C} \equiv \text{C} \equiv \text{O} \\
\text{HC} & \equiv \text{C} \equiv \text{C} \equiv \text{O} \\
\text{N} & \equiv \equiv \equiv \equiv \equiv \equiv \\
\end{align*}
\]

**N**=**N**

**F.C——C≡C.NO2**

**O——C——C≡C.NO2**

**HC——C≡C.F**

(bright orange-colored
rystals from MeOH), mp - darkens on heating
w/o melting up to 350°; explodes violently
on impact; was obt by diazotizing 2-nitro-
3,4,6-trifluoroanilino in conc HCl with aq
NaNO₂. The ppt melted near 80°, burned
explosively in a flame, and on crys from
MeO gave the above product (Ref 2)

3,4-Difluoro-2-nitro-1-diazo-6-oxide (?),

\[
\begin{align*}
\text{N} & \equiv \equiv \equiv \equiv \equiv \equiv \\
\text{O} & \equiv \equiv \equiv \equiv \equiv \equiv \\
\text{H} & \equiv \equiv \equiv \equiv \equiv \equiv \\
\end{align*}
\]

**N**=**N**

**O——C——C≡C.NO2**

**HC——C(F)≡C.F**

(bright orange crystals
from MeOH), mp 119-120° 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 chlorine atoms
*Refs: 1) Beil - not found 2) G.C. Finger
et al, JACS 73, 148 (1951) & CA 45, 7034
(1951)*
1,3-Diformodinoguanidine,
NH.NHCHO
\[ C\equiv\text{NH} \]
\[ \text{NH.NHCHO; mw 145.13, N 51.02%; was isolated in the form of its nitrate salt, C}_3\text{H}_7\text{N}_4\text{O}_5, \text{silky wh ndls (from alc); mp 155}\degree\text{C (5% yield), in addn to 3,4-diamino-1,2,4-triazole,} \]
\[ \text{C}_2\text{H}_6\text{N}_6\text{O}_3, \text{wh ndls (from alc), mp 198-99}\degree\text{C, when dianimoguanidine nitrate & formic acid in dioxane were refluxed on a steam bath for 2 hrs (Ref 2)} \]

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),
\[ \text{NH.CHOO; mw 88.07, N 31.81%; prisms, mp} \]
\[ 159-60\degree\text{C; can be prepd by heating, for several hrs at 100-30}\degree\text{C, hydrazine hydrate with an excess of the ethyl ester of formic acid, or by heating at 100}\degree\text{C hydrazine sulfate & formylhydrazine in abs alc; readily sol in w; diffe sol in alc; insol in eth (Refs 1, 4 & 5)} \]

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 sohn of disodium diformylhydrazine with neutral lead acetate (Ref 3)
Mericorous salt, HgC_2H_2N_2O_2, fine wh ppt; obtbn from diformylhydrazine, Na methylate & mercuric 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 alc; was prepd by mixing an ice-cold aq sohn of diformylhydrazine with Na ethylate (Ref 2)


Di-2-furoyl Peroxide or Furoyl Peroxide (called Difurfuroyloperoxyd or Furufuroylperoxyd in Ger),
\[
\begin{array}{c}
\text{HC} \\
\text{CH} \\
\text{O} \\
\text{HC} \\
\text{C} \\
\text{O} \\
\end{array}
\]

2; mw 178.14, O 53.89%;
lt-yel ndls (from alc), mp 86-87\degree\text{C, darkens on exposure to light; explodes violently on heating in a flame, or on impact or friction; sol in eth, acet methanol, chlfr, CCl_4 or benz; insol in w or petr eth; can be prepd by reacting aq Na peroxide & furoyl chloride at 0\degree\text{C (Refs 1, 2 & 3). Cooper (Ref 4) studied the effect of structure of discyl peroxides on their radical-induced decomp in vinyl monomers}}

Ref: 1) Beil 18, [267] 2) H. Gelissen & J.D. van Roon, Rec 43, 362 (1924) & CA 18, 2515 (1924) 3) N.A. Milas & A. McAleavy, 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-I.

Difuranylacroxy1 Peroxide or Bis(β-2-furyl-acroxy1) Peroxide (CA nomenclature)
\[
\begin{array}{c}
\text{HC} \\
\text{CH} \\
\text{H} \\
\text{H} \\
\text{O} \\
\text{HC} \\
\text{CH} \\
\text{C} \\
\text{C} \\
\text{O} \\
\end{array}
\]

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 ac & ethyl acet; sl sol in eth; was synthesized from Na peroxide & furylacryloyl chloride in anhyd eth soln at 0°(Ref 2)

Ref's: 1) Beil - not found 2) N.A. Milas & A. McAlvey, JACS 56, 1219 (1934) & CA 28, 4443 (1934) 3) Not found in later CA's thru 1961

Diglycerol and Derivatives

Diglycerol; Diglycerin; Diglyceryl Ether; Diglyceryl Alcohol; Bisdihydroxy propylxide; or 3,3'-Oxy-di(1,2-propanediol) (CA nomenclature) [called Diglycerin; Diglycerin-1-(2,3-dihydroxy-propyläther) or Bis-(2,3-dihydroxy-propyl)-äther in Ger],

\[
\text{CH}_2\text{CH(OH)}\text{CH}_2\text{OH}\quad \text{CH}_2\text{CH(OH)}\text{CH}_2\text{OH}
\]

CH₃CHOH.CH₂OH

viscous, hygro liq, bp 265-70° at 14-15 mm press, d 1.279 at 20°, viscosity at 50° 7.316 g/cm sec, mp 1.489 at 20°; v sol in hot water; sl sol in cold w; insol in eth; can be prep'd by gently boiling glycerin for 5-6 hrs & distilling off the water; by heating glycerin to 290-95° in the presence of 0.5% Na₂CO₃ or Na₂S; by the oxidation of diallyl-ether with KMnO₄ or H₂CO₃; or by other methods (Ref 1) See Refs

It is used for the prep of its expl tetranito deriv


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),

\[
\text{O}_2\text{N.O.CH}_2\text{CH(O.NO}_2\text{).CH}_2\text{)}
\]

mp 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 prep'd 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 expl - 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 NH₄ClO₄ or KClO₄ 21-30, MnO₂ 4-7, NaNO₃ 17-47.5, TNT 23-26 & TeNDG, NG or Dinitrochlorohydrin 2%, plus small amts of CaCO₃, S or woodmeal added


Diglycerolpentaerythritol Ether, C₁₁H₂₄O₈₃

mw 284.30, O 45.02%; crys, mp 72.5-74°, softens at 69° when heated rapidly; prep'd by hydroxylating diallyl pentaerythritol ether (Ref 2); is the parent compd of its hexanitrate deriv

Diglycerolpentaerythritol Ether Hexanitrate, C₁₁H₂₂O₈₅

\[
\text{(O}_2\text{NO)}_2\text{H}_2\text{C}_3\text{–O–H}_2\text{C–C–CH}_2\text{–O–C}_3\text{H}_5\text{O}_2\text{NO}_2\text{)}_2\quad \text{CH}_2\text{ONO}_2
\]
mw 554.30, N 15.16%; crystals, d 1.540 at 20°, 
nb 1.4878 at 20°; was prep'd by nitrating the 
parent compd (Ref 2). It is a possible gelu-
tinizer for NC
Refs: 1) Beil - not found 2) R. Evans & 
J.A. Gallagher, JACS 75, 1249(1953) & CA 
49, 3811(1955)

Diglycol (Diglykol in Ger). Same as Diethy-
leneglycol (DEG or DEGc).

Diglycolic Acid or Diglycolic Acid Mono-
amide may be represented as

![CH₂CO₂H](O\(\text{CH₂CONH₂}\))  
(See also Diglycolamidic Acid)

called Dimethyldithet-\(a, a'\)-dicarbonsäure-mono-
amid; Diglykolsäuremonooamid; or Diglykol-
amidsäure in Ger), mw 133.10, N 10.52%; 
prisms, mp 135°; sinters at 125-30°; par-
tially sol in w & alc; forms a Barium salt, 
Ba(C₄H₄N₂O₄)₂ + H₂O, crysrs; sol in w; insol in 
alc (Ref). Other props & method of prep 
are found in Beil 
Ref: Beil 3, 241-42

**NOTE:** No Azido, C₆H₆N₄O₄, or Nitrating, 
C₄H₄N₂O₆, derivs were found in Beil or in 
CA thru 1961

Diglycolamidic Acid and Derivatives

Diglycolamidic Acid or Iminodiacetic Acid, 
may be represented as HN(CH₂COOH)₂. 
Some of its derivatives are explosive. Eg: 
Diglycolamidic Diazeide (qv) and Nitroso 
derivs (qv)

Diglycolamidic Diazide, Iminodiacetic Axide or 
Iminosisoceticoxide (Diglykolamidsäure-
diazid or Iminodissigsäure-diazid in Ger), 
formerly called Diglycolamic Diazide 
HN(CH₂.CO.N₃)₂. Its nitrite, C₄H₅N₇O₂ + 
HNO₂, colorless needles (from eth), was ob-
tained (Ref 2) in small quantity by treating 
the trihydrochloride of diglycolamic acid 
dihydrizide with NaNO₂ in qg soln. The 
nitrite exploded on heating and decomposed in 
storage
crystals (from w), monoclinic prisms, mp 148°, 
bp - dec; forms a non-hygro 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:

$$
\text{OC(NH}_2\text{)}_2 + (\text{HOOC.CH}_2\text{)}_2\text{O} = 2\text{H}_2\text{O} + \text{OC(NH.CO.CH}_2\text{)}_2\text{O}
$$

$$
\text{OC(NH.CO.CH}_2\text{)}_2\text{O} + 6\text{HCHO} + \text{Ca(OH)}_2 = \text{OC(N(CH}_2\text{OH)}_2\text{)}_2\text{O.COC(CH}_2\text{OH)}_2\text{O}
$$

$$
\text{OC(NH.CO.CH}_2\text{)}_2\text{O} + 10\text{HCHO} + \text{Ca(OH)}_2 = \text{OC(N(CH}_2\text{OH)}_2\text{)}_2\text{O.COH.OH.C(CH}_2\text{OH)}_2\text{O}
$$

which on nitration of the hexamethylol &
octamethylol compds should yield hexa-
nitrate & octanitrate derivs that would be
powerful exps

Diglycolic Acid is now used in the manuf
of resins, plasticizers, and in org synthesis
(Refs 3 & 4)
Refs: 1) Beil 3, 234, (90) & 1276 2) H.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

DIII[1(3-guanyltriazene)-5-tetrazoly]-diazene or
Bis[1(3-guanyltriazene)-1-tetrazoly]-diazene
(called Bis-1,1(3-guanyltriazene)-5,5-aizotetra-
zole by Lieber & Smith; and Azotetrazol-
diazenitriunidin by Thiele),

\[
\text{H}_2\text{N.(HN:)C-NH-N:}: \quad \text{N:N-}: \quad \text{N:N-}: \quad \text{N:N-}: \quad \text{N:N-}
\]

\[
\text{H}_2\text{N.(HN:)C-NH-N:}: \quad \text{N:N-}: \quad \text{N:N-}: \quad \text{N:N-}
\]

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

DII(hexahydrobenzozyl)-peroxide. See Bis(hexa-
hydrobenzozyl)-peroxide in Vol 2, p B143-L

Dihyrazide-dihydro-tetrazine. See 3,6-Bis
(hyrazide)-1,2-dihydro-1,2,4-tetrazine in
Vol 2, p B143-R

DII(hyrazino)-benzene, Dinitro. See 1,3-Bis
(hyrazino)-4,6-dinitrobenzene in Vol 2, p
B143-R

Dihydrizinotoluene, C_7H_12N_4, may be con-
sidered 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),

\[
\begin{align*}
\text{H}_2\text{N}.\text{HN}.\text{C} & \text{C(NO}_2\text{)=C.NH}_2\text{;} & \text{mw} 242.20, \\
\text{N} & 34.70\%; \text{dk red ndls} + 2\text{H}_2\text{O (from di alcohol),} \\
\text{mp} - \text{dec} & \text{at } 173^\circ; \text{was obtd by heating 3,5-} \\
dibromo-2,4-dinitrotoluene with hydrazine- 
hydrate in alc on a water bath (Refs 1 & 2) 
\end{align*}
\]


\text{NOTE: } \text{No Azido, } \text{C}_7\text{H}_{11}\text{N}_7\text{, or Trinitro, } \text{C}_7\text{H}_{9}\text{N}_7\text{O}_6\text{, derivs were found in Beil or in } 
\text{CA thru 1961}

\text{Dihydrazone Oxide (called Oxalidimid-} 
säure-dihydrizid, "Carbohydrazinin" or 
"Cyanhydrazin" in Ger), 
(H_2N.N)\text{H}.\text{N}.\text{C}(\text{NH}_2\text{)}\text{.C}(\text{NH})(\text{NH}_2\text{)} \text{or} 
(H_2N.HN).\text{HN}.\text{C}(\text{NH})(\text{NH}_2\text{)}; 
\text{mw } 116.13, \text{ N } 72.37\%; \text{ndls} + \text{H}_2\text{O (from w} 
\text{or } 20\% \text{ alc), mp - dec; sol in water, but de-} 
\text{comp giving a wh ppt; insol in org solvs;} 
\text{can be prep by slowly introducing (CN)}_2 
\text{into N}_2\text{H}_4\text{.H}_2\text{O & cold 50\% alc, or in 60\%} 
\text{yield from dithioxamide in 20\% alc and} 
\text{N}_2\text{H}_4\text{.H}_2\text{O added dropwise, and quan-} 
\text{titatively from } \text{[C}(\text{NH})\text{.OC}2\text{H}_4\text{]}_2 \text{& excess} 
\text{N}_2\text{H}_4\text{.H}_2\text{O (Refs 1 & 2)}

\text{Dihydrazone Oxide forms salts, such as: } 
\text{Hydrazochloride, } \text{C}_2\text{H}_8\text{N}_6\text{.2HCl, yel ndls, swells w/o melting, more stable than its parent compd} 
\text{Nitrate, } \text{C}_2\text{H}_8\text{N}_6\text{.2HNO}_3\text{, mother-of-pearl-} 
crystalline; 
\text{Sulfate, } \text{C}_2\text{H}_8\text{N}_6\text{.H}_2\text{SO}_4\text{, wh crysts} 
\text{and other salts similar to those of NH}_3 \text{&} 
\text{Hydrazine Hydrate, some insol & others with charactristic colors (Ref 2)}

\text{The expl props of the compd & of its} 
\text{salts were not detd}
\text{Refs: } 1 \text{) Beil 2, 560 2) G. Dedichen,} 
\text{Avhandlong norske videnskaps-Akad Oslo, I,} 
\text{Mar-Naruv, Klasse 1936, No 5, 42 pp & CA} 
\text{31, 4985 (1937)}

\text{Dihydro- A prefix indicating the presence} 
of two additional hydrogen atoms in an or- 
ganic compd

\text{Di(hydroanthranol) or Dihydroxy-di(hydro-} 
anthracene). \text{See Bis(hydroanthranol) in} 
\text{Vol 2, p B144-L}

\text{4',4-Dihydro-5',5'-azinotetrazole; Azino-} 
di[(4-hydroxytetrazole)-5]; \text{(Ditetrazolyl-5)-} 
\text{hydrazine; 5,5'-Hydrazo-ditetrazole; or} 
\text{Hydrazo-di(tetrazoly1-5). See Bis(5-tetra-} 
zole)-hydrazine in Vol 2, p B157-R & in} 
\text{the paper of F.R. Benson, ChemRev 41,} 
\text{8(1947)}

\text{Dihydrodiketonebenzene; Dioxybenzene or} 
\text{Quinone. See Benzoquinone in Vol 2, p} 
\text{B79-R}

\text{1,3-Dihydro-2,2-dimethylperimidine [called} 
\text{2,2-Dimethyl-2,3-dihydro-perimidin or N.N'-} 
\text{Isopropyliden-naphthylendiamin-(1,8) in Ger],} 
\text{C}_1\text{H}_4\text{N}_2\text{; mw } 198.26, \text{ N } 14.13\%; \text{ndls (from w,} 
\text{mp } 117^\circ, \text{may be considered the parent} 
\text{compd of its tetrainodoteriv} 
\text{Ref: Beil 23, 205 & 51}

\text{1,3-Dihydro-2,2-dimethyl-4,6,7,9-tetranitro-} 
\text{perimidine or 4,6,7,9-Tetranitro-dimethyl-} 
dihydroperimidine,

\[
\begin{align*}
\text{H}_3\text{C} & \text{CH}_3 \\
\text{HN} & \text{C} \text{NH} \\
\text{O}_2\text{N.C} & \text{C} \text{C} \text{C} \text{C} \text{C.NO}_2 \\
\text{HC} & \text{C} \text{C} \text{C} \text{C} \text{CH} \\
\text{NO}_2 & \text{NO}_2 & \text{; mw } 378.26, \\
\text{N } 22.22\%; \text{orn-yel plates (from acet), mp } 280^\circ 
\text{(dec); was obtd by tetracoxisation of the} 
\text{parent compd by nitrosylsulfuric-glacial} 
\text{acetric acid (Ref 2)}
\text{Refs: } 1 \text{) Beil - not found 2) H.H. Hodgson} 
\text{& J.S. Whitehurst, JCS 1947, 81 & CA 41,} 
\text{3452 (1947)}
Dihydroimidazolone. Same as Imidazolidone

5,6-Dihydro-5-imino-2,4(1,3H)-s-triazinedione. See Anmelde in Vol 1, p A273-L.

4,5-Dihydro-5-isonitro-4′-methyl-4-methylene-1,1′,5′-trinitro-2,2′-biimidazolyl Methyl Ester or 4-Methyl-4′-methylene-1,1′,5′-trinitro-2,2′-biimidazolyl-5′-nitronic Acid Methyl Ester,

\[ \text{H}_3\text{C}=\text{C}=\text{N}=\text{N}-\text{O}_2 \text{N}=\text{C}=\text{N}-\text{O}_2 ; \]
\[ \text{H}_2\text{C}=\text{C}=\text{N}-\text{N}=\text{C}=\text{C}=\text{C}=\text{N}-\text{C}=\text{CH}_3 ; \]

m.w 356.21, N 31.46%: ltr-red crysts + H_2O, mp - 127° (dec), was obtbd as one product when the Na salt of tetranitro-biimidazolyl was shaken with (CH_3)_2SO_4 at RT (Ref 2)

Refs: 1) Beil - not found 2) K. Lehmscht, Ann 507, 224-25 (1933) & CA 28, 767 (1934)

1,2-Dihydro-1-methyl-6-nitroiminopyridine,
(called 5-Nitro-2-methylaminopyridine in Ger), \( \text{NC}_6\text{H}_4\text{N}(\text{NO}_3)\cdot\text{N}(\text{CH}_3)\cdot\text{NO}_2; \) m.w 198.14, N 28.28%: crysts (from w) or greenish plts (from petr eth), mp 59-60°; was obtbd by nitrating 5-nitro-2-methylaminopyridine, or in addn to other products by the action of (CH_3)_2SO_4 on 5-nitro-2-nitroaminopyridine in 10% NaOH or K_2CO_3 soln (Refs 1, 2 & 3)

Refs: 1) Beil 22, [338] 2) A.E. Tschitschibabin (Chichibabin) & R.A. Konowalow, Ber 58, 1716 (1925) & CA 20, 396 (1926) 3) A.E. Tschitschibabin (Chichibabin) & A.V. Kissananow, Ber 61, 1225 (1928) & CA 22, 4125 (1928)

Dihydropentaborane. See Pentaborane, Unstable, under Boron Hydrides in Vol 2, p B254-L.

Di(hydroperoxy)cyclohexyl-peroxide or Di(hydroperoxy)-dicyclohexyl-peroxide.
See 1,1′-Bis(hydroperoxy)cyclohexyl-peroxide in Vol 2, p B144-L.

1,1′-Dihydroperoxy-1,1′-dicyclohexylacetatelene.
See Vol 1, p A66-R under Acetylene Hydroperoxides and Peroxides

Di(hydroperoxydimethyl)-hexene. See 2,5-Bis(hydroperoxy-2,5-dimethyl)-hexane in Vol 2, p B144-R

1,6-Dihydroperoxy-(1,6-endoperoxy)-cyclodecone (?),

\[ \text{OOH} \]
\[ \text{H}_2\text{C}-\text{C}-\text{CH}_2 \]
\[ \text{H}_2\text{C} \]
\[ \text{O}-\text{CH}_2 \]
\[ \text{H}_2\text{C} \]
\[ \text{O}-\text{CH}_2 \]
\[ \text{H}_2\text{C}-\text{C}-\text{CH}_2 \]

m.w 250.24, O 44.76%; ndls
(from benz), mp 130°(dec), explodes when heated in a flame; instantly liberates I2 from KI and gives 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)

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 Fluorenone (Adduct). See 9,9'-Bis(hydroperoxy)-9,9'-fluorene + 2 Fluorenone (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),

HC — O —— CH2
HC—CH2 —— CH2; mw 84.11, O 19.02%; color mobile liq, having an ether-like odor, fr p -70°, bp 84.3°; flash p (Tag CC) 5 F, d 0.927 at 20°, nD 1.4180 at 25° (can be prepared by the catalytic dehydration of tetrahydrofurfil alcohol (Ref 3)
Fedoroff (Ref 2) detd that Dihydropyran completely gelatinized NC of 11.95 & 12.65% N contents. Grades of NC of higher N content were only partially gelatinized

6,7-Dihydro-5H-pyrrolotetrazole or Trimethylene-tetrazole,

H2C
CH2
C=N

H2C ——— N ——— N; mw 110.12, N 50.88%, colorless (from xylene), mp 110°; previously obtd by an intramolecular cyclization of y-azidobutyronitrile in chlf when treated with ClSO2H (Ref 4); was recently prepared by reacting 2-methylmercaptoxproline & a chlf soln of Hydrazoic Acid (Ref 5). Château (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

1,2-Dihydro-sym-tetrazine (called 1,2-Dihydro-1,2,4.5-tetrazin in Ger),

HC — NH —— NH

N — N ——— CH2; 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 prepared by reduction of sym-tetrazine with H2S in coldaq soln, or with Zn dust & glacial acetic acid in ether; and from hydrazoformaldehyde-hydrazone by soln in dil glacial acetic acid (Refs 1,2,3 & 4). Its expl props were not detd
1.2-Dihydro-sym-tetrazine-3,6-dicarboxylic Acid [called 1.2-Dihydro-1.2.4.5-tetrazin-dicarbonsäure-(3,6); Bis Diazoeussigsäure or “Triazoeussigsäure” in Ger], HOOC—C—NH—NH

N —N —C.COOH; mw 172.10, N 32.56%; ye! pils + 2H2O (from w) or anhyd ye! prisms, mp 149-55° (dec), when heared on a Pt foil it burns with hissing as a pro-plant; sol in acet & alc; insol in chlf, eth, CS2, 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 HNO2, some carmine and crysists, mildly expl, were formed. It forms numerous salts, some of which are unstable & expl when heated, Eg: Silver salt, Ag2C4H2N4O2, It-yel powd, explodes on heating; insol in w (Ref 2)

Refs: 1) Beil 26, 567-68  2) T. Curtius & J. Lang, J.PraktChem 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-dicarboxyl 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.

HC—NH—NH

N —NH —C:O; mw 100.08, N 55.99%; crysists (from alc), mp 187-88°, bp 315°; was obcd when (NH)22H2O was heated 4 hrs at 175° under 900 arm of CO. This compd is reported to be useful as a synthetic resin intermediate, antishinking agent for wool & rayon, and as a paper shrinking (Ref 2)

Refs: 1) Beil - not found  2) H.J. Sampson, USP 2589290 (1952) & CA 46, 11234 (1952)

Dihydro-triazone-diol, C6H3N3O2; mw 115.09, N 36.51%. Two isomers are described in the literature:

5,6-Dihydro-s-triazone-2,4-diol (called 2,4-Dioxo-hexahydro-1.3.5-triazin in Ger)

H2C—N —C.C.H  H2C—NH —C:O

HN—C(OH) —N  HN—C:C:O —NH

crysts (from formic acid), mp 245° (Refs 2 & 4) 1,6-Dihydro-as-1.2.4)-triazone-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),

H2C—NH —N  H2C—NH —NH

HO.C—N —C.C.OH  O.C—NH —C:O;

prismatic plates (from w), mp 221°; readily sol in w; diffic sol in alc; insol in common org solvs (Refs 1,3 & 4)


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; Acetyl-resorcin; Resacetophenon or Methyl-(2,4-dioxyphenyl)-keton in Ger],

HC—CO.CH —C.OH

HC—C(OH) —CH  ; mw 152.14, O 31.55%; ptls or ndls, mp 141-47°; prep & other props are found in Beil (Ref 1). Its 5-Nitro-, mp
D 1268

142°; 3,5-Dinitro-mp 166-67°; and 2,4,6-
Trinitro-dervs were prepd by Jadhav &
Merchant (Ref 2)

Refs: 1) Beil 8, 266, (613) & [294] 2)
G.V. Jadhav & J.R. Merchant, IndianChem-
Soc 28, 265 (1951); J Univ Bombay, Sect A,
19, Part 5 (Science No 29), 45 (1951) & CA
47, 6375, 6376 (1953)

DIHYDROXYANTHRACINONE
AND DERIVATIVES

Dihydroxyanthraquinone (called Dioxo-
antrachinon in Ger), HO.C6H3(CO)2C6H3.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- Purpuranthenin or Xanthopur-
parin (Ref 2)
1,4-Dihydroxy- or Quinizarin (Ref 3)
1,5-Dihydroxy- or Anthracurin (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 trivial 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
acetic acid, pyridine & higher-boiling aromatic
solvs are used to crystallize them; they dis-
solve in alkali & concd H2SO4. Addn of
boric acid to their sulfuric acid solvs gives
rise to distinctive color changes which are
helpful in characterizing the various hydroxy-
antraquinones (Ref 10). Other props and
methods of prep 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 & Other, 2nd
edit 2 (1963), pp 465ff by A.J. Cofrancesco

Nitro Derivatives of
Dihydroxyanthraquinone

Mononitro-dihydroxyanthraquinone,
O2N.C6H3(CO)2C6H3.OH2; mw 285.20,
N 4.91%. The following isomers are found
in Beil:
3-Nitro-1,2-dihydroxy- or 3-Nitro-alizarin,
long och-yel ndls (from benzol) or yel plts
(from glacial acetic acid, alc or chlf), mp 244°
(dec) (Ref 1)
4-Nitro-1,2-dihydroxyanthraquinone, golden-
yel ndls (from alc or glacial acetic acid), mp 289°
(dec) (Ref 2)
2-Nitro-1,4-dihydroxy- or 2-Nitro-quinizarin,
red ndls (from nitrobenzol or glacial acetic acid),
mp - not reported (Ref 3)
5-Nitro-1,4-dihydroxyanthraquinone, red
crystls (from glacial acetic acid), mp 244-45°
(Ref 4)
4-Nitro-1,8-dihydroxy- or 4-Nitro-chrysazin,
orn-yel crysts (from chlorobenzol), mp 232-34°
(Ref 5)
1-Nitro-2,3-dihydroxy- or 1-Nitro-bystazarin,
yel crysts (from toluene), mp - not reported
(Ref 6)

Other props & methods of prep 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

Dinitro-dihydroxyanthraquinone,
HO.C6H2(NO2)2(CO2)C6H2(NO2).OH; mw 330.20,
N 8.48%. Five isomers are found in Beil:
X, X-Dinitro-1,3-dihydroxyanthraquinone, Yel-
ndls (from glacial acetic acid), 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, Yel-
cryst flaks, mp - not reported (Ref 3)
1,4-Dinitro-2,3-dihydroxyanthraquinone,
C6H4(CO)2C6(NO2)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 prep 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, C14H5N3O10,
not found
Tetrinitrodiethyleneanthraquinone, C_{14}H_{4}N_{4}O_{12}; mw 420.20, N 13.33%. The following isomers are described in the literature:

2,4,6,8-Tetranitro-1,5-diethyleneanthraquinone or 2,4,6,8-Tetranitroanthraquinin,
HO.C_{6}H(NO_{2})_{2}(CO)_{2}C_{6}H(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-diethyleneanthraquinone,
structure as above, yel crystals, mp - not reported (Ref 2)
2,4,5,7-Tetranitro-1,8-diethyleneanthraquinone;
2,4,5,7-Tetranitro-chrysazin or Chrysazinic Acid;
structure as above, golden yel shiny plates, (from theaq 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_{2}N_{4}O_{12}; brick-red crystals, explodes on heating; nearly insol in alc & water
Calcium salt, CaC_{14}H_{2}N_{4}O_{12}; golden ndls, explodes on heating; sol in alc
Copper salt, CuC_{14}H_{2}N_{4}O_{12} + 4H_{2}O, dk-red crystals, loses water on heating and then explodes
Lead salt, PbC_{14}H_{2}N_{4}O_{12} + 5H_{2}O, brick-red crystals, loses water on heating and then explodes
Potassium salt, K_{2}C_{14}H_{2}N_{4}O_{12}; greenish shiny crystals, explodes on heating; diffic sol in water
Silver salt, Ag_{2}C_{14}H_{2}N_{4}O_{12}, dk-brn crystals, explodes on heating
Sodium salt, Na_{2}C_{14}H_{2}N_{4}O_{12} + 3H_{2}O, possesses same props as K salt
1,3,5,7-Tetranitro-2,6-diethyleneanthraquinone, 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,XX,X-Tetranitro-2,7-diethyleneanthraquinone, C_{14}H_{4}O_{2}{(NO_{2})_{4}}(OH); yel shiny plates (from dil HNO_{3}), mp >300°; almost insol in benz; sl sol in eth; v sl sol in glcer ac & HNO_{3}; sol in alkal; forms salts, some of which are probably expl (Ref 5)
X,XX,X-Tetranitro-X,7-diethyleneanthraquinone, yel crstls, mp >300°; sol in benz; sl sol in alc (Ref 6)

Methods of prepn & other props are given in Beil


Dihydroxyazoxybenzene and Derivatives. See under Azophenol (Azodiphenol) in Vol 1, pp A657-A658. Its nitrated derivative:
Hexanitrodiethyleneazoxybenzene 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

p-Dihydroxyazoxybenzene Dichromate,
[HO.C_{6}H_{4}.N_{2}.Cr.O_{7}]; N 12.73%; yel crstls; 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 Azophenol in Vol 1, p A671-L.

DIHYDROXYBENZALDEHYDE AND DERIVATIVES

Dihydroxybenzaldehyde (called Dioxyn-benzaldehyde in Ger), (HO)_{2}C_{6}H_{3}.CHO; mp 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-Dihydroxy-, 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 crstls (from water), mp 153-56° (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
Nitro Derivatives of Dihydroxybenzaldehyde
Mononitrodihydroxybenzaldehyde,
(HO)₂C₆H₂(NO₂)·CHO; mw 183.12, N 7.65%. Some of the possible isomers are found in the literature: 5-Nitro-2,4-dihydroxy-, yellow prisms (from benz), mp 146-49º (Ref 1 & 5); 2-Nitro-3,4-dihydroxy-, yellow needles (from eth + petr eth), mp 176º (Ref 2); 5-Nitro-3,4-dihydroxy-, yellow needles (from toluene or water), mp 106º (Ref 2); and 6-Nitro-3,4-dihydroxybenzaldehyde, crystals, mp 203-04º (dec) (Refs 3, 4 & 6).


3,5-Dinitro-2,4-dihydroxybenzaldehyde,
(HO)₂C₆H₂(NO₂)₂·CHO; mw 228.12, N 12.28%; yellow crystals (from alc), mp 170º; was obtd by nitrating the 2,4-dihydroxy deriv with fuming HNO₃.

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-benzalazin or Bis-(3-oxozenbal)-hydrazin in Ger], HO.C₆H₄.CH:N.N'=CH.C₆H₄.OH; mw 240.25, N 11.66%; yellow needles (from alc), mp 204-05º; 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.C₆H(NO₂)₃.CH:N.N'=CH.C₆H(NO₂)₃.OH; mw 510.25, N 21.96%; lt-yel needles, mp - explodes violently at 150-60º; sol in alc, 50% glacial 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.C₆H₄.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 Brenzecatechin in Ger), monoclinic crystals (from benz, water, petr eth or eth), mp 104-05º, bp 245º (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 crystals (from water, alc or acetic), mp 110.7º, bp 281º; d 1.2717; flash point (open cup) 339º 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 & 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 exps

1,4-Dihydroxybenzene; Hydroquinone or 1,4-Benzenediol (called 1,4-Dioxy-benzol or Hydrochinon in Ger), wh trigonal prisms (from w), mp 166º, bp 285º at 730 mm press; oral ingestion causes irritation of intestinal tract, dermatisis can result from skin contact, and its vapors cause eye irritation (Refs 3 & 4)

Other props & methods of prep are given in the Refs

7) Urbanński 1 (1964), 535-36
NITRODERIVATIVES OF DIHYDROXYBENZENE

Mononitrodihydroxybenzene, HO.C6H3(NO2).OH;
mw 155.11, N 9.03%. All possible isomers are found in Bell:
3-Nitro-1,2-dihydroxybenzene, yel ndls (from petr eth), mp 86°C (Ref 1)
4-Nitro-1,2-dihydroxybenzene, yel ndls (from eth + petr eth, benz or w), mp 174-76°C (dec)
(Ref 2)
2-Nitro-1,3-dihydroxybenzene or 2-Nitroresorcinol, orn prisms (from dil alc), mp 84-88°C, bp 232-34°C, d 1.3526 at 96.7°C & 1.2531 at 180.5°C; soln diazotized with water from 20 to 140°C
is reported; forms yel solns with acids; forms a eutectic with Styphnic Acid; can be prepd
by heating resorcinol & fuming H2SO4 at 100°C for 30 mins, cooling to 25°C, adding mixed acids & allowing to stand for 30 mins, addg
water and distilling with steam (Ref 3)

Its Lead Salt, known as Lead 2-Mononitro-
resorcinolate (LMNR),

\[
\begin{align*}
\text{HC} & \equiv \text{C-} \equiv \text{C-NO}_2 \\
\text{HC} & \equiv \text{C-} \equiv \text{NO}_2 \\
\text{Pb} & \\
\text{HC} & \equiv \text{C-} \equiv \text{C-NO}_2
\end{align*}
\]

mw 360.30, N 3.89%, NO2 group 12.77%, Pb 57.51%; reddish-brown microscopic crys; 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. Odiero
(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.
According 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 inten
tioned 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 It
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°C - 0.4 ml/40 hrs

Sampling. Use the same procedure as described under Normal LS Sample
Preparation of Dry Sample. Use the same
procedure as described under Normal LS 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°C
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 micro-
scopic 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
graduated having an ID of 20 mm. Cover the
graduare with a rubber stopper and shake
thoroughly until the sample is completely
wetted. After shaking the graduare, allow it
to stand for 2 hrs away from vibrations
and jarring. Read the settled volume and
cal the apparent density by dividing the wt
of sample in grams by observed volume in ml.
3) Acidity. Transfer 1.00 g of dry sample to
a 150-ml beaker and add 100 ml of freshly
boiled & cooled distilled 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°C. 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:

<table>
<thead>
<tr>
<th>pH Soln</th>
<th>A</th>
<th>B</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.0</td>
<td>-</td>
<td>1.78</td>
<td>8.22</td>
</tr>
<tr>
<td>5.0</td>
<td>-</td>
<td>3.68</td>
<td>6.32</td>
</tr>
<tr>
<td>6.0</td>
<td>8.77</td>
<td>1.23</td>
<td>-</td>
</tr>
<tr>
<td>7.0</td>
<td>6.23</td>
<td>3.77</td>
<td>-</td>
</tr>
<tr>
<td>8.0</td>
<td>4.65</td>
<td>5.55</td>
<td>-</td>
</tr>
<tr>
<td>9.0</td>
<td>1.75</td>
<td>8.25</td>
<td>-</td>
</tr>
</tbody>
</table>

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°C, cool in a desiccator and weigh

\[
\text{Percent of Lead} = \frac{68.32A}{W},
\]

where: \(A = \text{wt of ppt; and}
\[
W = \text{wt of dry sample}
\]

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 CO₂ 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 CO₂ 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 tianiurn. 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 obcd. Conduct a blank detn using the same reagents as above minus the sample

\[
\text{Percent of NO₂ Group} = \frac{0.7668(A-B)N}{W},
\]

where: \(A = \text{ml of ferric ammonium sulfate soln used for blank;}
\[
B = \text{ml of ferric ammonium sulfate soln used for sample;}
\]
\(N = \text{normality of ferric ammonium sulfate soln; and}
\[
W = \text{wt of dry sample}
\]

Note: Nitro group can also be detd by Titanous Sulfate Method, which is offered as an alter-
nate 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 I of Encycl, p XXVI to 120°C, determine the amt of gas evolved in 4 hrs of heating. More detailed description of the test is given in MIL-STD-286A (1961). Method 403.1.2 and in PATR 3279 (1965), pp 19-22 & Fig 15
4-Nitro-1,3-dihydroxybenzene, lemon yel ndls + $\frac{1}{2}$H₂O (from water), anhyd crysts (from clh or CCl₄), mp 120-22°C (Ref 4)
5-Nitro-1,3-dihydroxybenzene, crysts (from w), mp 157-59°C (Ref 5)
2-Nitro-1,4-dihydroxybenzene, dk-red crysts (from w), mp 132-34°C (Ref 6)

Other props & methods of prepn are given in Beil
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 718646 (1935) & CA 29, 6430 (1935) (Describes a compn which is fixed 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 mix) 10) E.B. Kerone & C.C. Carroll, USP 2177657 (1939) & CA 34, 1176 (1940) [Use of LMNR with Ba(NO₃)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 & KClO₃ 10%, or Cu powder 90 & LMNR 10%, both compns made with binding agents)

DINITRODERIVATIVES OF DIHYDROXYBENZENE
Dinitrodihydroxybenzene, HO.C₆H₂(NO₂)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°C (Ref 1)
4,5-Dinitro-1,2-dihydroxybenzene, hygr yel crysts (from nitrobenz + benz), mp 166-69°C (dec); crysts + H₂O, mp 80°C; sol in several org solvs, water & alkali. Its Disodium salt, Na₂C₆H₂N₂O₆, steel-blue crysts, explodes on heating (Ref 2)
2,4-Dinitro-1,3-dihydroxybenzene or 2,4-Dinitroresorcinol (2,4-DNR)
HC==C(OH) — C.NO₂
HC==C(NO₂) — C ; yel plates (from w), mp 147-48°C; 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 dinitroresorcinol or 2) nitration of resorcinol, followed by decarboxylation

For nitrosation, a weak (2.75%) aqueous soln of 3 mole resorcinol is acidified with 2 moles of sulfuric acid. The soln is cooled to ca -2°C, 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°, prepbd 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 nitrated 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 Urbanski (Ref 6, p 538).

After removing moisture from dinitroresorcinol 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 dinitroresorcinol 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 prep of DNR utilizing as starting materials 2,4-Dinitrophenol; 2,4-Dinitroaniline or metadichlorobenzene, but found them unsuitable on acct of very low yields. The same applies to the methods of oxidation of dinitroresorcinol 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, KC₆H₃N₂O₆; dr-red crys, explodes on heating, while its monohydrate, yel ndls (from w) is not explosive Lead 2,4-Dinitroresorcinate, abbreviated as LDNR.

\[
\begin{align*}
\text{HC} & \text{C} & \text{CNO₂} & \text{Pb} \\
\text{HC} & \text{C(NO₂)} & \text{O} & \\
\end{align*}
\]

O

\[\text{HC} = \text{C} - \text{CNO₂} \quad \text{Pb}\]

Next, 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 tetra-chloride 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 LS and it is less sensitive to impact and friction than Pb styphnate. It has therefore recently found application as a component of cap comps. It is valuable because it is safer to handle than LS.

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 comps. It was prepd by J.D. Hopper, PATR 480(1934) by adding slowly, with constant stirring, a soin of 10 g Pb nitrate in 60 ml of boiling w to a soin 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°/sec (Phillips gave 201°/sec); **Heat of Explosion** - 270 cal/g; **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 chloride 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 212.5°. Was first prepd in 1882 by Weselsky, Benedikt & Hibl 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

**Barium Salts are:** Ba(C₆H₃N₂O₃)₂, yel ndls, and BaC₆H₂N₂O₆, carmine-red flakes having a violet reflection. They explode violently on heating (Ref 4)

**Basic Lead Salt, known as Lead 4,6-Dinitroresorcinate, Basic (LDNR, Basic), or Diplumbic Dinitroresorcinate,**

\[
\begin{align*}
O & \quad \text{PbOH} \\
O_2N & \quad \text{C} \quad \text{C} \quad \text{CH} \\
\text{HC} & \quad \text{C(NO₂)} \quad \text{C} \quad \text{O} \quad \text{PbOH, mW 646.53, N 4.3%; exists in two forms: 1) microcrystalline, low density form and 2) a denser brick-red form; density values are not given in Ref 6; both products have, accdg to K.S. Warren, PATR 1448 (1944), the same value for sensitivity to impact}
\end{align*}
\]

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, pred 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 pred 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 conc 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°/5secs; Heat of Explosion - not given; 100° Heat Test - % loss of wt in 1 hr 48 hrs 0.4, in 2nd 48 hrs 0.0 and no expn 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 "dilumbric dinitroresorcinol" together with other ingredients

There is no US Military Specification for Basic LDNR

2,4,6-Dinitro-1,4-dihydroxybenzene, golden-yellow crystals + H2O (from w), mp 93-94°, anhyd crystals, mp 136-37°. Its Barium Salt, BaC6H2N2O6 + H2O, dk-blue crystals, when anhyd is very explosive (Ref 5)

Other props & methods of prepn are given in the Refs


TRINITRODERIVATIVES OF DIHYDROXYBENZENE

2,4,6-Trinitro-1,3-dihydroxybenzene; 2,4,6-Trinitroresorcinol; or STYPHNIC ACID (called 2,4,6-Trinitro-1,3-dihyroxy-benzol; 2,4,6-Trinitro-resorcin or Styphninsäure in Ger), abbreviated as TNR or StIA

O2N.C==C(OH) --- C.NO2

HC==C(NO2) --- C.OH; mw 245.11, N 17.14%; sl hygr crystals 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 prac- tically insol in their mixtures; heat of combustion 537 cal/mol. Styphnic Acid of high purity is prepd by sulfonation (93-98% H2SO4) of resorcinol at 50° and nitrating with 90% HNO3 at not over 65°. Low purity material is obtb by addg 10-20 parts of Na nitrite to the H2SO4 & resorcinol and nitrating with sl more HNO3 under the same conditions as above (Ref 1). Detailed description of prepn is given by Urbanski (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 Urbanski (Ref 18), are as follows:
- **Brisance** by Sand Test - 94% TNT or 71% MF; **Hygroscopicity** - slight (Ref 18, p 539);
- **Impact Sensitivity** - same as Picric Acid or 35% TNT for 50% impact work expls;
- **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**, BaC₆H₄NO₃O₈ + H₂O, orn plates, explodes on heating above 160°. See also Ref 25
- **Potassium salt**, KC₆H₄N₂O₈ + H₂O, lst-yel prisms (from w), explodes when heated on a Pb block, becomes anhyd on heating to 100°
- **Dipotassium salt**, K₂C₆H₄NO₃O₈ + H₂O, orn ndls (from w), explodes violently on heating, becomes anhyd at 120°
- **Sodium salt**, NaC₆H₄N₂O₈ + 3H₂O (from w), explodes violently on heating between 120° & 130°

The most important salts of Styphnic Acid are its lead salts:

**LEAD STYPH Nate, BASIC; Lead Hydroxide Styphnate; or Lead Hydroxide-2,4,6-Trinitroresorcinat**

[Chemical structure diagram]

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 Urbanski (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 nitrate 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

[Chemical structure diagram]

is possible for his salt. Depending on the condition of reaction, there is a possibility of formation of monobasic salt,

\[
\text{OPbOH}
\]

\[
\text{O}_2\text{N},\text{C}=\text{C}=\text{C}(-\text{O})_2
\]

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 isopropl 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...
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 charge 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 charge and 165 mg of LA as a base charge (p XII B); Percussion Primer T98 (p XIII B); Stab Detonator M46 - 115 mg with 300 mg LA as an intermediate charge and 145 mg of RDX as a base charge (p VII B); and Stab Detonator M47 - 15 mg with 110 mg LA & 34 mg RDX (p VII B). In Stab Detonators XM61E2, 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 charge and RDX as a base charge. 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 Stypnate are given after the Refs for Trinitrodihydroxybenzene and Derivatives.
LEAD STYPNATE, NORMAL (LSt) or Lead 2,4,6-Trinitroresorcinate (LTNR) [called Bleistypnhat, Bleitritroiresocinat or Trizinat in Ger; Stypnate de plomb in French; Stiftato di piombo in Ital; Stiftat svintsa or Trinitroresorsciatin svintsa (Teneres) in Russian; and Tricinato (Trizinato) or Trinitroresorcinato de plomo in Spanish],

\[
\text{O}_2\text{N.C} \to \text{C} = \text{C} = \text{C}_3\text{NO}_2 \quad \text{Pb} + \text{H}_2\text{O},
\]

mv of monohydrate 468.30, N 9.0%; yel-orn or reddish-brown needle-like crystals, 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, CCl₄, CS₂, benz, toluene, concd HCl & glac acetic acid. Sl sol in glycol diacetate (0.1% at RT) and soluble in 10% qq Amm acetate (Refs 20, 21 & 24).

It is depcmpt by concd nitric or sulfuric acid (Ref 24, p 7-11)

LSt was first prepd in 1914 by E. von Herz by adding a soln of Mg stypnate 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 congu 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 (prep'd by the reaction of Mg oxide and stypnic 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 Urbánsk (Ref 21, pp 218-20): 1) Method used at Wolftratshausen, 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 Stypnate, 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 prep of LST have been examined recently at PicArsn and the following confidential reports were issued:


**Properties of Normal LST**: Activation Energy is acdcdg Ref 14, p 194 75.39 kcal/mole & Induction Period 0.5-10 sec; acdcdg 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; Brissance - 24 g sand crushed by 0.4 g sample when ignited by MF vs 48 g for TNT and only 11 g when ignited by BkPdt fuse (Ref 20, p 193 & Ref 24, p 7-12); Explosion Temperature - 285°/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°C & 100% RH and 0.02% at 30°C & 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 - 15200 m/sec at 2.9, when initiated with blasting caps (Refs 20 & 24); Sensitivity to Electrostatic Discharge - 0.0009 joules; Specific Heats, cal/cm²°C - 0.141 at -50°C, 0.164 at 25°C, and 0.167 at 50°C; Stability in Storage at 75-80°C - not affected by being stored for 2 months; Vacuum Stability at 100-120°C - 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 (Sec 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 dinitrosoacetylate 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 Odiero 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, Sb sulfide (Grade I, class a) 60.52% (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 (dextrinized 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


Lead Salts of Stypnic Acid. Analytical Procedures. Two lead salts of stypnic acid are used in military primary compons: 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 distilled 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 yellow 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 mix 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 distilled water, 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 Stiphonate (Basic LSt)


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 crystals
2) Crystal Form. The material shall consist of discrete crystals
3) Crystal Dimension. 95% of the crystals shall have a max dimension between 7 & 20 microns. The length of the crystals shall not be greater than 3 times its 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%

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. Prep 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 Mononitroresorcinat
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 determined by titanosus 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 K2Cr2O7 standard 136 (or equivalent) in an oven at 180° for 2 hrs, cool, dissolve 9.8070 g in ca 900 ml distilled water and dilute to 1 liter in a volumetric flask. This is a primary standard.

b) Acetic Acid Solution (25%). Add 250 ml of glacial AcOH to 750 ml distilled water

c) Titanosus Chloride Solution (0.2N). Can be prep from either 20% titanosus chloride
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 FeNH₄(SO₄)₂.12H₂O 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), show 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 w/ std 0.2000 N 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 titanos 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
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 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)


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 "Crystalline Dimension"
2) Crystal Form. The material shall be crystalline and the longest dimension of any crystal shall not be greater than three times the next longest axis. Hexagonal plates are acceptable.
3) Crystal Dimension. The largest dimension of any crystal shall not exceed 0.5 mm.

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:

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
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 containing 50 g of "secondary" sample, prep 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°C. 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 cm² 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 crystals 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 crystals 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 sieve in a container of water. After most of the material has passed thru the sieve, wash the remainder with a gentle stream of water 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

\[
\text{Percent of Material Insoluble in Acetate} = \frac{100A}{W}
\]

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 adtn 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 white 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 PbSO₄

\[
\text{Percent Lead} = \frac{68.32A}{W}
\]

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 PbO₂ 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 Syphnite, Test 11

*Note:* In the old Spec JAN-L-757A (1947) are described the following tests which are not included in the new Spec:

a) *Purity.* This method is also described in MIL-STD-650 (1969), Method 408.1 and recommended for detn of LS& 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 water until the filtrate collected in a 250 mL volumetric flask is clear. After diluting with water to the mark and mixing, a 25 mL aliquot is pipetted to a 1 liter volumetric flask and diluted with water 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 J Amer Optical Soc 31, 638 (1941)]). Then the optical density of a soln at the 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 LS& is calc'd from the equation

\[
\text{Percent LS&} = \frac{29.41A}{WD}
\]

where: \( A = \) optical density (corrected) of LS& 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 LS&. Place each cap in a loading block (located behind a barricade), insert a plunger 0.20 inch in diam and subject the LS& 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 in one end of a piece of safety (Bickford) fuse, 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 covet, 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 L.A. 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 L.A. 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 L.A and calc the average of the corrected values 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, Tetrycane, Ba nitrate and Sb sulfide is described in Vol 1 of Encycl. p A586-L and in Std-Methods Chem Analysis 28 (1963), p 1369.

In a polargraphic procedure employed by D.W. Traas & H.L. Lightenberg, Chem & Ind (London) 1962, 2040 & CA 58, 3262 (1963), LSt is removed from a primer mix containing Tetrycane 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 Tetrycane 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 Tetrycane as dtd by A.M. Wild in Chem & Ind 1957, 1543. Percentage of LSt is calcd by subtracting from 100% the percent of Tetrycane.

In a polarographic procedure of A.M. Wild, Chem & Ind 1963 (20), 819-20 & CA 59, 3710 (1963), Lead Dinitroresorcinate, LSt, NC (varnish), and Tetrycane are dted 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 0.01N HCl at RT using 20 ml of acid in four 5 ml portions, wash the sinter with alc, dry and weigh. Extract Tetrycane 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 Stphosphate 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 dted, and primary compns analyzed by Bolligan's extraction, titration of polynitrophenols with methylene blue by using PhNO₂ as an extractant. NO₃ interference is avoided by extn of
the Styphnic Acid with iso-BuCO Me be-
fore the titration

Other Lead Salts of Trinitroresorcinol

Other Pb salts and derivatives of Trinitro-
resorcinol include:

**Lead Acetate Styrphate**, 
Pb₂(C₂H₃O₂)₂[CO₂H₃NO₃O₈], yel crystals, mp — 
explodes at 315°, d 3.1 at 30°, explodes on impact & by friction; was ob td by treating 
Styphnic Acid with 2 moles of Pb acetate 
in w at 55°, or from Pb Styrphate & aq Pb acetate soln (Ref 1a)

**Lead Triethylhydrazido Hydroxy Styrphate**, 
Pb[(Pb₂C₂H₅)₃(OH)(C₂H₃NO₃O₈)], yel crystals, 
mp — explodes at 298°; obtd by treating an 
aq soln of triethyllead acetate with 1 mol 
of Na hydrogen styrphate at 65° and intro-
ducing alkaline Pb(NO₃)₂ soln 
**Bis(Hydroxyditylhydrazido) Styrphate.** See 
Bis(diethyl) Lead Styrphate, Basic in Vol 2 
of Encycl, p B141-L and Ref 1a 
**Bis(Triethylhydrazido) Styrphate.** See Bis(tri-
eethyl) Lead Styrphate in Vol 2 of Encycl, 
p B160-L and Ref 1b

**2,4,5,6-Trinitroresorcinol-3,5-di(β-nitroxy) Ether Ethyl or Trinitroresorcin diglycolether** 
Dinitroto or 2,4,6-Trinitro-1,3-Bis(2-hydroxy-
ethoxy)benzene Dinitrate in CA [called 
2,4,6-Trinitro-1,3-bis-(2-nitrooxy)-benzol 
in Ger], 

\[
\text{O}_2\text{N.C}==\text{CH} — — — \text{C.NO}_2
\]

\[
\text{C}==\text{C(NO}_2) — — \text{C}
\]

\[
\text{OCH}_2\cdot\text{CH}_2\text{ONO}_2 \quad \text{O.CH}_2\cdot\text{CH}_2\text{ONO}_2
\]

mg 423.21, N 16.55%; ndls (from alc), mp 75°, 
bp — desc at 175° & deflgr at 265°; readily sol 
in acid & benz; mod sol in alc, eth & chlf; 
sol in petr eth & w; was prep'd by nitrating 
4,6-dinitro-1,3-bis(2-hydroxy-ethoxy)-benzene 
with anhyd HNO₃ & concd H₂SO₄ (Refs 1b, 
2 & 4)

Blatt (Ref 3) reported the compd has F1 
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 Tetra-
nitroresorcin in Ger), HO.C₆(NO₂)₄.OH;
mw 290.11, N 19.31%; col crysts(from chlf 
or CCl₄), has bitter taste, mp 152°, bp 
explodes; gives yel colored solns in w or 
alc; was prep'd by nitrating 5-nitroresorcinol 
with concd HNO₃ & concd H₂SO₄ (Refs 1, 
2 & 3)

Its **Monomethylether** deriv, HO.C₆(NO₂)₄.O.CH₃; 
mw 304.13, N 18.42%, col crysts (from chlf 
or CCl₄), bitter tasting, mp 115°, bp — explodes; 
and **Monoethylether** deriv, HO.C₆(NO₂)₄.O.C₂H₅; 
mw 318.16, N 17.61%; col crysts (from chlf 
or CCl₄), bitter tasting, mp 110°, bp — explodes; 
were prep'd by Blanksma (2) 
**Refs:** 1) Beil 6, 833 2) J.J. Blanksma, Rec 
27, 35, 36 (1908); PrKAcadWetenschAmsterdam 
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 
more reactive & more sensitive for use as an expl) 
4) Blatt OSRD 2014 (1944) (Listed under Nitro 
Compounds)

**DIHYDROXYBENZOIC ACID** 
AND DERIVATIVES

**Dihydroxybenzoic Acid,** (HO)₂C₆H₃.COOH; 
mw 154.12, O 41.53%. The five possible isomers 
are described in Beil: 2,3-Dihydroxy — [called 
Benzocatechin-carbonsäure-(3) in Ger], crysts 
+½H₂O (from saturated aq soln), mp — becomes 
anhyd at 100°, mp 203-06° (Ref 1); 2,4-Dihydroxy — 
called Resorcin-carbonsäure-(4) in Ger), ndls 
+3H₂O (from eth), mp — anhyd at 100° & dec 
200-06°(Ref 2); 2,5-Dihydroxy — [called Hydro-
chinon-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 or plates + H₂O (from w), mp 148-67° with 
decom (Ref 4); 3,4-Dihydroxy— [called Benzocatechin-
carbonsäure-(4) or Protocatechusäure in Ger), 
ndls or plates + H₂O (from w), mp 196-200° & melts 
at 194-98° (dec) (Ref 5); and 3,5-
Dihydroxybenzoic Acid [called Resorcin-
carbonsäure or α-Resorcylsäure in Ger), 
prisms or ndls + ½H₂O, becoming anhyd 
at 100-05° & melting at 232-40° (Ref 6)

Other props & methods of prepn are found in 
Beil
Trinitrodihydroxybenzoic Acid, $(O_2N)\text{C}_6\text{O(OH)}_2这个世界。COOH, was not found in Beil nor in CA thru 1967

2,4-Dihydroxybenzonitrile. Its dinitro deriv was prep'd by Lindemann et al (Refs 1 & 2): 3,5-Dinitro-2,4-dihydroxybenzonitrile (called 3,5-Dinitro-2,4-dicyano-benzonitril in Ger), $(\text{HO})_2\text{C}_6\text{H}(\text{NO}_2)_2\text{CN}; mw 225.12, N 18.67%; yel crystals (from dil HCl), mp 175°, was prep'd by reaction of 3,7-dinitro-6-hydroxyindoxylazine with 10% NaOH and acidifying with dil HCl. The Disodium salt, $\text{Na}_2\text{C}_7\text{H}_3\text{N}_3\text{O}_6$, crystals (from a little w), mp - detonates at 230° sol in alk or glc acet ac; sl sol in benz & Nitrobenz; was obtd on acidifying the above reaction mix with acetic acid Refs: 1) Beil 10 [256] 2) H. Linde- 

mann et al, Ann 456, 292 (1927) & CA 21, 3363(1927)

Dihydroxybenzophenyltriadole. See Benzophenyltriadole, Dihydro in Vol 2, p B79-

Dihydroxybenzoquinone AND DERIVATIVES

2,5-Dihydroxy-3-benzoiyloquinone (called 2,5-Dioxy-benzochinon-(1,4) in Ger),

$$\text{O:C} \quad \text{C(OH)} \quad \text{CH} \quad \text{C:O}$$
mw 140.09, O 45.68%; exists in two modifications: metastable form, ltyl lfts or prisms (from acet ac) which, on warming in light or moistening with water or ether, transforms to a stable form, om-yel ndls or lfts (from ac) or rhomb (through sublimation), mp - sublimes undecompanied at 100-50° & dec at 211°; mod sol in hot alc, acet, glc acet ac & w; was prep'd 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. Dahl, Ber 57, 82(1924) & CA 18, 2157(1924)

Nitroderivatives of Dihydroxybenzoic Acid

Mononitrodihydroxybenzoic Acid, 

$O_2N\text{C}_6\text{H}_2\text{(OH)}_2\text{COOH}; mw 199.12, N 7.03%.

The following derivs are found in Beil & in CA thru 1967: 4-Nitro-2,3-dihydroxybenzoi 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 + ½H₂O (from w), mp 215°(dec); forms several colored crystal salts (Ref 1); 3-Nitro-2,5- 
dihydroxybenzoic Acid, yel ndls (from benz or w), mp 158°(Ref 2); 4-Nitro-2,3-dihydroxybenzoic Acid, brn crystals (from w), mp 242°, bp - sublimes w/o decomp (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


3,5-Dinitro-2,4-dihydroxybenzoic Acid (called 3,5-Dinitro-2,4-dicyano-benzoic acid or 3,5-Dinitro-β-resorcylic acid in Ger), 

$(O_2N)\text{C}_6\text{H}_2\text{(OH)}_2\text{COOH}; mw 244.12, N 11.48%; ltyl ndls + ½H₂O (from 2.5N HCl or chl), mp - (anhyd salt) 195-205°; can be prep'd by nitration of β-resorcylic acid (Ref 1). Its Ammonium salt, $(NH_4)\text{C}_7\text{H}_2\text{N}_2\text{O}_6$, citron-yel ndls, deligsr on heating; Potassium salt, $\text{KC}_7\text{H}_2\text{N}_2\text{O}_6$, ltyl ndls (from w), mp - explodes; and Dipotassium salt, $\text{K}_2\text{C}_7\text{H}_2\text{N}_2\text{O}_6$, ltyl ndls (from w), mp - explodes (Refs 1 & 2)

Monoazido, C₆H₃N₃O₄, and Diazido, C₆H₂N₂O₄, derivs were not found in Beil

3-Nitro-2,5-dihydroxy-1,4-benzoquinone, C₆H₄NO₆, mw 205.10, N 7.57%; golden-yel ndls, mp-not reported; mod sol in w; prepared by warming 1 part of 3-nitro-2-hydroxy-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 (1889)

3,6-Dinitro-2,5-dihydroxy-1,4-benzoquinone or Nitranilic Acid [called 3,6-Dinitro-2,5-dioxy-benzochinon-(1.4) or Nitraniläsure in Ger]

O:C

\( \text{C(NO}_2\text{)} \quad \text{C(OH)} \quad \text{C(NO}_2\text{)} \quad \text{C:O} \)

mw 230.09, N 12.18%; hygr lfts, mp 86-87°, bp-deflgr at 170°; mod sol in w & alc; insol in eth; was prep'd by reacting quinone with fuming HNO₃ and keeping the mix at -5° to -10° for several days (Refs 1 & 2), or by nitrating hydroquinone diacetate with mixed acids (Ref 6), which gives a 75% yield (Ref 7)

Some of its salts are expl, such as:

Barium salt, BaC₆N₂O₆, golden-yel leaflets, deflgr on heating; insol in w (Refs 1 & 2)

Calcium salt, CaC₆N₂O₆, yel crys (Refs 1 & 2)

Disodium salt, Na₂C₆N₂O₆, dk-red monoclinic prisms, mp-deflgr violently on heating (Refs 1 & 4)

Hydroxylomine salt, 2NH₄O + C₆H₃N₂O₄, dk-yel lfts, mp-explodes on heating (Refs 1 & 5)

Lead salt (basic), PbC₆H₄N₂ + 2PbO, red powd, mp-deflgr at 215°; explodes on impact or frictional influences; insol in hot w (Refs 1 & 8)

Potassium salt, K₂C₆N₂O₆, ltyel ndls, existing in two modifications, both monoclinic, mp, deflgr more violently than Sodium salt; very sol in hot w; diff sol in cold w (Refs 1 & 4)

Silver salt, Ag₂C₆N₂O₆, yel crys, mp-deflgr on heating (Refs 1 & 2)


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-Nitrocyclohexapentons-(1.2.3.4.5) in Ger;

\( \text{O} \quad \text{N} \quad \text{N} \quad \text{N} \quad \text{N} \)

\( \text{O:C} \quad \text{C(NO}_2\text{)} \quad \text{C(OH)} \quad \text{C:O} \)

or

\( \text{O:C} \quad \text{C(NO}_2\text{)} \quad \text{C(OH)} \quad \text{C:O} \)

mw 211.09, N 19.91%; yel plts or ndls + 4H₂O (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 & glacial acet ac; insol in common org solvs; can be prep'd by treating 6-nitro-3-amino-1,2,4,5-tetroybenzene with NaNO₂ & HCl, ag HNO₃, or nitrosylsulfuric acid. Its Sodium salt, Na₂C₆N₂O₆ + 2H₂O, 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
Dihydroxybenzyl-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'-bistetrozole). See 5,5'-Bis(1-hydroxytetrazole) in Vol 2, p B148-L

Dihydroxylitolalyl or Dihydroxydimethylbiphenyl. See Bicetisol in Vol 2, p B112-R

Dihydroxybutane or Butylene glycol. See Butanediol in Vol 2, p B368-R

Dihydroxy-isobutane or iso-Butylene glycol. See iso-Butanediol in Vol 2, p B370-L

Dihydroxycyclohexyl-peroxide or Dihydroxycyclohexyl-peroxide. See Bis(1-hydroxycyclohexyl)-peroxide in Vol 2, p B145-R

Dihydroxydiaza pentane. Same as Dihydroxytrimethylene-diamine

Dihydroxydiethylamine. Same as Diethanolamine or Diethylolamine

Dihydroxydiethyl nitramine. See under Diethanolamine and Derivatives

Dihydroxydiethyl oxamide. See Diethylol oxamide and Derivatives

Dihydroxydi(2-hydronecatriene). See Bis-(2-hydronecatriene) in Vol 2, p B144-L

Dihydrodiketobenzene. One of the names for Benzoylquinone which is described in Vol 2, p B79-R

Dihydroxymethyl diazoaminobenzene or Dihydroxymethyl triazene. Its tetranitro deriv:

2,2'-Dihydroxy-4,4'-dimethyl-3,5,3',5'-tetranitrodiazooaminobenzene

(called 3,5,3',5'-Tetranitro-2,2'-dioxo-4,4'-dimethyl diazoaminobenzol in Ger),

\[ \begin{align*}
O_2N.C-\overset{\text{OH}}{\rightarrow} & C.N:N.NH.C=\overset{\text{OH}}{\rightarrow} C.NO_2 \\
H_3C.C-\overset{\text{NO}_2}{\rightarrow} & CH & \text{CH}=\overset{\text{NO}_2}{\rightarrow} C.CH_3
\end{align*} \]

mw 437.28, N 22.42%; golden yel plates, mp explodes violently on heating to 160°; was prep 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. Oppenheimer, Ber 9, 1095 (1876) 3) Not found in later Refs

Dihydroxymethylethylmethane or 2,2-Dimethyl-1,3-propanediol, (CH₃)₂C(CH₂OH)₂ (Ref 1). Its dinitrate:

2,2-Dimethyl-1,3-propane Dinitrate (called Dimethylol dinitrate Dinitrate in CA), (CH₃)₂C(CH₂ONO₂)₂; mw 194.15, N 14.43%; lc yel liquid explosive; was prep by Spaeht (Ref 2) by nitrating 2,2-dimethyl-1,3-propane-diol 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. Spaeht, USP 1883045 (1933) & CA 27, 845-46 (1933) 3) M.S. Fishbein, Voennaya Khim 1933, No 6, 3-8; Chem Zentr 1934 II, 1074-75 & CA 29, 7077 (1935)

Dihydroxydimethyl dioxyxipyperazine. Its dinitro deriv:

1,4-Dinitro-3,6-dioxo-2,5-dimethylpiperoxazine,

\[ \begin{align*}
O_2N.N & \overset{\text{CO}}{\rightarrow} CH(CH_3) \\
& \overset{\text{N.NO}_3}{\rightarrow} CH(CH_3) \quad \text{CO}
\end{align*} \]

mw 232.16, N 24.14%; adls (from MeOH or chl); mp dec 136°; readily sol in hot chl; sol in eth; almost insol in cold w; dec in hot w; was prep from 3,6-dioxo-2,5-dimethylpiperoxazine by reaction of abs HNO₃ 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(hydroxyethoxy)-amine. See Diethylol-amine

Di(hydroxyethoxy)-ether. See Diethyleneglycol

Di(hydroxyethoxy)-oxamide. See Diethylol-oxamide

Di(hydroxyethoxy)-peroxide. See Diethylol-peroxide

Di(hydroxyethoxy)-phenylene Ether. See Bis-(hydroxyethoxy)-benzene in Vol 2, p B145-R

Di(hydroxyethyl)-piperezine or Diethylol-piperazine. See N,N'-Bis(2-hydroxyethyl)-piperazine in Vol 2, p B146-R

Di(hydroxyethyl)-sulfamide. See Diethylol-sulfamide


**Dihydroxylaminobenzene and Derivatives**

**Dihydroxylaminobenzene** (called Dihydroxylaminobenzol or Phenylendenedihydroxylamin in Ger), C₈H₄(NH.OH)₂, may be considered as the parent compd of its derivs:

1,4-Di(nitrosohydroxylamin)-benzene (called 1,4-Bis-nitrosohydroxylamin-benzol or "p-Dinitroso-phenilen-dihydroxylamin" in Ger).

\[ \text{HO.N(NO).C₈H₄.N(NO).OH} \] or \[ \text{HO.N(NC₈H₄.N(NO).OH} \]

known only in the form of salts (Refs 1 & 2) *Disodium Salts, Na₂C₈H₄N₂O₂ + H₂O, reddish yel, delicates >250°C (See also Ref 3) *Ferrie Salt, dk-red ppt, explodes at 215°C; almost insol in acet & pyridine

*Silver Salt, not explosive*


**4,6-Dinitro-1,3-dihydroxylaminobenzene** (called 4,6-Dinitro-1,3-dihydroxylaminobenzol or 4,6-Dinitro-phenyl-(1,3)-dihydroxylamin in Ger), \[ O₂N.C == C(NH.OH).CH \]

\[ \text{HC == C(NO₂) -- C.NH.OH} \]

mw 230.14, N 24.35%, brn-yel ndls + H₂O (from dil hot alc), mp-decomposes explosively at 184°C, stable for months when pure; when allowed to stand for days at RT in dil alc, it separates in thick brck-red tablets, mp 172°C (decomp), changing back into yel form on recrystra from dil alc; sol in dil NaOH or NH₄OH with deep red-ben color; can be prep by boiling 4,6-dinitroresorcinc-diphenylether, 3,4,6-\[ \text{HONH(O₂N)₂C₈H₄O₃C₈H₄}_2 \]

in alc with hydroxylamine, NH₃OH, filtering, evaporating and again boiling in alc with hydroxylamine (Refs 1 & 2)
Dihydroxylaminomethane, \( \text{CH}_2(\text{NHOH})_2 \), may be considered the parent compd of dinitroso deriv, although not found in the literature nor used to prep it:

\( \text{N,N'}\)-Dinitroso-\( \text{N,N'}\)-dihydroxylamino-methane (called Bis-nitrosohydroxylaminomethan, Methyliden-bis-nitrosoglyoxylamin or Methylenebisamininitamin in Ger),

\( \text{CH}_2\{\text{N(NO)}\_2\text{O}\}_2 \) or \( \text{CH}_2\{\text{N(O):O:NO}\}_2 \); 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-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) prep'd many salts and determined their decreasing order of expl effect, when primed with MF in the Lead Plate Test, as follows: Ti, Sn, Na, K, Fe, Cu, Cd & Ba. VonHerz (Ref 2) patented the use of some of these metallic salts in mixts with Pb Picrate, Pb Stphynate, and others as compns for detonators & percussion caps.

Ref's: 1) Beil 1, 592, [651] & [806]; 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 Cong Intern Quim Pura Aplicada, 4,438-46 (1934) & CA 30, 3649 (1936); 5) M. Piskorz & T. Urbański, Bull Acad Polon, Ser Sci Chim 11 (11), 607-13 (1953) (English) & CA 60, 12791 (1964)

m-Dihydroxylone Dioxonide (called Dioxonid des α-Cyclooctadiens in Ger),

\[
\begin{array}{c}
\text{CH}_2 - \text{CH}_2 - \text{CH} - \text{CH}_2 \\
\text{O}_3 \text{O}_3 \\
\end{array}
\]

Ref's: 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

Dihydroxymethylantraquinone and Derivatives

1,8-Dihydroxy-3-methylanthraquinone, Chrysophanic Acid, Methylchrysazin, Chrysophanol (called 4,5-Diocylo-2-methylanthracinon; 3-Methyl-chrysanin; Chrysophanol; or Chrysophanäsüre in Ger),

\( \text{HO,CO}_6\text{H}_4(CO)_2\text{C}_6\text{H}_4(\text{CH}_3)\text{OH} \); mw 254.23, O 25.17%, golden yel prills (from alc, benz or petr ether), mp 194-96°C, bp-sblimes & explodes; can be prep'd by diazotizing 5-amino-4-hydroxy-2-methylanthraquinone in concd HSO_4, and warming the soln to 120°C; 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 KCIO_3, 736, sugar 480, hard wheat bolted flour 320, chrysophanic acid 2 & curcuma (coloring material) 1 part

Ref's: 1) Beil 8, 470 (725), [510] & [808]; 2) V.L. Bedier, USP 981969 (1911) & CA 5, 1190 (1911)

1,8-Dihydroxy-3-methyl-2,4,5,7-tetrinitroanthraquinone or 2,4,5,7-Tetrinitro-3-methylchrysan (called Tetranitrochrysohanol or Tetrinitrochrysanäsüre in Ger),

\[
\begin{array}{c}
\text{HO,CO}_6\text{H}(\text{NO}_2)_2 \text{CO}_6\text{H}_4(\text{NO}_2)_2(\text{CH}_3)\text{OH} \\
\end{array}
\]

mw 434.23, N 12.90%; long onnd hs (from acet acid), mp-decomp at 300°C; sol in glacl acet acid; almost insol in cold w; can be prep'd by nitrat of chrysohanol with excess concd HNO_3; forms red crystalline
salts of K, Mg & Ca (Refs 1 & 3). On heating with HNO₃ (d 1.5) the Tetranitro compd gives 2,4,6-Trinitro-m-hydroxybenzoic Acid (Ref 2). No expl props of the Tetra-
nitro compd were reported


**NOTE:** No higher nitrated derivs of Chrystal-
phanic 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 Hydranaphthoquinon in Ger), HO.C₁₀H₈.OH; mw 160.16, O 19.98%. All of the isomers as parent cmpds 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]

**Dinitrodi(hydroxynaphthalene),**

HO.C₁₀H₄(NO₂)₂.OH; mw 250.16, N 11.20%. The following derivs are described in Beil: 1,8-Dinitro-2,7-dihydroxynaphthalene, yel crystals (from anisole), mp - dec at 250° (Ref 1)

2,4-Dinitro-1,5-dihydroxynaphthalene, golden-
bn ndls (from acet acid), mp - dec 247° (Ref 2, p 5273)

2,6-Dinitro-1,5-dihydroxynaphthalene, orange colored pillets (from acet acid), mp - dec 246° (Ref 2, p 5274)

2,4-Dinitro-1,8-dihydroxynaphthalene, red prisms (from MeOH or H₂O), mp - dec 180-82° (Ref 2, p 5284)

4,5-Dinitro-1,8-dihydroxynaphthalene, bm 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 prep and other props are given in the Refs


2,4,6-Trinitro-1,5-dihydroxynaphthalene, HO.C₁₀H₄(NO₂)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-acetoxy-naphthalene with 2% NaOH (Refs 1 & 2). No expl props are reported


2,4,6,8-Tetranitro-1,5-dihydroxynaphthalene, HO.C₁₀H₂(NO₂)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 HNO₃ (d 1.5) at 0°, by direct nitration of 1,5-dihydroxynaphthalene in acetic anhydride by diacetyl orthonic acid in acet anhyd at 0° during 1½hrs (Refs 1, 2 & 3). No expl props were reported


**Tetranitro-2,7-dihydroxynaphthalene,**

HO.C₁₀H₂(NO₂)₄.OH; mw 340.16, N 16.47%; yel crystals (from acet acid), mp 227° with expl decomp; was prepd by adding dropwise fuming HNO₃ to 2,7-dihydroxy-
naphthalene in acetic acid (Ref 2). No other expl props were reported
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

Dihydroxyphenyl-anthranone and its Hexanitrate. See under Bis(hydroxyphenyl)-anthranone and Derivatives in Vol 2, p B147-L

Dihydroxyphenyl-dimethyl-methane. See Di(phenylol)-dimethyl-methane in this Vol

Dihydroxyphenyl-propane and its Dinitro Compound. See under Bis(hydroxyphenyl)-propane in Vol 2, p B147-R

Dihydroxyphenyl-sulfone and its Nitro Compound. See under Bis(hydroxyphenyl)-sulfone and Derivatives in Vol 2, p B148-L

Dihydroxypyrindinecarboxylic Acid, C_{6}H_{6}NO_{4}, mw 155.11, N 9.03 %. Several isomers are described in Beil (Ref 1). The 2,6-Dihydroxypyrindine-4-carboxylic Acid is the most important (Ref 1, p 254). Its dinitro deriv is expl:

3,5-Dinitro-2,6-dihydroxypyrindine-4-carboxylic Acid [called 3,5-Dinitro-2,6-dioxy-pyridin-carbonsäure-(4); 3,5-Dinitro-2,6-dioxy-isonicotinsäure; or Dinitrocitrazinsäure in Ger],

\[
\text{HO.C} = \text{N} = \text{C.OH} \\
\text{O}_{2}\text{N} = \text{C} = \text{C.NO}_{2} \\
\text{COOH}
\]

mw 245.11, N 17.14%; yel ndls + 2H_{2}O (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)

Dihydroxyquinone. See Dihydroxybenzoquinone in this Vol

Dihydroxytetramethylenetetramine or Dihydroxytriazabehaplane,

HO.CH_{2}.NH_{2}.CH_{2}.NH_{2}.CH_{2}.CH_{2}.NH_{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-triazahaplane; or 2,4,6-trinitro-2,4,6-triazahaplane diol-1,7-dinitrate (Code name ATX & NBSX), O_{2}NO.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-triazahaplane 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)

Dihydroxytetrazonane or Dihydroxypentamethylenetetramine. See its nitrated derivs 1,9-Dinitroxy-1,3,5,7,9-pentamethylenetetraamine, and 1,8-Dinitroxy-1,3,5,8-pentamethylenetetraamine, 2,4,6,8-tetranitramine in this Vol

Dihydroxy-tetrazole. See Bis(hydroxytetrazole) in Vol 2, p B148-L.


Dihydroxytoluene and Derivatives
Dihydroxytoluene (called Dioxy-methylbenzol or Dioxy-toluol in Ger), CH₃C₆H₃(OH)₂; mw 124.13, O 25.78%. All of the known isomers are found in Beil
Refs: 1) Beil 6, 872, 874, 878, 882, 896, 897, (426, 428, 431, 437, 439), [858, 859, 861, 865, 875, 877, 881, 882] & 14492, 4495, 4512, 4514, 4531

Mono-nitro-di-hydroxytoluene,
CH₃C₆H₃(NO₂)(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°C (Ref 1)
5-Nitro-2,4-dihydroxytoluene, orn-yel ndls (from gasoline-benzene), mp 125°C (Ref 1)
6-Nitro-2,3-dihydroxytoluene, scarlet red ndls (from eth + petr eth), mp 117-18°C (Ref 2)
X-Nitro-2,5-dihydroxytoluene, yel-brn ndls (from petr eth) or yel-red leaflets (from dil alc), mp 122-24°C (Ref 3)
5-Nitro-3,4-dihydroxytoluene, golden-yel plctts (from w) or ndls (from petr eth), mp 82-83°C (Ref. 4)
6-Nitro-3,4-dihydroxytoluene, lt-yel ndls (from benz or w), mp 180-82°C (dec) (Ref 5)
2-Nitro-3,5-dihydroxytoluene, yel ndls + H₂O (from w) or brn crys + C₆H₆ (from benz), mp becomes free of solvents at 122°C; forms crystalline salts (Ref 6)
4-Nitro-3,5-dihydroxytoluene, orn-colored ndls (from alc), mp 127°C; forms salts (Ref 6)

Other props & methods of prep are found in the Refs

Dinitrodihydroxytoluene,
CH₃C₆H₃(NO₂)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°C (Ref 1, p 873)
4,6-Dinitro-2,3-dihydroxytoluene, yel-brn prisms + H₂O (from w or 50% alc) or yel tablets (from chlf) or orn-yel ndls (from benz), mp 149-53°C (Ref 1, p 877)
2,6-Dinitro-3,4-dihydroxytoluene, yel prisms (from w), mp dec 172°C (Ref 2)
2,4-Dinitro-3,5-dihydroxytoluene, yel plctts (from alc), mp 164.5°C; forms a Ba salt (Ref 1, p 890)
X, X-Dinitro-X, X-dihydroxytoluene, golden-yel ndls (from alc), mp 109-110°C (Ref 1, p 891)

Other props & methods of prep 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-methylbenzol or eso-Trinitro-ocin in Ger),

\[
\begin{align*}
\text{O}_2\text{N} & \quad \text{C} \quad \text{C} \quad \text{C} \quad \text{NO}_2 \\
\text{HO} & \quad \text{C} \quad \text{C} \quad \text{C} \quad \text{OH} \\
\text{NO}_2 & 
\end{align*}
\]

mw 259.13, N 16.22%; yel ndls + 2C₆H₆ (from benz) or lemon-yel rodlets (from MeOH), mp 171-72°C with weak explosion; was prep by heating 2,4,5,6-Tetranitro-m-cresol (See Vol 3, p C358-L) with water; readily sol in hot w, hot benz; sl sol in cold w & ether; forms salts such as K₂C₆H₅N₃O₈, orn ndls; Ag₂C₆H₅N₃O₂ & BaC₆H₅N₃O₂ + 3H₂O, yel ndls; PbC₆H₅N₃O₈, yel ndls; and with naphthalene C₁₂H₈N₂O₈ + C₁₀H₈, yel ndls, mp 120°C (Refs 1 & 2)

No expl props were reported for the trinitro compd
Di(hydroxytoluene)-triazene. Its tetranitro deriv, 3,5,3',5'-Tetranitro-2,2'-dihydroxy-4,4'-dimethyl-dibenzenetriiazene is described in this Vol under Di(hydroxydimethyl)azobenzene.

Dihydroxy-triazaoctane. See Dihydroxy-tetramethylene-triamine

Dihydroxytriazaoctane. Same as Dihydroxypentamethylene-triamine

Dihydroxytriazinylformaldehyde, Dihydroxytriazinylmethanol Oxime or 3,4-Dihydro-6-hydroxy-4-oxo-s-triazine-2-carboxaldehyde Oxime (CA Nomenclature).

\[
\text{N(C(H\_N.OH).NH\_C(O)N.C.OH; mw 156.10, N 35.89\%. This compd and many of its salts were prep'd and described by Ostrogovitch & Crasu (Refs 2 & 3). The salts and their description are too many & detailed to list here. The hydrated mono-Ba salt, (C\textsubscript{4}H\textsubscript{3}N\textsubscript{2}O\textsubscript{2})\textsubscript{2}Ba.3H\textsubscript{2}O, honey-yel crystals, becomes orange-ochre when heated on a Pt foil and decomposes violently with a "Pharaoh's serpent\" effect. }
\]


Di(hydroxytrichloroethyl)-peroxide. See Bis(o-hydroxy-β,β,β-trichloroethyl)-peroxide in Vol 2, p B148-R

Dihydroxytrimethylenediamine or Dihydroxydiazopentane,

OH,CH\textsubscript{2}.NH\textsubscript{2}.CH\textsubscript{2}.NH\textsubscript{2}.CH\textsubscript{2}OH, may be considered as the parent compd of its nitrated deriv, although not used to prept:

1,5-Dinitroxytrimethylene-2,4-dinitromine or 1,5-Dinitroxy-2,4-diazopentanediol-1,5-dinitrate

\[
\text{N(NO\textsubscript{2})\_2.CH\textsubscript{2}(CH\textsubscript{2}O)\_2.CH}_2\text{CH}_2\text{CH}_2\text{O}\_2.CH\textsubscript{2}CH_2\text{CH}_2\text{CH}_2\text{NO}_2\textsubscript{2}.
\]

This compd was prep'd during WWI in Gt.Britain 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 prep'd from MEDNA (methylendinitrimesine)

Dimidazole. See Biimidazole in Vol 2 of Encycl, p B115-L

Dimidazoline. See Biimidazoline in Vol 2, p B115-R

Diminooxalic Acid Dihydroxide & Derivitives

Diminooxalic Acid Dihydroxide,

H\textsubscript{2}N.HN-\text{-C(NH)-C(NH).NH.NH\textsubscript{2}}\text{, mw 116.13, N 72.37\%; ndls (from di alc), mp >250\(^\circ\)mod sol in w; sl sol in alc; insol in eth. It was prep'd by Curtius & Dedichen (Ref 2) on interaction of cyanogen (CN\textsubscript{2})\text{ with 2 moles of hydrazine hydrate.}

Some of its salts are explosive, such as:

\[
\text{Dimino-oxalic Acid Dihydroxide Dinitrate, O\textsubscript{3}NH.H\textsubscript{2}N.HN-C-C-NH.NH.HNO\textsubscript{3} (NH NH)
\]

\[
\text{mw 242.14, N 46.28\%; it yel crystals, mp dec at 147-149\(^\circ\) 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 prep'd at US Rubber Co Laboratory, Passaic, NJ (Ref 3) by adding concd nitric acid to an aqueous soln of dimino-oxalic acid hydrxide, cooling to 0\(^\circ\) and adding ethanol. A more detailed description of prept is given on p34 of Ref 3}

Its Silver salt, C\textsubscript{2}H\textsubscript{5}N\textsubscript{4}+2AgNO\textsubscript{3}, explodes when heated above 100\(^\circ\)(Ref 1, p 1594)

Dimino-oxalic Acid-bis(4,4,4-trinitro-3-methylbutylidene) Dihydroxide,

\[
\text{CH\textsubscript{2}.CH\textsubscript{2}.CH(CH\textsubscript{2}XNO\textsubscript{2})\_8 (CH\textsubscript{2}.CH\textsubscript{2}.CH(CH\textsubscript{2}XNO\textsubscript{2})\_8}
\]

\[
\text{mw 526.40, N 31.93\%; burned quite vigorously, but could not be detonated by a hammer. Was prep'd at US Rubber Co Lab (Ref 4)
\]

Refs: 1) Beil 2, 560 & 1594 2) T. Curtius & G. Dedichen, JPraktChem 50(2),

1,4-Diimino-quinine. See Benzoquinone-1,4-dimine in Vol 2 of Encycl, p 883-L

[3,6-Diimino-1,2,3,6-tetrahydro]-sym-tetrazine or 3,6-Dimino-sym-tetrazine,

\[
\begin{align*}
\text{HN:C} & \quad \text{NH} \\
\text{N} & \quad \text{N} \\
\text{C:NH} & \quad \text{or} \\
\text{H}_2\text{N:C} & \quad \text{N} \\
\text{N} & \quad \text{N} \\
\text{C:NH}_2
\end{align*}
\]

mw 112.10, N 74.98%; violer leaflets (from ammoniacal water), mp - expl ca 120°; may be prepd by evaporating an aq soln of aminoquinidine hydrochloride with KOH soln, while bubbling air thru the soln. Forms salts, some of which are explosive, eg:

\textit{Hydrobromide}, yel Ils, expl at ca 120° \textit{Picrate}, yel amorphous pdr; expl


\textbf{Diiodoacetylene or Diidoethylene, IC:CI, mw 277.86; col crys, mp 78.5°, bp decomps >80°, extremely toxic. Can be prepd by dissolving iodine in liq NH₃ and passing acetylene into the soln; explodes by frictional forces, deflagrates on heating (Ref 1) 


\textbf{Diiododiacetylene or Diiodobutadiyne, IC:C:CI, mw 309.86; crys, mp - explodons 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) 

\textit{Refs:} 1) Beil 1, 266, [246] & [1057] 2) A. Baeyer, Ber 18, 2276(1895)

\textbf{Diiododiphenyl Diazide, Diazidobiphenyl Diiodide or Dinitrosoobiphenyl Diiodide, (N₈)I.C₆H₄-C₆H₄.I(N₈), 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) 

\textit{meta:} crysts, mp 171°; same props as ortho (Ref 2) 

\textit{para:} crysts, mp 174°; same props as ortho (Ref 2) 

Above isomers obtd when molecular proportions of iodosphenylazide, N₈.C₆H₄.IO, and iodoxyphenylazide, N₈.C₆H₄.IO₂, 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) 


\textbf{Diiodofumaric Acid (Dijiodofumaräure in Ger) (Called Acetylenecarbonsäure by Bruck) HOOC.CI:CI:COOH, mw 303.90; nds from eth + benz) or crys (from eth+toluene), mp - dec at 192°, sol in w, alc, eth, ace 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, Ag₂C₄O₄I₂, puffs off ca 140°

\textit{Refs:} 1) Beil 2, 747 & [641] 2) P. Bruck, Ber 24, 418-19(1891) and 26, 845 & 848(1893)

\textbf{4,4'-Diiodosodiphenyl} (called 4,4'-Dijodosodiphenyl in Ger), OIC₆H₄.C₆H₄.IO; mw 438.02, chocolate-brown pdw, mp 127° & explodons at 198°; was obtd by reaction of CI in a chloroform pdw, mp 127° & explodons at 198°; was obtd by reaction of Cl in a chloroform soln with 4,4'-diiodo diphenyl & NaOH. The compd is insol in cold glacial ace acid. Its salts are also expl, 4,4'-dil(iodiobicloro) diphenyl, Cl₂I.C₆H₄.C₆H₄.ICl₂, red-brown unstable powd or yel amorph ndls, mp - dec at 154° 

\textbf{4,4'-Tetraacetyl Ester of Diododiphenyl,}
Diisobutylphthalate. See under Diisobutylphthalate

Diisopropanolamine. See under Dipropylamine and Derivatives

Diisopropanolamine Trinitrate. See under Dipropylamine Trinitrate

Diisopropyl Ether. See under Dipropyl Ether

Diisopropyl-urea. See under Dipropyl-urea

Di(isoquinoline)-diazido-copper. See under Diquinolinio-diazido-copper

Diketopiperazine and Derivatives

Diketopiperazine; 3,5-Dioxopiperazine; Glycine Anhydride; Cycloglycylglycin or Lactam of Glycylglycin

HN-CO.CH₂-NH-CO.CH₂, mw 114.10, N 24.55%, col plates, mp ~ subl 260°; bp dec 275° (Lange), 295° (Ref 4). Can be prep either by methods given in Beil (Ref 1) or by dehydrating intramolecular glycine, H₂N.CH₂.CO.OH, on heating 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, Ag₂C₄N₂O₂H₄; expl on heating (Ref 1, p 265)

1,4-Dinitrodiketopiperazine,

(O₂N)₃-N.C.O.CH₂-N(NO₂).C.O.CH₂, mw 204.10, N 27.45%, OB to CO₂ ~31.4%, white crstls, mp dec 143-147°; bp ~ deflagrates and burns quietly w/o expln. Can be prep, accdg to direction of Franchimont & Friedman (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 exptl comparable in sensitivity with TNT, but unstable in storage (decomposing in 48 hours on heating to 75°C); 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

Dimeryl Peroxide (Alperox C) or Dodecanyl Peroxide, \( (C_{12}H_{24}CO)O_2 \), mw 398.61, O 16.06%; active O 8.04%. The commercial product of the Lucidol Div, contains a minimum of 95.0% of dimeryl peroxide with min active O 3.76%. It consists of wh soft granules with mp 48–50°C; insol in w; sl sol in alcohol; sol in esters, vegetable oils and pet solvents; very sol in chlorobenzene, chl, CS2, CCl4, toluene, dichloromethane, ethylenedichloride and trichloroethylene. Can be prep 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 decomp 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 propellant compositions


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 Fluorennonperoxyd.** 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$_3$CO)$_2$C$_6$H$_4$(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 prepared from 2,4-(CH$_3$O)$_2$C$_6$H$_4$COOH$_3$ by treating with NH$_3$ and oxidizing the amine by treating with hypochlorite in alk medium (Ref 7); and by hydrogenating 2,4-dimethoxy-nitrobenzene in acetic acid with palladium as catalyst

2,5-*Dimethoxyaniline;* white crystals (sublimation), mp 74°–76°; was obtained when 2,5-dimethoxy-bromobenzene reacted with diethyl malonate in the presence of a catalyst (Ref 6)

2,6-*Dimethoxyaniline;* crystals (from petr eth), mp 75.5°–77°; was prepared by hydrogenating 2,6-(MeO)$_2$C$_6$H$_4$NO$_2$ in alic over Raney Ni at 80° (Ref 8)

3,4-*Dimethoxyaniline;* crystals, mp 86°, bp 172°–74° at 24mm press; prepared by treating a boiling soln of 4-nitroveratrole in alc containing Pd-C catalyst with N$_2$H$_4$·H$_2$O (Ref 9)

3,5-*Dimethoxyaniline;* crystals, mp 46°, bp 115° at 0.5mm press; obtained by hydrolysis of 3,5-(MeO)$_2$C$_6$H$_4$NHAc with 15% alc NaOH by refluxing for 4 hrs (Ref 4)

Other props are given in the Refs

**Mononitrodimethoxyaniline,**

(H$_3$CO)$_2$C$_6$H$_4$(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 chl-flt petroleum), mp 93° (Ref 5)

5-Nitro-2,3-*dimethoxyaniline* (called 3-Amino-5-nitroveratrole by Bell & Millar) (called 5-Nitro-3-amino-benzocatechin-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-teracol or 3-Nitro-4-amino-benzocatechin-dimethyläther in Ger), red 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-benzocatechin-dimethyläther in Ger), om-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 abstrakt (4)

Their methods of prepn & other props are given in the Refs

**Dinitrodimethoxyaniline,**

(H$_3$CO)$_2$C$_6$H$_4$(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°; prepared by treating 4,5-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-dimethoxyxaniline, yel-bm ndls (from MeOH); prep by heating 4,5-dinitro-3-acetamino-vertrole with concd sulfuric acid at 100°C (Ref 1, p396)

Other props are given in Refs. No expl props are reported


Trinitromethoxyxaniline,
(H₃CO)C₆(NH₂)(NO₂)₃(OCH₃), 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,  
H₃CO,C₆H₅(CH(O)).OCH₃, mw 166.17. Two isomers are known: 2,4-Dimethoxybenzaldehyde, ndls (from aq alc), mp 69-70°C, bp 165°C at 10mm (Ref 1) and 3,4-Dimethoxybenzaldehyde or Vanillin Methyl Ether, ndls (from eth), mp 44-47°C, bp 280-85°C (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, C₆H₄(N₃)₃O₃, was not found in the literature thru 1966


Dimethoxybenzene and Derivatives.  
Dimethoxybenzenes, (H₃CO)C₆H₅(OCH₃), mw 138.16. Three isomers are known: ortho- or veratrol, crystals, mp 22.5°C, bp 207.1°C (Ref 1); meta- or Resorcinol dimethyl ether, liq, mp -52°C, bp ca 217°C (Ref 2) and para- or Hydroquinone dimethyl ether, liq, mp 56°C, bp 212.6°C (Ref 3). Their prepn and other props are in Beil

Mononitrodimethoxybenzene,  
(H₃CO)C₆H₅(NO₂)(OCH₃); mw 183.16, N 7.65%. All possible isomers are found in the literature, but none is reported to be expl (Ref 3)

Dinitrodimethoxybenzene,  
(H₃CO)C₆H₅(NO₂)₂(OCH₃); mw 228.16, N 12.28%. All isomers are described in the literature, but none is expl (Ref 4)

Trinitrodimethoxybenzene,  
(H₃CO)C₆H(NO₃)₃(OCH₃); 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, crystals (from benz or acet acid), mp 144-45°C; sol in hot alc, eth, toluene or butanol; sl sol in w, heptane & methanol; prep by nitration of veratrole with concd HNO₃ & H₂SO₄ at 100°C (Ref 5)

3,4,6-Trinitrodimethoxybenzene, lt yel ndls (from alc), mp 174°C; sl sol in ethyl acetate; diff sol in chf, alc & acetic acid; obtbd by reaction of 2,4,6-trinitro-benzcatechin-1-methyl ether, dimethyl sulfate & Na₂SO₄ in hot xylene (Ref 6)

2,4,6-Trinitro-1,3-dimethoxybenzene or 2,4,6-Trinitroresorcinol-dimethylether, almost col pltls (from alc) or lt-yel ndls (from alc), mp 124-25°C; sol in alc & eth; prep by nitrating 2,4-dinitro- & 4,6-dinitroresorcinol-dimethylether with HNO₃ & concd H₂SO₄ in the cold (Ref 7)

4,5,6-Trinitro-1,3-dimethoxybenzene, col crysts (from alc), mp 193°C; obtbd by nitrating 5-nitroresorcinol-dimethylether or 4,5-dinitroresorcinol-dimethylether with HNO₃ & concd H₂SO₄ (Ref 8)

Trinitrohydroquinone-dimethylether, yel ndls (from alc), mp 100-01°C; diff 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 HNO₃ 1 part & concd H₂SO₄ 2 parts (Ref 9)

Other methods of prepn & props are found in the Refs

Tetranitrodimethoxybenzene. Not found in the literature thru 1966


**Dimethoxybenzophenone and Derivatives**

Dimethoxybenzophenone (4,4') or p-Anisoylanisole, (H₃C)OC₆H₄·CO·C₆H₄(NO₂)₂, mw 242.26, nds from alc, mp 144-45°. Its prepn and other props are in Ref 1.

Forms on nitration tetranitro- and penta-nitro compounds:

Dimethoxy-3,3',5,5'-tetranitro-benzophenone, (H₃C)OC₆H₄(NO₂)₂·CO·C₆H₄(NO₂)₂, mw 422.26, N 13.27%; crys, mp 197° (2nd form on recryst from acet, mp 182°). Can be prep'd 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, (H₃C)OC₆H₄(NO₂)₂·CO·C₆H₄(NO₂)₂·CO·C₆H₄(NO₂)₂; mw 467.27, N 14.99%; crys (from acet); mp 209°; was obtd by nitration of the tetranitro deriv with a mixt of nitric acid and sulfuric acid (Refs 1a & 4). No expl props of this compd are reported.


**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, (CH₃)₂OC₆H₄·S·C₆H₄(NO₂)₃·S; mw 624.48, N 13.46%; yel tablets (from acet acid) with 2 moles acet ac, mp 228-30°; readily sol in acet ac; nod sol in benz & eth acet; diff sol in alc, eth, chlf & petr eth; was obtd by reaction of 2,4-dimethoxy-1,5-dimercaptobenzene & 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, Monatshfte 35, 1488 (1914) & CA 9, 911 (1913)

**Dimethoxy-methylbenzene**. See Dimethoxy-toluene

**o,o'-Dimethoxybenzoyl Peroxide**

H₃COC₆H₄·O·O·C₆H₄·OCH₃; mw 246.25, O 25.99%; crys, 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 decomd suddenly with a shattering effect & a loud report; a 10g sample at 75° exploded after 30 mins. A 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)


**Di(methoxymethyl)-peroxide or Bis(methoxy-methyl)-Peroxide**

(H₃COCH₂)₂O₂; mw 122.12, O 52.41%; crys; decomp into HCOOH & HCHO by Fe salts; was prep'd when ether solns of HCHO & of hydrogen peroxide were brought together and the ether evaporated; this peroxide & H₂O₂ in ether, or anhyd HCHO & H₂O₂ gives the expl.

**Hydroxymethyl Hydroperoxide** (Ref 2)

Dimethoxynaphthalene and Derivatives

Dimethoxynaphthalene, \( \text{H}_2\text{C}=\text{O} \cdot \text{C}_1\text{H}_4\text{H}_4\text{OCH}_3 \), mw 188.22. The various derivs are described in Beil (Ref 1)

2,4,6,8-Tetranitro-1,5-dimethoxy-naphthalene, (\( \text{H}_2\text{CO} \). \( \text{C}_1\text{H}_{10} \text{H}_2\text{(NO}_2 \text{)}_4 \text{OCH}_3 \)), mw 368.22, N 15.22%; orn-colored crystals (from toluene), mp 255° (dec); prepd by nitratng either 2,4-dinitro- or 2,4,8-trinitro-1,5-dimethoxynaphthalene with nitric acid (d 1.5) in AcOH (Ref 2 & 3)


Dimethoxypentamethylenetetramine and Derivatives

1,9-Dimethoxy-1,3,5,7,9-pentamethylenetetramine or 2,4,6,8-tetramine or 1,9-Dimethoxy-2,4,6,8-tetrazanone, (\( \text{H}_2\text{CO} \). \( \text{C}_2\text{H}_4\text{NH} \cdot \text{CH}_2\text{NH} \cdot \text{CH}_2\text{NH} \cdot \text{CH}_2\text{NH} \cdot \text{CH}_2\text{N(OCH}_3 \text{)}_3 \)), mw 192.27, N 29.14%. May be considered as the parent compd of its tetranitro deriv although not used to prepd it:

2,4,6,8-Tetranitrotriamine-1,9-dimethoxy-1,3,5,7-pentamethoxy or 2,4,6,8-Tetranitro-1,9-dimethoxy-2,4,6,8-tetrazanone, (\( \text{H}_2\text{CO} \). \( \text{C}_2\text{H}_4\text{N(NO}_2 \text{)}_2 \cdot \text{CH}_2\text{N(NO}_2 \text{)}_2 \cdot \text{CH}_2\text{N(NO}_2 \text{)}_2 \cdot \text{CH}_2\text{N(NO}_2 \text{)}_2 \cdot \text{CH}_2\text{N(OCH}_3 \text{)}_3 \)), mw 372.26, N 30.01%; crystals (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-tetrazonane (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)

Dimethoxyphenyl and Derivatives

3,5-Dimethoxyphenol or Pyrogallol Dimethyl Ether, (\( \text{H}_2\text{CO} \). \( \text{C}_2\text{H}_4\text{H}_4\text{OH} \)), mw 154.16, crystals, mp 55–56°, bp 262.7°. Other props and method of prepn in Beil (Ref 1)

Dimethoxypicric Acid or 2,4,6,11-Trinitro-3,5-dimethoxyphenol, (\( \text{H}_2\text{CO} \). \( \text{C}_2\text{H}_4\text{H}_4\text{N(NO}_2 \text{)}_2 \cdot \text{OCH}_3 \)), mw 289.16, N 14.53%; crysts; mp 76.5°; was prepd by the following reactions: 3,5-dinitromethoxybenzene with AlCl₃ at 130° yielded (O₂N₇)₅C₆H₈OH which by Blanska 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. Schubach & F. Mergenthaler, Ber 58B, 2732(1925) & CA 20, 1395(1926) 4) Not found in later refs thru 1966

2,4-Dimethoxyphenylosinosinic Acid and Derivatives

Dimethoxyphenylarsinic Acid, (\( \text{CH}_3\text{O} \). \( \text{C}_2\text{H}_4\text{H}_4\text{As(OH)}_2 \)), mw 263.09, ndls (from w), mp 242–43°; sol in MeOH, aic & acct ac; sl sol in w; v sl sol in eth (Ref 1)

x-Dinitro-3,4-dimethoxyphenylosinosinic Acid, (\( \text{H}_2\text{CO} \). \( \text{C}_2\text{H}_4\text{H}_4\text{N(NO}_2 \text{)}_2 \cdot \text{OAs(OH)}_2 \)), mw 352.08, N 7.95%; crysts, mp decomp at 215° and explodes at higher temps. Can be prepd by treating 3,4-dimethoxyphenylosinosinic acid with abs HNO₃ & concd H₂SO₄ (Refs 2 & 3)


1.1-Di-o-methoxyphenyl-5,5'-azotetrazole or 5,5'-Azobis-[1-(o-methoxyphenyl)-1,2,3,4-Tetrazole],

\[
\begin{align*}
\text{N} \rightarrow \text{C}_6\text{H}_4(\text{OCH}_3) \\
\text{N} \rightarrow \text{C}_6\text{H}_4(\text{OCH}_3)
\end{align*}
\]

mw 378.35, N 37.02%; yel crysts, mp 190° (dec); sol in hot aic.; insol in w & eth. Its prepn and other props are not given in abstract (Ref 2). It is described in Ref 3

Dimethoxytetramethylenetriamine and Derivatives

1,7-Dimethoxy-1,3,5,7-tetramethylenetriamine or 1,7-Dimethoxy-2,4,6-triazabane, 
(H₃CO)₂.CH₂.NH.CH₂.NH.CH₂.NH.CH₂(OCH₃), 
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-tetramethylenetriamine or 1,7-Dimethoxy-2,4,6-triazabane, 
(H₃CO)₂.CH₂.NH₂.N₂. - 
 CH₂.N₂. - CH₂.N₂.CH₂(OCH₃), 
mw 298.22, N 28.18%; crystls, mp 100-104°. Was prepd in Canada as one of the products of nitrolysis of hexamine (hexamethylenetetramine). See Ref 3 also


Dimethoxytoluene and Derivatives

Dimethoxytoluenes or Dimethoxy-methylbenzenes, H₃C.C₆H₄(OCH₃)₂, mw 152.19. The known isomers are found in Beil (Ref 1)

Mononitrodimethoxytoluenes,
H₃C.C₆H₄(NO₂)(OCH₃)₂. All derivs are described in Beil (Ref 2)

Dinitrodimethoxytoluenes,
H₃C.C₆H₄(NO₂)(OCH₃)₂. These isomers are described in Beil (Ref 3)

Trinitrodimethoxytoluenes or Trinitrodimethoxy(methylbenzenes,
H₃C.C₆(NO₂)(OCH₃)₃, mw 287.19, N 14.63%

4,5,6-Trimethoxy-2,3-dimethoxytoluene, ndls (from alc), mp 131-32° (Ref 4, p428)

3,5,6-Trimethoxy-2,4-dimethoxytoluene, no description given (Ref 4, p861)

2,5,6-Trimethoxy-3,4-dimethoxytoluene, yel prisms (from alc), mp 100-101° or ndls (from MeOH), mp 73-74° (Ref 4, pp433, 872)

2,4,6-Trimethoxy-3,5-dimethoxytoluene, crystls, mp 69.5° (Ref 4, p891)

The methods of prepn & other props are found in the Refs. These compds are probably mild expls


Dimethylacetylene and Derivatives

Dimethylacetylene, Butyne-2 or Crotonylene,
H₃.C:C.CH₃, mw 54.09, col liq, fr p -32.2°, bp 27°. Other props & prepn in Beil

(Ref 1)

Hexanitrodimethylacetylene,
(O₂N)₂C:C:C(NO₂)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


7.8-Dimethyl-acetylenediurein,

This German name, as well as the following two names, is given in Beil 26, 445 & (131): 2.2'-Dioxo-4.5-dimethyl-hexahydro-[imidazo-4',5':4.5-imidazol].β-γ and β-γ-Diureylen-butan

This compound can be nitrated to a dinitro deriv. See β-γ-Diureylen-butan and Derivatives

Dimethylamine and Derivatives

Dimethylamine, (H₃C)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)

Acclg 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,
(H₃C)₂N—P—OC₂H₅

CN

(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] & 1931

**Dimethylamine Chloride, (H₃C)₂N·HClO₃**, mw 113.54, N 12.34%; was prep'd by Levi by treating 1 mol of concd aq soln of dimethylamine (previously neutralized with 2N sulfuric acid) with 1 mol of Ba chloride 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(1), 209(1922) & CA 16, 2474(1922)

**N-Nitrodimethylamine or Dimethylnitramine,** (H₃C)₂N·NO₂, mw 90.08, N 31.10%, OB to CO₂ =88.8%; col ndls, sp gr 1.090 at 72.3°, mp 57–58°, bp 187° at 758mm, nD 1.4452 at 72.3°, sol in w, alc & eth. Can be prep'd 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 Cyclotrimethylene-trinitramine described in Vol 3 of Encycl, p C630-R.


**N-Nitrosodimethylamine or Dimethylnitrosamine,** (H₃C)₂N·NO, mw 74.08, N 37.82%, yel oil, bp 154°, d 1.0061 at 20°, nD 1.4368 at 20°.

heat of combstn at Cv detd by Swienteslawski 394.3cal/g; prep'd 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).


**1,1'-Dinitramino-N-nitro(dimethylamine** (called Trinitrodiaminodimethylamine by DiCerrione.), O₂N·N(CH₃₂NHO₂)₂, mw 310.12, N 26.93%. A secondary product formed in the nitration of hexamethyleneetetramine to trimethyleneetritramine. Description of this compd is not given in the abstract (Ref 2).


**Dimethylaminoaniline and Derivatives**

**N,N-Dimethylaminoaniline,** p-Aminodimethylaniline or Dimethyl-p-phenylenediamine, (H₃C)₂N·(C₆H₄)·NH₂, mw 130.19, N 20.57%; col ndls, sp gr 1.041 at 15°/15, mp 41°, bp 262.3°. Other props and method of prep in Refs 1 & 2.

**Pentanitrodimethylaminoaniline,** N,N₁,2,4,6-Pentanitro-N,N₁-dimethyl-p-phenylenediamine,
Dimethylamino-anthraquinone. See Bis(methylamino)-anthraquinone in Vol 2 of Encycl, p B148-R

Dimethylaminoazobenzene and Derivatives p-Dimethylaminoazobenzene,
\(\text{H}_2\text{C}_6\text{N}.\text{C}_6\text{H}_4.\text{N}^+\text{N}^-.\text{C}_6\text{H}_4\), mw 225.28, N 18.65%; yellow, mp 116-17°, bp decomposed. Other props & prep are in Refs 1 & 4

4,6,4'-Trinitro-3-dimethylaminoazobenzene,
\(\text{O}_2\text{N}.\text{C}_6\text{H}_4.\text{N}^+\text{N}^-.\text{C}_6\text{H}_4(\text{NO}_2)_3.\text{N}^-.\text{CH}_2\text{CHO}\), mw 360.28, N 23.33%; orn red powd (from glacial acetic acid), mp 214-16° (sinters at 190°); prepd by boiling quinone oxime-2,4-dinitro-5-dimethylaminophenylhydrazone with AcOH & conc nitric acid (Ref 2 & 3)

Ref's: 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

Dimethylaminoazobenzene and Derivatives p-Dimethylaminoazobenzaldehyde,
\(\text{H}_2\text{C}_6\text{N}.\text{C}_6\text{H}_4.\text{CHO}\), mw 149.19, N 9.39%; yellow, mp 74-75°, bp 176-77° at 77mm; sol in hot w and in alc, eth & AcOH. Can be prepd by mixing dimethyline, 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


Mono-nitrodimethylaminobenzaldehyde,
\(\text{H}_2\text{C}_6\text{N}.\text{C}_6\text{H}_4.(\text{NO}_2).\text{CHO}\), mw 239.19, N 17.57%. Two isomers are found in Beil:
6-Nitro-2-dimethylaminobenzaldehyde, yellow (from petr eth), mp 105° (Ref 1)
3-Nitro-4-dimethylaminobenzaldehyde, yellow (from alc), mp 103-05° (Ref 2)

Neither compd is indicated to be exp

Ref's: 1) Beil 14, 28 2) Beil 14, 39 & (364)

NOTE: No higher nitrated derivs of Dimethylaminobenzaldehyde were found in the literature thru 1966

\(\text{N},\text{N},\text{N}^+\text{-dimethyl-aminobenzene}, (\text{H}_2\text{C})\text{N.C}_6\text{H}_4\text{.N}\) See \(\text{N},\text{N}^+\text{-Dimethylaniline}

1,3-Di(methylamino)-benzene, \((\text{H}_2\text{C}.\text{HN}).\text{C}_6\text{H}_4\). See \(\text{N},\text{N}^+\text{-Dimethyl-}m\text{-phenylenediamine}

3,5-Dimethyl-1-aminobenzene, \(\text{H}_2\text{N}.\text{C}_6\text{H}_4(\text{CH}_3)_2\). See 3,5-Dimethylaniline or m-Xyolidine

1,2-Di(N-methylaminobenzene) or \(\text{N},\text{N}^+\text{-Di phenyl-ethylenediamine}
\(\text{H}_2\text{C}_6\text{N}.\text{NH}.\text{CH}_2-\text{CH}_2.\text{NH}.\text{C}_6\text{H}_4\). See Bis (anilino)-ethane in Vol 2 of Encycl, p B131-L

Dimethylaminobenzeneoctazolao Azide,
\(\{(\text{3-p-Dimethylaminophenylazo}-5-azido]-\text{asym-triazole}, or 5-Azido-3-(4-dimethylamino-1-benzene)-azotriazol-1,2,4.

German names: 5-Azido-3-[4-(dimethylamino-1-benzol]-azo]-triazol-1,2,4 or Azido-5[directions 4-benzol]-azo]-3-triazol-1,2,4

\(\text{N},\text{N}^+\text{.NH}.\text{N}\)

\(\text{HC}:\text{C}:\text{C}:\text{N} \text{.CH}_2.\text{CH}_3\)

mw 257.26, N 49.00%; barontaux-red pd; 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 conc HCl, followed by addn of dimethyline in conc HCl

Ref's: 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,
HO₂C₆H₄.N(CH₃)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,
HO₂C₆H₃(NO₂)₂.N(CH₃)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 glc acet ac; insol in cold w & benz
(Ref 4, p445)

No expl props are reported

3,5-Dinitro-4-dimethylaminobenzoyl Azide,
N₈OC₆H₃(NO₂)₂.N(CH₃)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 NaN₃ (Ref 6).

Its expl props are not reported

3,x,x-Trinitrodimethylamino-p-benzoic Acid,
HO₂C₆H(NO₃)₃.N(CH₃)₂, mw 300.19, N 18.67%; yel plts, mp 193°; was prepd by nitratng 3-nitro-4-dimethylaminobenzoic acid with concd H₂SO₄ & HNO₃ (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)

Di(methylamino)-benzenophenone. See Bis(methylamino)-benzenophenone in Vol 2, p B149-L

Dimethylamino-benzenoneimide. Same as Auramine as described in Vol 1, p A507-R

N,N-Dimethylaminobiphenyl and Derivatives

N,N-Dimethyl-p-aminobiphenyl, N,N-Dimethyl-p-xenylamine; N,N-Dimethyl-p-biphenylamine; N,N-Dimethyl-p-phenylamino or N,N-Dimethyl-diphenylamine, C₆H₅.C₆H₄.N(CH₃)₂, mw 197.27, N 7.10%; plts (from alc or eth), mp 123–26°. Its salt, C₆H₅.N₂O₅S, in warm dil HCl gives colorless ndls product which is expl (Ref 1)

3,4-Dinitro-4-dimethylaminobiphenyl,
O₂N.C₆H₅.C₆H₃(NO₂).N(CH₃)₂, mw 287.27, N 14.63%; orn-red plts (from alc), mp 137–38°; prep'd by nitratng either 3-nitro- or 4'-nitro-4-dimethylaminobiphenyl (Ref 2)

2,6,2',4'-Tetronitro-N,N-dimethyl-p-aminobiphenyl or N,N-Dimethyl-2,6,2',4'-tetrinitro-xenylamine,
(O₂N)₂.C₆H₅.C₆H₃(NO₂)₂.N(CH₃)₂, mw 377.27, N 18.56%; large ndls (from alc), mp 122°; was obtd by nitratng either 4-dimethylaminobiphenyl or 3-nitro-4-dimethylaminobiphenyl with HNO₃ & AcOH (Ref 3).

Its expl props were not reported


Dimethylaminocyanophosphoric Acid, Monomethylster of Dimethylaminooxy-cyanophosphine Oxide or Ethylphosphoryldimethylamino-cyanadate,

\[
\begin{align*}
\text{(H}_2\text{C})_2\text{N} & \cdot \text{P} \cdot \text{O} \cdot \text{C}_6\text{H}_5, \\
\text{CN}
\end{align*}
\]
invented and known in Germany before WWII as Tabun Trilon B3, 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 prep'd 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-phosgene Oxide or Monoisopropyester of methylfluorophosphoric Acid (called Nerve Gas by G. Schrader).

\[ \text{CH}_2(\text{CH}_3)_2\text{CH.O.C.H(\text{CH}_3)}_2; \]

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 160 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 methylpinacoloyxyfluorophosphine Oxide, Monopinacol ester of methylfluorophosphoric Acid or Pinacolylmethylphosphonofluoridate.

\[ \text{O} \quad \text{H} \]
\[ \text{H}_3\text{C.P.O.C.C(\text{CH}_3)}_3; \]
\[ \text{F} \quad \text{CH}_3 \]

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 Cyclohexylmethylphosphonofluoridate or Cyclohexylic-ester of methylfluorophosphoric Acid,

\[ \text{CH}_2(\text{CH}_3)_2\text{CH.O.P.CH}_3; \]

(Ref 4, p C168)
Refs: 1) Captain Collomp, Revue Mensuel de l'Armée de l'Air, No 37, October 1949

\[ \text{Di(methylamine)-diazido-copper (II) or} \]
\[ \text{Di(methylamine)-diazido-copper (II) Diazide,} \]
\[ [\text{Cu(CH}_2\text{NH}_2)_2][\text{N}_3]; \]

\[ \text{mw 209.76, N 53.42%; crystalls smelling like methylamine; sinters at 84°, mp 126-28°; bp expl at 180-90° on a preheated block; does not detonate by impact; burns in flame with crepitation; unstable in air but can be stored in a closed container; insol in neutral solvents, but dissolves in some basic liquids, such as pyridine; hydrolyzed by water. Can be prep'd by dissolving Cu(N_3)_2 in dimethylamine and addg an acid to ppt the salt.} \]

Refs: 1) Beil 4 [98] 2) A. Cirulis & M. Straumanis, ZAnorgChem 251, 345 (1943) & CA 37, 6574 (1943)

\[ \text{Dimethylaminodiphenyl. Same as Dimethylaminobiphenyl described in this Vol} \]
Dimethylaminodiphenylamine and Derivatives

4-Dimethylaminodiphenylamine,
(H₃C)₂N·C₆H₄·NH·C₆H₄₂, mw 212.28, N 13.20%; ndls (from petr. eth), mp 130° (Ref 1)

2,4,2',4',6'-Pentanitro-3-dimethylaminodiphenylamine,
(H₃C)₂N·C₆H₄(NO₂)₂·NH·C₆H₄(NO₂)₂, mw 437.28, N 22.42%; brn-yel crysts (from glacial acetic ac); mp 149°; was prep'd by reacting dimethylamine & 2,4,6,2' (?),3',4'-hexanitrodiphenylamine in MeOH (Refs 2 & 3). No expl props are reported


Di-methylamino-diphenylmethane. See Bis-(methylamino)-diphenylmethane in Vol 2 of Encycl, p B149-R

2-Dimethylaminooethanol and Derivatives

2-Dimethylaminooethanol: Deanol at 2-
Dimethylethanolamine, (H₃C)₂N·CH₂·CH₂·OH, mw 89.14, N 15.71%; col liq, sp gr 0.987 at 20°/4, fr p -59°; bp 135° at 758mm, flash p (open cup) 105°F. Can be prep'd from ethylene oxide and dimethylamine. Used as chemical intermediate, pharmaceutical and corrosion inhibitors (Refs 1 & 3)

2-Dimethylaminooethanol Nitrate,
(H₃C)₂N·CH₂·CH₂·ONO₂, mw 134.14, N 20.89%; prisms from accl, mp 63°; was prep'd by reaction of 97% HNO₃ on 2-dimethylaminooethanol nitrate, C₄H₁₀N₂O₃·HNO₃, at -5° (Refs 2 & 4). It forms a salt,

2C₄H₁₀N₂O₃·H₂PtCl₆+2H₂O, orn-red prisms, mp 204-05° (dec) (Refs 2 & 4).


3β-Dimethylamino-1,2,4-triazole Dicarboxylate,

HC
NH
N
C.CH₂·CH₂·N(CH₃)₂·(C₆H₄N₃O₇)₂;

mw 598.40, N 23.41%; ndls (from w), mp 181-82°, exp at higher temp. Was prep'd by refluxing for 2 days a mixt of α(1,2,4-

triazolyl-3)-N,N-dimethylacetamide with LiAlH₄ and anhydrous tetrahydrofuran, followed by treatment with Picric Acid.


Dimethylaminohydroxylaminosobenzene and Derivatives

Dimethylaminohydroxylaminosobenzene,
(H₃C)₂C₆H₄·N·N·C₆H₄·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-dimethylaminoo-4-hydroxylaminosobenzene,
(H₃C)₂C₆H₄(NO₂)₂·N·N·C₆H₄·NHOH or
(H₃C)₂C₆H₄·NH·N·C₆H₄·N·OH; mw 332.29, N 21.73%; red-bm crysts, mp 227°; sol in NaOH wth 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. Botsche, Ber 54, 675 (1921) & CA 15, 2844 (1921)

Dimethylaminomethylnitrotoleulene and Derivatives

Dimethylaminomethylnitrotoleulene,
(H₃C)₂N·C₆H₄(NHCH₃)·CH₃; mw 164.24, N 17.06%. May be considered as the parent compd of its nitratd derivs:

3,5-Dinitro-2-dimethylamino-4-methylaminotoluene (Ref 1),
(H₃C)₂N·C₆H(NO₂)₂·(NHCH₃)·CH₃; mw 254.24, N 22.04%; yel ndls (from alc), mp 115°; prep'd from 3,5,5-trinitro-4-methyl-
aminotoluene in hot alc by treating it with a 33% alc soln of dimethylaniline (Refs 2 & 4, p565)

3,5-Dinitro-2-dimethylamino-4-methylnitroanisole, 
\((\text{H}_3\text{C})_2\text{N.C}_6\text{H}_4\text{H(NO}_2\text{)}_3\text{N(NO}_2\text{)CH}_3\) \text{CH}_3; \text{mw} 299.24, N 23.41%; brn-yel nds (from alc), mp 126–27\(^\circ\); prep'd by heating 2,3,5-trinitromethyltrimethylnitrourethane in alc with a 33% alc soln of dimethylaniline in a sealed tube on the water bath for 20 mins (Refs 3 & 4, p528)


Di(methylaminomethyl) -methylamine. See Bis(methylaminomethyl)-methylamine in Vol 2 of Encycl, pB149-R

Dimethylaminophenol and Derivatives

Dimethylaminophenol, \((\text{H}_3\text{C})_2\text{N.C}_6\text{H}_4\text{OH}; \text{mw} 137.18, N 10.21%\); three forms: ortho, prisms, mp 45\(^\circ\); meta, mp 85\(^\circ\); and para, mp 78\(^\circ\) are listed in Beil (Refs 1, 2 & 3)

Dinitrodimethylphenol, 
\((\text{H}_3\text{C})_2\text{N.C}_6\text{H}_4(\text{NO}_2\text{)}_2\) \text{OH; mw} 227.18, N 18.50%. The following isomers are described in the literature:
4,6-Dinitro-2-dimethylphenol, yel powd (from glc acet ac), mp 218–20\(^\circ\) (Ref 4, p124)
2,4-Dinitro-3-dimethylphenol, no description given (Ref 4, p137)
2,6-Dinitro-3-dimethylphenol: yel crystals, mp 191–95\(^\circ\); forms expl salts: Ammonium, \(\text{NH}_4\text{C}_6\text{H}_4\text{N}_2\text{O}_6\); gold-yel plts, mp 195\(^\circ\); Barium, \(\text{Ba(C}_6\text{H}_4\text{N}_2\text{O}_6\)) \text{2} + 1\text{H}_2\text{O}, ppt of orn-red crystals; Potassium, \(\text{KC}_6\text{H}_4\text{N}_2\text{O}_6\); orn-red ndls; and Silver, \(\text{AgC}_6\text{H}_4\text{N}_2\text{O}_6\); lt-red crystals, mp–expl violently at ca 140\(^\circ\) (Refs 5 & 6)
4,6-Dinitro-3-dimethylphenol, crystals, mp 231\(^\circ\) (Ref 4, p138 & Ref 7)
2,6-Dinitro-4-dimethylphenol, purple-colored ndls having a copper luster (from alc), mp 182–83\(^\circ\) (Ref 4, p190)

Lipmann & Fleissner (Ref 6) prep'd in 1885 a silver salt of dinitrocompd and found that it exploded violently at ca 140\(^\circ\)


NOTE: No higher nitrated derivs of Dimethylanaphenol were found in the literature thru 1966

[(3-p-Dimethyaminoarylazo)-5-azido]-triazole. See Dimethylaminobenzeneazotriazole Azide

Dimethylaminophenyl-methylamine and Derivatives

Dimethylaminophenyl-methylamine

\((\text{H}_3\text{C})_2\text{N.C}_6\text{H}_4\text{N(CH}_3\text{)}_2; \text{mw} 150.22, N 18.65%\) (Ref 1)

2,4,6-Trinitro-3-dimethylaminophenyl-methylnitroso, \((\text{H}_3\text{C})_2\text{N.C}_6\text{H}_4(\text{NO}_2\text{)}_3\text{N(NO)}\text{CH}_3; \text{mw} 314.22, N 26.75%\); yel crystals, mp 132\(^\circ\); prep'd by reacting a warm glc acet acid soln of \(\text{N}_2\text{N}_2\text{N}_2\text{N}_2\text{N}_2\text{-tetranitro-m-phenylene diamine}

with nitric acid (Refs 2 & 3)

Its expl props are not reported

Refs: 1) Beil 13, not found 2) Beil 13, 61 3) Wurst & Morley, Ber 12, 1815 (1879) 4) No later refs found thru 1966

Dimethylaminopropane and Derivatives

2,2-Dimethylaminopropane or Dimethylisopropylamine, \(\text{CH}_3\text{C(CH}_3\text{)}_2\text{C}_2\text{H}_5; \text{mw} 87.16, N 16.07%\). Blomquist attempted to prep the following compd:

2,2-Dimethyl-1,3-dinitroaminopropane, 
\((\text{H}_3\text{C})_2\text{C(CH}_2\text{N}_2\text{HNO}_2\text{)}_2\) \text{gi; mw} 192.18, N 29.16%. Synthesis was undertaken but not completed because of diff 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_, (H₃C)₂N.C₆H₄N, mw 122.17, N 22.93%. The 2-Dimethylamino-, liq. fr p 15°, bp 200–01 (Ref 1) and 4-Dimethylamino- plts (from eth), mp 114° (Ref 2) derivs are known. They both form crystalline add salts

3,5-Dinitro-2-dimethylaminopyridine,
(H₃C)₂N.C₆H₂(NO)₂N; 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; prped by nitrating 2-dimethylaminopyridine with 2 Moles HNO₃ & concd H₂SO₄ at low temp (Refs 3 & 4)

No expl props are reported


NOTE: No higher nitrated derivs are found in the literature

Dimethylaminosuccinic Acid and Derivatives

_dimethylaminosuccinic Acid_ (Dimethylaminobereistsäure, in Ger),

N(CH₃)₂

HOOC–CH–CH₂–COOH; mw 161.16, N 8.69%; monoclinic prisms, mp 185°; sol in dil acids & alkalis; v sl sol in cold w (Ref 1)

Dimethylaminosuccinic Acid Dihydrizide,

N(CH₃)₂

H₂N.HN.OC.CH–CH₂–CO.NH.NO₂;

mw 189.22, N 37.01%; ndls (from alc), mp 147°; sol in w & alc; insol in eth; obtbd by warming inactive dimethylaminosuccinic acid dimethyl ester & hydrazine hydrate in alc. Forms a Hydrochloride salt, C₆H₁₆N₆O₂ + HCl, deliquescent powd (Refs 2 & 3)


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, 

(H₃C)₂N.C–NH–N

N–N;

mw 113.13, N 61.91%; very hygro plts (from ethyl acet), mp – dec ca 80°; mod sol in alc & ethyl acet; v sl sol in eth; prped by heating 5-aminoazetrole with an excess of methyl iodide in a tube at 160–65°; not stabile as a free base. It forms salts, such as C₂H₅N₅ + HCl, ndls (from alc), mp 241° (dec); mod sol in w;

C₂H₅N₅ + HCl + AuCl₃, yel plts (from w), mp 164°;

2C₂H₅N₅ + 2HCl + PtCl₄, yel prisms (from w), mp 200°; mod sol in w; sl sol in alc; and

_Picate_, C₂H₅N₅ + Cu₂(N₅)₂O₇, ndls, mp 203° (dec); v sl sol in cold w


Di(methylamino)-toluene. See Bis (methylamino)-toluene in Vol 2 of Encycl, p B150-L

Dimethylammonium-diaziodichlorocuprate,

(H₃C)₂NH₂[Cu(N₅)₂Cl], mw 229.14, N 42.79%, cryssts, 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,

(H₃C)₂NH₂[Cu(N₅)₂NO₃], mw 255.68, N 43.83%, cryssts, mp – explodes at 215°; method of prepn not given in CA
Refs: 1) Beil – not found 2) M. Straumanis & A. Cirlulis, ZAnorgChem 252, 9(1943) & CA 38, 3564(1944)

Dimethylammonium Nitrate (Code named in Germany as DI-Solz), (H₃C)₂NH₂·NO₃; mw 108.10, N 25.92%; crystals, mp – decomposes explosively above 120°C; unstable at temps above 100°C. Was prepd during WWII in Germany by treating aqueous dimethyamine (qv) with nitric acid of sp gr 1.42, followed by vacuum distillation. Dissolves in water and is hydrolyzed 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.


Dimethylammonium-pentaazidocuprate,
(H₃C)₂NH₂[(N₃)₅Cu₅N₅Cu(N₃)₅]; mw 383.29, N 58.47%; crystals, mp – explodes at 201°C; method of prepn not given in CA

Ref s: 1) Beil – not found 2) M. Straumanis & A. Cirlulis, ZAnorgChem 252, 9(1943) & CA 38, 3564(1944)

Dimethylammonium-tetrazidocuprate,
[(H₃C)₂NH₂]₂Cu(N₃)₄; mw 323.82, N 60.56%; crystals, mp – explodes at 210°C; method of prepn not given in CA

Ref s: 1) Beil – not found 2) M. Straumanis & A. Cirlulis, ZAnorgChem 252, 9(1943) & CA 38, 3564(1944)

DIMETHYLANILINES AND DERIVATIVES

Dimethylanilines, C₆H₅.N, mw 121.18, N 11.56%

Two types of these comds, are known: N,N-Dimethylaniline or N,N-Dimethylphenylamine. C₆H₅·N(CH₃)₂. In this type, two methyl groups are attached to N of amino group

2,3-, 2,4-, 2,5-Dimethylanilines
2,3-, 2,4-, 2,5-Dimethylanilines
2,3-, 2,4-, 2,5-Dimethylanilines
2,3-, 2,4-, 2,5-Dimethylanilines

or α-, β-, γ-Xylidines, (H₃C)₂C₆H₃·H₂N.

In these comds two methyl groups are attached at various positions in the benzene ring.

Following are the descriptions of these comds and of their derivatives:

N,N-Dimethylaniline; N,N-Dimethylphenylamine; or N,N-Dimethylanilinobenzene,
C₆H₅·N(CH₃)₂. This comd 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-methylintramidine,

(\(\text{O}_2\text{N}\))₃C₆H₆·N

commonly known as Tetralin.

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°C; nD 1.5582, fr p 2.5° bp 194.2°C, 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°C
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 (T₁) 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 throughout 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 (t₁) 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:

\[ \text{EmStemCorrection} = 0.000159 (t - t₁)n \]

where 0.000159 is the coefficient of expansion of mercury in glass.

Calculate the "corrected freezing point" of the sample by adding the value calculated from the above formula to the "uncorrected freezing point" (t)

Note: For samples containing 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 (ycl),
titrate with N/10 HCl soln; if it turns acidic (red), titrate with N/10 NaOH soln.

% Alkalinity, as NaOH = $\frac{0.801 \times AB}{C}$

% 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) with 200ml distd w until no further soln takes place. After allowing the mixt to settle, filter and use the filtrate as indicated below:

Piper 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-<i>N</i>,<i>N</i>-dimethylaniline,  
(H₄C)₂N.C₆H₄.NO, mw 150.18, N 18.65%;  
 gm plltts (from eth or petr eth + benzol),  
dk-gm plltts + 1 C₆H₄ (from benzol), mp 85°;  
d 1.145 at 20°; can be prepbd by reaction of nitrosoguanidine & dimethylaniline in concd HCl, and by other methods

It forms many solts and addn compds, some of which decomp on heating; for example, one salt, 3C₅H₁₀N₂O + 2SiF₄, yel amorph powd, defflagrates or explodes mildly on heating above 150° (Ref 14 & 18)

Mononitro-<i>N</i>,<i>N</i>-dimethylanilines,  
(H₄C)₂N.C₆H₄.NO₂, mw 166.18, N 16.86%.  
Three isomers are known, none of them is explosive:

2-Nitro-<i>N</i>,<i>N</i>-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-<i>N</i>,<i>N</i>-dimethylaniline, red crysts, sp gr 1.313 at 17°, mp 60–61°, bp 280–85°. Prepn & other props are in Ref 3

4-Nitro-<i>N</i>,<i>N</i>-dimethylaniline, yel ndls, mp 163–65°. Prepn & other props are in Ref 4

Dinitro-<i>N</i>,<i>N</i>-dimethylanilines,  
(H₄C)₂N.C₆H₃(NO₂)₂, mw 211.18, N 19.90%.  
Four isomers are known and they may be considered as weak expls:

2,4-Dinitro-<i>N</i>,<i>N</i>-dimethylaniline, yel crysts, sp gr 1.476 at 17°; mp 87°. Can be prepbd by condensation of N,N-dimethylanilne with 2,4-dinitrochlorobenzene or by nitration of dimethylaniline in glc acet acid. Its heat of combstn at Cᵥ & 18° is 1053.4 kcal/mole and heats of formation 6.4 at Cᵥ and 10.75 at Cₚ (Refs 5 & 27)

2,5-Dinitro-<i>N</i>,<i>N</i>-dimethylaniline, yel crysts, sp gr 1.315 at 18°, mp 112°. Can be prepbd by nitrating 3-nitrodimethylaniline with NaNO₂ & dil HCl, and by several other methods (Ref 6)

2,6-Dinitro-<i>N</i>,<i>N</i>-dimethylaniline, orn-yel ndls (from dil alc), mp 78°. Can be prepbd by reacting 2-chloro-1,3-dinitrobenzene with dimethylaniline in dil alc (Ref 7)

3,4-Dinitro-<i>N</i>,<i>N</i>-dimethylaniline, crysts, sp gr 1.423, mp 174–76°. Can be prepbd by nitrating 3-nitrodimethylaniline with 20% HNO₃ (Ref 8)

Trinitro-<i>N</i>,<i>N</i>-dimethylanilines,  
(H₄C)₂N.C₆H₃(NO₂)₃, mw 256.18, N 21.87%, N (of NO₂) 16.44%. Three isomers are known, of which 2,4,6-trinitro was examined as a possible military expl:

2,3,4-Trinitro-<i>N</i>,<i>N</i>-dimethylaniline, brownish crysts, sp gr 1.552 at 17°; mp 154°. Can be prepbd along with the 2,4,5-trinitro deriv by warming of 3-nitrodimethylanilines 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-<i>N</i>,<i>N</i>-dimethylaniline, orn-red crysts (from acet or benz), mp 198°, sp gr 1.585 at 17°. Can be prepbd by heating N,N-dimethyl-N'-phenyl-4,6-dinitro-1,3-phenylene-diamine with aniline & alc in a tube at 110–20°, or by other methods (Ref 10)

2,4,6-Trinitro-<i>N</i>,<i>N</i>-dimethylaniline; 2,4,6-Trinitro-<i>N</i>,<i>N</i>-dimethylphenyldiamine; 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 prepbd by interaction of picryl chloride with dimethylanilne (Refs 11, 15, 21 & 23)
2,3,4,6-(or 2,4,5,6)-Tetranitro-N,N-dimethylaniline or Tetralita No 1, 
(H₃C₂)₄N,C₆H₄(NO₂)₄, mw 301,18, N 23.26%, 
N (of NO₂) 18.60%, mp 125-26°C; expl on heating; mp 153°C (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 solid 
slowly into concd nitric acid at 30°C. It has 
begged 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 Tetranitrometilalanilina (Reps 12, 
16, 17 & 22, p T3)

Pentanitro-N,N-dimethylaniline, Pentanitro-
N,N-dimethylphenylamine, Pentalita or 
Tetralita No 2, (H₃C₂)₂N-C₆H₄(NO₂)₃, mw 
346.18, N 24.28%; crys, mp 125-29°C, 
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°C; the 
reaction was accompanied by a copious 
evolution of nitrogen oxides. The resulting 
crys in the slurry were separated from the 
acids by centrifuging (Reps 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 NO₂ groups, because of 
difficulty of direct nitration of the group in 
meta position
Pentanitrocompld was used in Spain as 
a military explosive, under the name of 
Pentalita or Tetralita No 2 (Reps 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 penta-
nitrodiblanilines and their uses as 
military expls under the names of Tetralita 
No 1 and Tetralita No 2 or Pentalita) 
17) A. Forster & W. Couison, 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) 
(Nitrosod-N,N-dimethylaniline) 19) J.C. 
Earl & A.W. Mackney, JProcRysoc 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 
(Pentanitrodiblanilane); p 73 (Tetra-
nitrodiblanilane) 23) A.H. Blatt, 
OSRD 2041(1944) (2,4,6-Trinitrodimethylanil 
ine), 24) Vivas, Feigenspan & Ladreda, 
Vol 2(1946), 297-98 (Pentalita) 
25) F.M. Lang, MP 30, 373-92(1948) (Act 
of nitrous acid on N,N-dimethylanil 
ine) 26) L. Horn er & C. Betz el, Ann 579, 
175-92 (1953) & CA 48, 2625(1954) (N,N-Dimethyl-
2,4,6-trinitramilane) 27) L. Méard & 
M. Thomas, MP 38, 49(1956) (Prepn and 
props of N,N-Dimethyl-2,4-dinitroanil 
) 28) CondChemDict (1961), 393-R (N,N-Di 
methylanilane) 29) No later refs on 
Pentanitro- and Pentanitro derivs were 
found in the literature thru 1966

Dimethylanilines or Xyldines, also called 
Aminoxylanlenes, Aminodimethylbenzenes or 
Dimethylphenylamines, 
(H₃C₂)₂C₆H₈-NH₂, mw 121.18, N 11.56% 
Following six isomers exist: 
2,3-Dimethylaniline or (vic)c-Xylidine, liq, 
sp gr 0.991 at 15°C, mp < minus 15°C, bp 223-
224°C; sl sol in w; sol in alc & eth (Reps 1 
& 14) 
3,4-Dimethylaniline or (uns)c-Xylidine, 
crys, sp gr 1.076 at 17.5°C, mp 49-50°C, 
bp 224-226°C; v sl sol in w; sol in petr eth 
(Ref 2) (Not in Rep 14) 
2,6-Dimethylaniline or (vic)m-Xylidine, liq, 
sp gr 0.980 at 15°C, fr p 10-12°C, bp 216-217°C, 
isol in w; sl sol in alc & eth (Reps 3 & 14)
2,4-Dimethylaniline or (trans m-Xyldine, 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 (symm m-Xyldine), oil, sp gr 0.972 at 20/4°, bp 221–222°; solubilities not given (Ref 5) (Not in Ref 14)
2,5-Dimethylaniline or o-Xyldine, 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, \((\text{H}_3\text{C})_2\text{C}_4\text{H}(\text{NO}_2)_2\), NH_2, mw 211.18, 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, chl & benz (Ref 7, p479)
5,6-Dinitro-2,3-dimethylaniline, orn ndls (from alc), mp 143°; sol in acet, eth acet, warm benz & warm chl (Ref 7, p479)
2,6-Dinitro-3,4-dimethylaniline, orn-red ndls (from alc), mp 143°; sol in chl, benz & eth acet (Ref 4, p471)
5,6-Dinitro-3,4-dimethylaniline, dk, orn-red ndls (from acet), mp 212°; v sl sol in alc, benz & chl (Ref 7, p482)
3,5-Dinitro-2,6-dimethylaniline, yel plts (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 & chl (Ref 7, pp1130 & 613)
3,6-Dinitro-2,4-dimethylaniline, crys (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, crys, mp 94° (Ref 7, p1132)
2,6-Dinitro-3,5-dimethylaniline, yel crys, mp 101° (Ref 7, p1132)
3,4-Dinitro-2,5-dimethylaniline, crys, mp 172° (Ref 7, p490)
3,6-Dinitro-2,5-dimethylaniline, crys, 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 prep are given in the Refs

2,4-Dinitro-3,6-dimethyl-nitraniline; 4,6-Dinitro-2,5-dimethylphenyl-nitramine,
\((\text{H}_3\text{C})_2\text{C}_4\text{H}(\text{NO}_2)_2\), NH(NO_2); mw 256,18, N 21.87%; ndls (from alc + water), mp 130° (dec); mod sol in alc, aceto 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 nitate.
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-Trimethoxy-3,5-dimethyl-1-aniline or
2,4,6-Trimethoxy-3,5-dimethoxy-1-xyldine,
\((\text{H}_3\text{C})_2\text{C}_4\text{H}(\text{NO}_2)_2\), NH_2, mw 256,18, N 21.87%; yel crys, mp 206°; can be prepd by heating 2,4,6-trimethoxy-3,5-dimethoxy-1,3-dimethylanilene with alcoholic NH_3 in a tube, or from 5-bromo-2,4,6-trimethoxy-1-xyldine with alc NH_3 at 230° (Ref 9)
Tetranitrodimethylanilene or Tetranitro-1-xyldine, C_8H_7N_4O_6, not found in the literature thru 1966

Pentanitro-m-xyldene; 2,4,6-Trinitro-3,5-dimethyl-1-dinitroaniline or 2,4,6-Trinitro-3,5-dimethyl-1-dinitroaminobenzene,
\((\text{H}_3\text{C})_2\text{C}_4\text{H}(\text{NO}_2)_2\), NH(NO_2); mw 346,18, N 24.28%; crys, mp – explodes at high temp.
This compd is listed by Duin & Brackmann (Ref 11) as a brisant nitro expl, but its method of prep is not given in the abstract. It was considered as not having all of the desirable props for use in ammunition
Refs for Xyldenes: 1) Beil 12, 1101
N,N-Dimethylaniline-p-Azide,
(H₃C)₂N.C₆H₄.N₃; mw 162.19, N 34.55%;
yel leaflets, mp 40°, explodes at higher
temp. Horner & Gross (Ref 2) report its
depn: N,N-Dimethyl-p-aniline (13.6g) in
2N sulfuric acid is diazotized with 7g
NaNO₂ at 0–5°, treated with 2g Cu powd
and then with 7g NaN₃ in 50cc H₂O, the
mxt kept 2 hrs, excess 2N NaOH added,
kept 5 hrs at 0°, steam distd, the distillate
extrd with acet, and the extract dried &
evapd, giving the product. Ugi et al (Ref
3) report the prep as follows: N,N-Dimi-
thylaniline HCl salt (0.050 mole) in 50cc
2N HCl dld with 20–30g crushed ice
is treated while stirring with 3.70g NaN₃ in
15cc water, neutralized with CaCO₃ treated
with 4.0g NaN₃ 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)
(UV spectrum)

N,N-Dimethylaniline-p-azo-picrate,
(H₃C)₂N.C₆H₄.N₃ + C₃H₅N₃O₇; mw 391.30,
N 25.06%; yel ndls (from MeOH), mp 128°,
probably explodes at higher temp. Prep n:
to the residue from evapn (in darkness)
of an alc-chl sln 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 sln freed of PA by aq Na₂CO₃, and the
acet sln 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-diazone Hydroxide
and Derivatives
N,N-Dimethylaniline-4-diazone Hydroxide or
N,N-Dimethylaminobenzene-4-diazone Hydro-
dioxide, (H₃C)₂N.C₆H₄.N₃(OH). A hypo-
thetical compd known in the form of its
salts, some of which are expd:
Chloride, (H₃C)₂N.C₆H₄.NCl, mw 183.64,
N 22.88%; pale yel crys (from alc + eth),
mp – puffs off at ca 130°; sol in cold w &
in alcl; insol in eth; decmpd by hot w.
Hantzsch (Refs 1 & 2) prepd it by the low-
temp diazotization of N,N-dimethyl-p-phenyl-
enediamine with Na nitrite, while Stolle
(Ref 4) treated N,N-dimethyl-p-phenylen-
diamine hydrochloride with anil nitrite in
alcoholic medium
Perchlorate, (H₃C)₂N.C₆H₄.N(N).ClO₄, mw
247.64, N 16.98%; crysts, mp – exp on heat-
ing; 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,
(H₃C)₂N.C₆H₄.NH[(N₃)₂CuN₃Cu(N₃)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
NH₃; was obtd by dissolving Cu(N₃)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)(tetrozolyl-5)]-
diazone, called by Benzon Tetrazole-5-
azo-4-N,N-dimethylanilino,
(H₃C)₂N.C₆H₄–N.N.C.NH.N
II — II;
mw 217.23, N 45.14%, pale reddish lfts
(from alc), mp – begins to decomp ca 150°,
 expl ca 155°; diff sol in w & in eth; al 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. (H₃C)₂N.C₁₂₄.N.C(Na).N

mw 239.22, N 40.99%; Carmine red shiny crystals, 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 Methoxxyxylol or Dimethylanisol in Ger), H₃CO.C₂H₅(CH₂)₂; mw 136.19. Six isomers are found in the literature:
2,3-Dimethylanisole, crysts, mp 29⁰, bp 190⁰ & 85⁰ at 18mm press (Ref 1)
2,4-Dimethylanisole, liq, bp 192⁰, d 0.974 at 16⁰, nD 1.5190 at 16⁰ (Ref 2)
2,5-Dimethylanisole, oil liq, bp 186-88⁰, d 0.9693 at 12.8⁰, nD 1.5182 at 12.8⁰ (Ref 3)
2,6-Dimethylanisole, col oil, bp 181-83⁰, d 0.9619 at 13.6⁰, nD 1.5053 at 13.6⁰ (Ref 4)
3,4-Dimethylanisole, liq, bp 200-01⁰, d 0.9744 at 13.7⁰, nD 1.5198 at 13.7⁰ (Ref 5)
3,5-Dimethylanisole, col oil, bp 194⁰, d 0.9569 at 20⁰, nD 1.5110 at 20⁰ (Ref 6)

Other props & methods of prep are given in the Refs

4-Nitroso-2,6-dimethylanisole,
H₃CO.C₂H₂(NO)(CH₂)₂; mw 165.19, N 8.48%; yell plicts, mp 51⁰; was prepd by reaction of 4-nitroso-2,6-dimethylanisole with diazomethane in eth (Refs 1 & 2)
Refs: 1) Beil 6, 11740 2) F. Fichter & R. Gunst, Helv 22, 267, 274(1939)

Monanitrodimethylanisole,
H₃CO.C₂H₂(NO₂)(CH₂)₂; mw 181.19, N 7.73%. The following isomers are known:
5-Nitro-2,4-dimethoxyanisole, ndls (from alc), mp 56-57⁰; sl sol in alc & eth (Ref 1)
6-Nitro-2,4-dimethoxyanisole, prisms (from eth), bp 268-69⁰ (Ref 2)
3-Nitro-2,5-dimethoxyanisole, ndls (from MeOH), mp 62-62.5⁰; sol in w (Ref 3)
4-Nitro-2,6-dimethylanisole, liq ndls (from alc), mp 91-92⁰ (Ref 4)
6-Nitro-3,4-dimethoxyanisole, yel crystals (from alc), mp 79⁰ (Ref 5)
2-Nitro-3,5-dimethoxyanisole, exists as tablets (from MeOH), mp 43-46⁰ & as ndls (from dil MeOH), mp 35-35.5⁰ (Ref 6)
4-Nitro-3,5-dimethoxyanisole, ndls (from dil MeOH), mp 50-53⁰ (Ref 6)

Other props & methods of prep 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, 11740 5) Beil 6, 240 & 11731 6) Beil 6, 11765

Dinitrodimethylanisole,
H₃CO.C₂H₂(NO₂)₂(CH₂)₂; mw 226.19, N 12.39% Two isomers are known:
2,4-Dinitro-3,5-dimethylanisole, crysts (from alc), mp 172-75⁰; was prepd by heating 2,4-dinitro-3,5-dimethylanisole with methy liodide & aq NaOH (Ref 1)
2,4-Dinitro-3,6-dimethylanisole or 4,6-Dinitro-2,5-dimethylanisole, almost colorless ndls (from alc), mp 60⁰; 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, 11767 2) Beil 6, 497

Trinitrodimethylanisole,
H₃CO.C₂H₂(NO₂)₃(CH₂)₂; mw 271.19, N 15.50%. Two isomers are known:
3,4,6-Trinitro-2,5-dimethylanisole, crysts, mp 146⁰; was prepd by heating 2,4-dinitro-3,6-dimethylanisole with HNO₃ & H₂SO₄ (Ref 1)
2,4,6-Trinitro-3,5-dimethylanisole, ndls (from alc), mp 124-27⁰; obtd, with 2,4-dinitro-3,5-dimethylanisole, by nitrating 3,5-dinitro-3,5-dimethylanisole with a mix of concd HNO₃ & H₂SO₄ at -5⁰ (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(methyl-tetrazolyl)-dioxide. See Bis(1-methyl-1,2,3,4-tetrazolyl)-diazene in Vol 2 of Encycl, p B151-L and: M.M. Williams et al, JPhysChem 61, 263 (1957). They give heats of combstn 770.5 kcal/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, p 672-L.

Dimethylazoxyethane. See Azoxypropane in Vol 1, p A671-R

Dimethylbenzaldehyde and Derivatives

Dimethylbenzaldehyde (called 1'-Oxo-trimethyl-benzol, Dimethylbenzaldehyde or Dimethyl-1-formyl-benzol in Ger), \((\text{H}_3\text{C})_2\text{C}_6\text{H}_4\text{CHO}\); mw 134.17. The following isomers are known:

- 2,3-Dimethylbenzaldehyde, isolated as Oxime, crysts, mp 80-82° & as Semicarbazone, 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 prep are found in Beil.

Refs: 1) Beil 7, i1071 2) Beil 7, 310, (165), [240] & i1072 3) Beil 7, 311, (166) & i1072 4) Beil 7, 312, [240] & i1073

2-Azido-3,5-dimethylbenzaldehyde,
\((\text{H}_3\text{C})_2\text{C}_6\text{H}_4\text{N}_3\text{CHO}\); mw 175.19, N: 23.99%; ndls (from petr eth), mp 33-34°; volatile on steam bath; sol in org solvents; v sl sol in w; obt from the oxime of 3-oxo-5,7-dimethylindiazene,

\[
\text{C} \equiv \text{N} \equiv \text{OH}
\]

with NaOH and distilling on a steam bath.
The Azide on oxidation with \(\text{KMnO}_4\) yields 2-Azido-3,5-dimethylbenzoic Acid. Expl props of the Azide are not reported (Ref 1).

Refs: 1) Beil 7, 313 2) E. Bamberger & Demuth, Ber 34, 1317 (1901) 3) No later refs found thru 1966

Mono nitrodimethylbenzaldehyde,
\((\text{H}_3\text{C})_2\text{C}_6\text{H}_4\text{NO}_2\text{CHO}\); 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°; obt by nitrating the 2,4-isomer with \(\text{HNO}_3\) & concd \(\text{H}_2\text{SO}_4\) (Ref 1, p311)
- 6-Nitro-2,5-dimethylbenzaldehyde, yel ndls or pillets (from alc), mp 120°; prep from reaction of the 2,5-isomer with \(\text{KNO}_3\) & concd \(\text{H}_2\text{SO}_4\) in the cold (Ref 2)
- 2-Nitro-3,5-dimethylbenzaldehyde, ndls, mp 102-03°; sol in alc & petr eth; obt by nitrating the 3,5-dimethyl isomer with \(\text{KNO}_3\) & concd \(\text{H}_2\text{SO}_4\) in the cold (Ref 1, p313)

4-Nitro-3,5-dimethylbenzaldehyde, col crystals (from petr eth), mp 42-44°. Its prep is described by Steiner & Sorkin (Ref 3):

To 16g nitromesitylene, \((\text{H}_3\text{C})_2\text{C}_6\text{H}_4\text{NO}_2\cdot\text{CH}_3\)

in 48g \(\text{CS}_2\) 30g \(\text{CrO}_2\text{Cl}_2\) in 40ml \(\text{CS}_2\) 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 \(\text{Na}_2\text{SO}_4\) giving 8.8g crystals hydrolyzed with 10% \(\text{H}_2\text{SO}_4\) & 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 Mono nitro derivs are reported.
Dinitrodimeethylbenzaldehyde,
(H₃C)_2C₆H(NO₃)₂,CHO; not found in Beil
nor in CA thru 1966

Trinitrodimeethylbenzaldehyde,
(H₃C)_2C₆H(NO₃)₃,CHO; not found in Beil
nor in CA thru 1966

Dimethylbenzene and Derivatives

Dimethylbenzene or Xylene, C₆H₄(CH₃)₂, mw 106.16. Three isomers are known:
1,2 (or o-) Dimethylbenzene, col liq, sp gr 0.880 at 20/4°C, fp minus 25.2°C, bp 144.4°C;
insol in w; sol in alc & eth (Ref 1)
1,3 (or m-) Dimethylbenzene, col liq, sp gr 0.864 at 20/4°C, fp minus 47.9°C, bp 139.1°C;
insol in w; sol in alc & eth (Ref 2)
1,4 (or p-) Dimethylbenzene, plts, sp gr 0.861 at 20/4°C, mp 13.3°C, bp 130.4°C; insol
in w; sol in alc; v sol in eth (Ref 3)

Other props & methods of prep 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, N₃C₆H₄(CH₃)₂; mw 147.18, 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-bm aroma
tic smelling oil, bp 87–88°C; was obtd
from 4-amino-1,3-dimethylbenzene by diazo
tizing in HCl soln, followed by treating the diazonium chloride salt with concd aq
NH₃ (Refs 1, 4 & 5). No expl props are
reported
1-Azido-1,4-dimethylbenzene or p-Xylyl

Azide, aromatic liq, bp 94°C at 12mm press,
deglq on rapid heating; was prepd by warm
1-Nitrosohydroxime-1,4-dimethylbenzene,
CH₃C₆H₄CH₂N(NO₂),NH₂, with 10% H₂SO₄
on a water bath and distilling with steam
(Ref 2)
2-Azido-1,4-dimethylbenzene, yel oil, bp
90°C at 15mm press, volatile with steam; was
obtd by reacting 2,5-dimethylphenylhydroxime
with NaNO₂ & 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, J.OrgChem
26, 4110(1961) & CA 56, 8606–07(1962)

Mononitroso-dimethylbenzene, ON.C₆H₄(CH₃)₂;
mw 135.16, N 10.36%. Six isomers are known:
3-Nitroso-1,2-dimethylbenzene, wh ndls, mp
91–91.5°C; sol in benz & warm alc; sl sol in
eth & chlf; v sl sol in petr eth (Ref 1)
4-Nitroso-1,2-dimethylbenzene, yel-bm ndls
(from alc), mp 44–45°C; mod sol in petr eth;
sol in acet & warm alc (Ref 1)
2-Nitroso-1,3-dimethylbenzene, col ndls, mp
141.5°C (dec); sol in benz & chlf; sl sol in
warm alc & acet; v sl sol in eth; diff sol
in petr eth (Ref 2)
4-Nitroso-1,3-dimethylbenzene, wh prisms
(from alc), mp 42.5°C; sol in petr eth & eth;
sol in acet & chlf; vol in steam (Ref 3)
5-Nitroso-1,3-dimethylbenzene, crysts, mp
59°C(Ref 4)
2-Nitroso-1,4-dimethylbenzene, wh ndls
(from alc), mp 101.5°C; readily sol in eth &
petr eth (Ref 5)

Other props & methods of prep are
found in the Refs
Refs: 1) Beil 5, 367 2) Beil 5, 377 &
5, [841] 5) Beil 5, 387 & [861]

Mononitrodimeethylbenzene, O₂N.C₆H₄(CH₃)₂;
mw 151.16, N 9.27%. Six isomers are known:
2-Nitro-1,2-dimethylbenzene, almost color
less ndls (from alc), mp 15°C; bp 240°C; d
1.4020 at 20°C, nD 1.5441 at 20°C(Ref 1)
4-Nitro-1,2-dimethylbenzene, yel prisms
(from alc), mp 28.5–30°C, bp (decomp), sol
in common org solvs (Ref 2)
2-Nitro-1,3-dimethylbenzene, liq, crystals on cooling below RT, bp 221–222°, 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 279° at 739 mm press (Ref 4)
2-Nitro-1,4-dimethylbenzene, lt yel liq, bp 239–41°, d 1.132 at 15°, n\textsubscript{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]

Nitrosodinitrodimethylbenzene,
O\textsubscript{2}N\cdot C\textsubscript{3}H\textsubscript{2}(NO)(CH\textsubscript{3})\textsubscript{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 reac-
tion of H\textsubscript{2}SO\textsubscript{4} 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
preparing 6-nitro-4-hydroxylamino-1,3-
dimethylbenzene with FeCl\textsubscript{3} in alc (Ref 1, p844 & Ref 2)
4-Nitroso-5-nitro-1,3-dimethylbenzene, gm-
yel ndls (from alc), mp 134°(dec); was prepd by
reacting an aq Na\textsubscript{2}SO\textsubscript{4} 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-hydroxyl-
amino-1,4-dimethylbenzene with FeCl\textsubscript{3}
in aq alc in the cold (Ref 1, p863 & Ref 2)
Their expn props are not reported
Refs: 1) Beil 5, [843], 844 & 863]
165(1935) & CA 29, 2931(1935)
3) R. Kuhn et al, Ber 70, 1293, 1296, 1314(1935)
& CA 31, 6211, 6239(1937)

Azidonitrodimethylbenzene,
O\textsubscript{2}N\cdot C\textsubscript{6}H\textsubscript{4}(CH\textsubscript{3})\textsubscript{2}.N\textsubscript{3}; mw 196.16, N 14.28%
Two isomers are known:
4-Azido-5-nitro-1,3-dimethylbenzene, wh
ndls (from alc), mp 60°; sol in common org
solv; was obtd by reaction of NH\textsubscript{3} on the
diazoperbromide of 5-nitro-4-amino-1,3-
dimethylbenzene; by heating under vacuum
at 130°, the 4,5-Dinitroso-1,3-dimethyl-
benzene 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 expn props are not reported
Refs: 1) Beil 5, 381, 382
2) E. Grand-
mougin & (?) Michel, Ber 25, 3342(1892)
3) T. Zincke & (?) Schwarz, Ann 307, 47
(1899)
4) No later refs found

Dinitrosodimethylbenzene, C\textsubscript{6}H\textsubscript{4}N\textsubscript{2}O\textsubscript{2}; mw
164.16, N 17.07%. Two isomers are known:
4,5-Dinitroso-1,3-dimethylbenzene,
\[
H\cdot C\equiv C(CH\textsubscript{3})\rightarrow C\cdot N\cdot O
\]
H\textsubscript{2}C\cdot C\equiv CH --- C\cdot N\cdot O;
plkts (from benz), mp 108–09°; vol in
steam; readily sol in common org solv;
less sol in benz & 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,
O\cdot N\cdot C\textsubscript{6}H\textsubscript{4}(CH\textsubscript{3})\textsubscript{2}.N\textsubscript{2}O (?);
mp 250°; diffc
sol; prepd by heating a soln of 1,4-dimethyl-
benzoquione-dioxime in NaOH with K
ferric cyanide soln (Ref 1, p659 & Ref 2)
No expn 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,
O\cdot N\cdot C\textsubscript{6}H\textsubscript{4}(NO\textsubscript{2})(CH\textsubscript{3})\textsubscript{2}.N\textsubscript{2}O; mw 209.16, N
20.09%; yel ndls (from abs alc), mp 116°;
mod sol in benz & glas 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 expn props are not reported
D 1323

Refs: 1) Beil 7, 657  2) (?) Drost, Ann 313, 313 (1900)

5,6-Dinitrosobenzene, 2-nitro-1,4-dimethylbenzene or 5-Nitro-4,7-dimethylbenzofuroan-1,3-oxyd or 5-Nitro-4,7-dimethylbenzofuroxan,

\[
\begin{array}{c}
\text{O}_2\text{N} \\
\text{CH}_3 \\
\text{CH}_3
\end{array}
\]

yel crysts (from benz., mp 81\(^\circ\)); mod sol in alc & benz; decomp by alkalies; was prepd by heating 3,5-dinitro-2-azido-1,4-dimethylbenzene at 105-30\(^\circ\) (Refs 1 & 2).

Its expl props are not reported


**Dinitrodimethylbenzene**, \((\text{O}_2\text{N})_2\text{C}_6\text{H}_2(\text{CH}_3)_2;\) mw 196.16, N 14.28%. Fourteen isomers are known:

3,4-Dinitro-1,2-dimethylbenzene, ndls (from alc), mp 82\(^\circ\), bp-explodes on heating in a tube at 413\(^\circ\); sol in org solvs; obt by nitrating o-xylene (Ref 1, p369; Ref 2, p181; Ref 3, p287 & Ref 4, p822)

3,5-Dinitro-1,2-dimethylbenzene, crystals (from alc), yel ndls (from alc, or chl + petr eth), mp 75–76\(^\circ\); sinters at 71\(^\circ\), bp-explodes on heating in a tube at 438\(^\circ\); sol in benz, acit, chl & eth acet; prepd by nitrating 4-nitro deriv with HNO\(_3\)/H\(_2\)SO\(_4\) mixed acid (Ref 1, p369; Ref 2, p181 & Ref 4, p823)

3,6-Dinitro-1,2-dimethylbenzene, ndls (from alc), mp 56–60\(^\circ\) & 89–90\(^\circ\); sol in benz and 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\(^\circ\); 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**, \((\text{O}_2\text{N})_2\text{HC}_6\text{H}_4\text{CH}_3;\) obt by reaction of NO\(_2\) & CuSO\(_4\) on o-xylene at 15–20\(^\circ\); forms a Potassium salt, yel ndls (from aq KHC\(_2\)O soln)(Ref 4, p823)

2,4-Dinitro-1,3-dimethylbenzene, yel crysts (from alc), mp 84–86\(^\circ\); prepd by reacting 4-nitro-2-amino-o-xylol with K persulfate & H\(_2\)SO\(_4\) in the cold, and HNO\(_3\) (d 1.48) at 40–45\(^\circ\) (Ref 1, p379; Ref 3, p295 & Ref 4, p844)

2,5-Dinitro-1,3-dimethylbenzene, col crystals, becoming yel in the light (from alc), mp 101\(^\circ\); 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\(^\circ\); prepd by warming 4-nitroso-5-nitro-1,3-dimethylbenzene with aq H\(_2\)O\(_2\) & HNO\(_3\) in glacial acetic acid on a water bath (Ref 1, p380; Ref 3, p295 & Ref 4, p844)

4,6-Dinitro-1,3-dimethylbenzene, col prisms (from alc), mp 92–94\(^\circ\); 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**, \((\text{O}_2\text{N})_2\text{HC}_6\text{H}_4\text{CH}_3;\) prisms (from alc or benz), mp 53–54.5\(^\circ\), obt by reacting an excess of NO\(_2\) on m-xylene in the presence of CuSO\(_4\) at 15–20\(^\circ\); forms a Potassium salt, K\(_2\)C\(_6\)H\(_4\)N\(_2\)O\(_4\), crystals (from w), mp 125\(^\circ\)(dec) (Ref 4, p844)

2,3-Dinitro-1,4-dimethylbenzene, monoclinic prisms (from alc or benz), mp 90–93\(^\circ\); obt 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\(^\circ\) & 147–48\(^\circ\); difc 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\(^\circ\); 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)
4-Azido-2,5-(or 5,6)-dinitro-1,3-dimethylbenzene, wh plts (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, plts (from dil alc), mp 71–73°; mod sol in benz & acet ac; sl sol in alc; v sl sol in benzene; prep from 3,5-dinitro-p-xyloldiazonium-2-perbromide thru reaction with aq NH₃; 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).

Ref: 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), (O₃N)₃C₆H(CH₃)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 ac; was obtd with other isomers on nitrating 1,1-dimethylcyclohexane with a mixt of 1 vol HNO₃ & 2 vols H₂SO₄ on a w bath (Ref 1). Marqueyrol & Lioriette (Ref 5) detd its press when fired in a 1.125cc bomb with 1 g priming chge to be 1670kg/cm² & 3080 at d 0.3.


TRINITROXYLENE, Commercial (TNX) or Xylyl, known in Russia as K7ill or Kzill, 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 sub stance prepd by nitration of commercial xylene with mixed nitric-sulfuric acid. Accdg to Pepin Lehalleur (Ref 1), Solomonina developed during WWI one-stage nitration of xylene with a yield of 75%. TNX is insol in water, is slightly more sensitive to initition 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 sensi-
tive to initiation. This is due, according to Shilling (Ref 2, p99), to the exceptionally small crystalline structure of cast mixtures 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 charge (such as Tetryl) and can be initiated by a very strong detonator alone. TNX has also been used in industrial expls.


H₂N(H₃C)C₄H₃-C₆H₅(CH₃)NH₂, 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-dimethylidiphenyl or 2,6-Dimethylbenzidine,
H₂N.C₆H₄-C₆H₂(CH₃)₂.NH₂, ndls (from benz + pett eth), mp 124°; sol in alc, eth & benz; sl sol in pett eth & hot w; forms salts (Ref 3, p115)
5,5'-Diamino-2,2'-dimethylidiphenyl or 5,5'-Diamino-2,2'-ditolyl,
H₂N(H₃C)C₆H₅-C₆H₃(CH₃).NH₂, crysts, mp 96–98°, bp 225–228° at 13mm press; v sl sol in chlf; sol in eth, benz, hot alc & hot pett eth; forms salts (Ref 3, p115)
6,6'-Diamino-2,2'-dimethylidiphenyl 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'-dimethylidiphenyl or 2,3'-Dimethylbenzidine, amorph mass, bp 243–46° (Ref 1, p256 & Ref 3, p116)
2,4'-Diamino-3,3'-dimethylidiphenyl or 3,3'-Dimethylidiphenylene, ppt; forms a Hydrochloride salt, C₁₄H₁₆N₃+2HCl, ndls, v sol in w (Ref 1, p256)
4,4'-Diamino-3,3'-dimethylidiphenyl; 3,3'-Dimethylbenzidine or o-Tolidine, plitts, mp 126–29°; sol in alc & eth; sl sol in w; forms numerous cryst salts with inorg & org comps (Ref 1, p256, Ref 2, p79 & Ref 3, p116)
6,6'-Diamino-3,3'-dimethylidiphenyl, crysts, mp 80–83° (cited in Ref 5)
2,2'-Diamino-2,2'-dimethylidiphenyl, 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)

Dimethylbenzidine and Derivatives

Dimethylbenzidine or Tolidine, C₁₄H₁₆N₂;
mw 212.28, N 13.20%. Eleven isomers exist: N,N'-Dimethylbenzidine, [-C₆H₄.NH.CH₃]₂,
ndls (from alc), mp 74–76° & 81–82°; sl sol in alc, eth, chlf & benz; v sl sol in cold pett eth; forms salts (Ref 1, p220 & Ref 3, p97)
N,N'-Dimethylbenzidine,
H₂N.C₆H₄-C₆H₄.N(CH₃)₂, plitts (from alc or w), mp 146°; becomes green & bm on warming in the light; sol in dil HCl &
H₂SO₄; forms salts (Ref 3, p97)
4,4'-Diamino-2,2'-dimethylidiphenyl; 2,2'-Dimethylbenzidine or o-Tolidine,

Dimethylbenzidine or Tolidine, C₁₄H₁₆N₂;
mw 212.28, N 13.20%. Eleven isomers exist: N,N'-Dimethylbenzidine, [-C₆H₄.NH.CH₃]₂,
ndls (from alc), mp 74–76° & 81–82°; sl sol in alc, eth, chlf & benz; v sl sol in cold pett eth; forms salts (Ref 1, p220 & Ref 3, p97)
N,N'-Dimethylbenzidine,
H₂N.C₆H₄-C₆H₄.N(CH₃)₂, plitts (from alc or w), mp 146°; becomes green & bm on warming in the light; sol in dil HCl &
H₂SO₄; forms salts (Ref 3, p97)
4,4'-Diamino-2,2'-dimethylidiphenyl; 2,2'-Dimethylbenzidine or m-Tolidine,
3,5,3',5'-Tetranitro-N,N'-dimethylbenzidine,
\([-C_6H_2(NO_2)_2N\cdot NH \cdot CH_3]_2\); mw 392.28, N 21.43%;
red ndls (from phenol), mp-dec at 282°; sol
in hot phenol; insol in glacial ace & 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'-diamino-
dimethyldiphenyl or N,N',3,5,3',5'-Hexanitro-
N,N'-dimethylbenzidine,
\([-C_6H_2(NO_2)_2N\cdot NH \cdot CH_3]_2\); it yel ndls
(from concd nitric acid & pptd with w), mp-
explodes above 220°; sol in phenol & anilne;
insol in alc; was prepd by nitrating the tetra-
nitro 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'-dimethyl-
benzidine; N,N',4,4',6,6'-Hexanitro-N,N'-
dimethyl-3,3'-biphenyldiamine (CA nomenclature);
or 4,4',6,6'-Tetranitro-3,3'-bis(methyl-
nitramino)biphenyl (called by van Alphen),
\([-C_6H_2(NO_2)_2N\cdot NH \cdot CH_3]_2\); crystals, mp 210°;
soley in alc; was prepd by nitrating the
corresponding tetranitro-dimethylamino deriv
with HNO_3-H_2SO_4 on a w bath (Ref 2). Its
explos 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 Acid, Xylic or Methyltoluic Acid,
(H_3C)_2C_6H_4COOH, mw 150.17, O 21.31%.
Six isomers exist:
2,3-Dimethylbenzoic Acid, crystals (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,
crystals (from cyclohexane by sublimation), mp
125–26°, bp 267° at 727 mm press; sol in
toluene, xylene, benzene, chin & acet; al-
most insol in cold w (Ref 2)

2,5-Dimethylbenzoic Acid, crystals (from cyclo-
hexane), mp 132–34°, bp 268°, vol in steam;
sol in alc; v sl sol in cold w (Ref 3)
2,6-Dimethylbenzoic Acid, crystals (from w or
cyclohexane), mp 115.8–17°; sol in eth; v sl
sol in cold petr eth (Ref 4)
3,4-Dimethylbenzoic Acid, crystals (from alc or
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, crystals (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]
6) Beil 9, 536, (210), [354] & [2444]

2-Azido-3,5-dimethylbenzoic Acid,
(H_3C)_2C_6H_4(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-dimethylbenzalde-
hyde with K_Mn_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_4(NO_2)_2COOH; mw 208.17, N 13.46%;
crystals, mp 166°; prepd by reacting x,x-
dinitroso-2,4-dimethylphenylglyoxylic acid
with K_2Cr_2O_7 & H_2SO_4 (Refs 1 & 2). Its
explos props are not reported.
Refs: 1) Beil 9, 534 2) Claus, JPraktChem
[2] 41, 490 (1890)

Mononitrodimethylbenzoic Acid,
(H_3C)_2C_6H_4(NO_2)COOH; mw 195.17, N 7.18%.
Several isomers are known:
3-Nitro-2,4-dimethylbenzoic Acid, crystals (from
alc or benzine + benzene), mp 195–98°; sol
in alc, eth, chlf & CS₂; sl sol in hot w; v sl in cold w; forms cryat 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, crys (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, l-vel crys (from dil alc), mp 225.5–227°, crys (from w)
+ 1 mol water, mp 174–76° (Ref 5)

Other props & methods of prepns are given in Refs
5) Beil 9, [2446]

x-Nitrosos-α-nitro-2,4-dimethylbenzoic Acid (?)
(H₅C)₃C₆H(ONO)₂COOH, mw 224.17, N 12.50%; ndls (from hot w), mp 256°; sol in
hot w; sl sol in acet ac; almost insol in CS₂ & benz; was prepd by heating 2,4-dimethyl-
phenylglyoxylic acid with an excess of concd HNO₃ (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 (1899)

Dinitrodimethylbenzoic Acid,
(H₅C)₃C₆H(NO₂)₂COOH; mw 240.17, N 11.67%.
Only three isomers are known:
3,5-Dinitro-2,4-dimethylbenzoic Acid, crys (acetie 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 KMnO₄ (Refs 1 & 4). Its
expl props are not reported
2,4-Dinitro-3,3-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 prism., mp 215.5–216°; mod sol in alc & eth; sl sol
in hot xylene; v sl sol in chlf & w; prepns (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 Dimethyl-
benzoic Acid were found in the literature thru 1966

Dimethylbenzophenone and Derivatives

Several isomers are known, but only one has
been nitrated to form a tetranitro deriv:
2,4-Dimethylbenzophenone, (H₅C)₃C₆H₃CO-C₆H₃,
oil, bp 362° & 178–80° at 12mm press; was prepd
by treating m-xylene with benzoyl chloride &
AlCl₃ in CS₂ and by other methods (Ref)
Ref: Beil 7, 449, (239), [386], 12176

Azido, C₁₈H₁₈N₃O, and Diazido, C₁₈H₁₈N₆O,
derivs, not found in the literature thru 1966

Mononitro, C₁₈H₁₈NO₂, and Dinitro, C₁₈H₁₈N₂O₄,
derivs. Several are described in the literature,
but none is expl

Trinitrodimethylbenzophenone, C₁₈H₁₁N₃O₇,
mw 345.26, N 12.17%. Two isomers are known:
3,5,3'-Trinitro-2,4-dimethylbenzophenone,
(H₅C)₆(ONO)₂C₆H₃CO-C₆H₃, mp 139–40°; mod
sol in glac acet ac, benz & acetone; sl sol in
hot alc; v sl sol in eth, petr eth & CS₂; insol
in w; can be prepd by nitrating 2,4-dimethyl-
benzophenone; 3'-nitro-2,4-dimethyl- or 3,5-
dinitro-2,4-dimethylbenzophenone with mixed
acid (Refs 1 & 3)
3',3'-Dinitrodimethylbenzophenone,
C₁₈H₁₁(NO₂)₂; 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, 4087 (1929)

3,5,3',5'-Tetranitro-2,4-dimethylbenzophenone,
(H₅C)₆(NO₂)₃C₆H₃CO-C₆H₃(NO₂)₅; 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 prep'd by nitratimg 3',5'-dinitro-2,4-dimethylbenzenophenone 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. Chardonness, 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,

(H₃C)₂C₆H₄CH₂NH₂; mw 135.20, N 10.36%; liq, bp 217–18°, d 0.9500 at 20°, nD 1.53046 at 20.5°; forms a Picrate, C₆H₄N⁺+C₆H₅N₂O₇, yel crystals 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,

(H₃C)₂C₆H₄(NO₂)₂CH₂NH₂; mw 225.20, N 18.66%; liq; obtd by heating the acetyl deriv, (H₅C)₂C₆H₄(NO₂)₂CH₂NH.CO.CH₃, with HCl (d 1.1) in a tube at 150°; forms a H₂dichloride salt, C₆H₁₄N₂O₄·HCl, plts, dec at 245°; and a Picrate, C₆H₁₄N₂O₄·C₆H₄N₂O₇; 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,

HC — C(CH₂,N₃) — C(CH₃)

HC — C(CH₃) — CH

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 prep'd by Mayer by treating the nitroso-hydraxine, C₆H₁₃N₃O, 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

**a,c-Dimethylbenzyl Hydroperoxide (DMBH) or Cumene Hydroperoxide.** Its properties are described in Vol 3, pp C574-R & C575-L, but method of prep, 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 prep from dimethylbenzylcarbinol, 90% H₂O₂ and concd H₂SO₄ 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 preps involving 90% hydrogen peroxide be carried out on a small scale (no more than 5g 90% H₂O₂)

**Dimethylbiguanides**

1,1-Dimethylbiguanide or N,N-Dimethyl N'-guanlyguanidine,

(H₃C)₂N,C(NH)NH.C(NH₂)H₂:NH, mw 129.17, N 54.22%; known as salts, such as, Hydrochloride, C₆H₁₄N₃HCl, prisms (from w), mp 235°; and Picrate, mp 219° (Refs 1 & 3)

1,2-Dimethylbiguanide or N,N'-Dimethyl N'-guanlyguanidine,

H₃C.N.C(NH₂)₃:NH.C(NH₂)₃:NH; known as the Hydrobromide salt, C₆H₁₄N₃H₂HBr, crystals (from w), dec at 240–45° (Ref 2, p579)

1,5-Dimethylbiguanide,

H₃C.NH.C(NH₂)NH.C(NH₂)NH.CH₃ Sulfate salt, C₆H₁₄N₃H₂SO₄, dec at 200° (Ref 2, p572)
Dimethyl-biimidazole and Derivatives

**4,4'-Dimethyl-2,2'-biimidazole.**

\[
\begin{align*}
\text{HC} & \quad \text{NH} \\
\text{H}_3C\text{C} & \quad \text{N} \quad \text{C} \quad \text{C} \\
\text{N} & \quad \text{C} \quad \text{C} \quad \text{N} \quad \text{C.CH}_3
\end{align*}
\]

mw 162.19, N 34.55%; isolated as the Dipicrate, C\(_6\)H\(_{11}\)O\(_4\)+2C\(_6\)H\(_3\)N\(_3\)O\(_7\)·ndls, dec 230° (Ref 4).

**4,4'-Dimethyl-1,1',5,5'-tetranitro-2,2'-biimidazole.**

\[
\begin{align*}
\text{H}_3C\text{C} & \quad \text{N} \quad \text{C} \quad \text{C} \\
\text{N} & \quad \text{C} \quad \text{C} \quad \text{N} \quad \text{C.CH}_3
\end{align*}
\]

mw 342.20, N 32.75%; it yel crys, mp – dec 239–40°; was obtd when the anhyd Na salt of 4-methyl-1,1',5,5'-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\(_6\)H\(_{11}\)N\(_4\)O\(_9\)Na\(_2\), which in hot MeOH·Me ONa yields 1,1'-dimethoxy-4,4'-dimethyl-5,5'-dinitro-2,2'-biimidazole, dec at 242° (Ref 3).


**Dimethyl-biphenyl; Dimethyl-diphenyl or Ditolyl.**

See Bitolyl in Vol 2 of Encycl, p B163

**N,N-Dimethyl-p-biphenylylamine.** See N,N-Dimethylaminobiphenyl

**N,N'-Dimethyl-N,N'-bis[phenyl]-urea; N,N'-Dimethyl-carbanilide or N,N'-Dimethyl-diphenylurea.** See Centralite 2 in Vol 2, pp C137ff

**N,N'-Dimethyl-N,N'-bis[2,4-dinitrophenyl]-urea.** See Tetrantoicentralite, 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; vs sl sol in w & chlf;

almost insol in eth & petr eth (Ref 1)

**1,5-Dinitroso-1,5-dimethylbiuret,**

HN[C.O.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;

vs sl sol in cold w & petr eth; was obcd when 1,5-dimethylbiuret reacted with N\(_2\)O\(_4\) 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\(_3\)C\(_2\))\(_2\)BOOC\(_3\)H, mw 87.92; col liq of negligible vap pressure at –118°; compd is expl; two occurred during transfer in vacuo & one occurred during prep; 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 cmpds with NH\(_3\) and pyridine

Refs: 1) Beil, not found  2) R.C. Petry & F.H. Verhoek, JACS 78, 6416–17 (1956) & CA 51, 4936 (1957)

**Dimethylbutadiene and Derivatives**

2,3-Dimethyl-butadiene-1,3 or Di-isopropenyl, [H\(_3\)C: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,**

\[
\begin{align*}
\text{CH}_3 & \quad \text{O} \\
\text{O} & \quad \text{CH}_3 \\
\text{C} & \quad \text{O} \\
\text{C.CH}_3 & \quad \text{CH}_2 \\
\text{CH}_2 & \quad \text{X}
\end{align*}
\]

mw (114.14)x, O 28.04%; yel, honeylike substance, exploding violently at ca 140°

Was prepd by oxidation of 2,3-dimethylbuta-
diene 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
Re fs: 1) Beil 1, 256, (118), [232] & 1991 l
(Dimethylbutadiene) 2) Beil (Peroxide
Polymer - not found) 3) K. Bodendorf,
ArchPharm 271, 1 (1933) 4) P.N. Kogerman,
SitzberNaturforscbGesUnivTartu 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,
$\text{H}_3\text{C} \cdot \text{C} \cdot (\text{CH}_3)\gamma \cdot \text{CH}_2 \cdot \text{CH}_3$; mp 86.17, liq, sp gr
0.649 at 20°/20, fr p -99.7°, bp 49.7°, fl p
-32°C. Can be prep 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,
$\text{CH}_3 \cdot \text{CH} \cdot (\text{CH}_3) \cdot \text{CH} \cdot (\text{CH}_3) \cdot \text{CH}_3$; liq, sp gr
0.662 at 20°/4, fr p -128.4°, bp 58.0°,
flash p 29°C. Can be prep by alkylation
of ethylene with isobutane using Al chloride
catalyst. Used as a component of high oc-
tane fuel (Refs 2 & 8)
2,2-Dimethyl-1,3-dinitrobutane,
$\text{(O}_2\text{N})\text{H}_3\text{C} \cdot \text{C} \cdot (\text{CH}_3)\gamma \cdot \text{CH} \cdot (\text{NO}_2) \cdot \text{CH}_3$; mw 176.17,
N 15.90%, OB to CO$_2$ minus 127%; liq,
bp 128-32° at 9 mm press; was prep when a
mixture of nitro-tert-butyl alc, nitroethane &
piperidine was kept at RT for 3 months;
after neutralization & fractionation, the pro-
duct was obt (Refs 3 & 7)
2,3-Dimethyl-2,3-dinitrobutane or sym-
Tetramethyl-dinitroethane,
$\text{CH}_3 \cdot \text{C} \cdot (\text{CH}_3) \cdot (\text{CH}_3) \cdot \text{C} \cdot (\text{CH}) \cdot \text{CH}_3$;
$\text{NO}_2$ $\text{NO}_2$
mw, N & OB to CO$_2$ - same as
above; fts (from alc), sp gr 1.25; mp decmp
at 210–14°; explodes above 360°; sublimes
at 120–50° at 15 mm pressure; insol in w; sl
sol in ligroin, petroleum eth; sol in alc,
toluene, acet ester, hot eth, acetone, chl
and benzene; can be prep by shaking 2-
brom-2-nitropropane with a large am of pal-
verized 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 sensi-
tivity to impact to TNT, but difficult to de-
tonate completely; heat of combstn 898.8
kcal/mole; sl hygroscopic but fairly stable to
heat. Its power was not determined on an
account of incomplete detonation (Refs 4 & 6)

2,2-Dimethyl-3,3-dinitrobutane,
$\text{(H}_3\text{C})_2\text{C} \cdot (\text{NO}_2)_2 \cdot \text{CH}_3$; crysts, having a
strong camphor odor, very volatile, mp
173–74°; easily sol in ether; obtd in a small
quantity from pinacoline oxime & N$_2$O$_4$, in
addn to pinacoline nitramine (Ref 5)

NOTE: No higher nitrated derivs of Dimethyl-
butane were found in the literature thru 1966
Re fs: 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 1944 (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)

Dimethylbutane and Derivatives
Dimethylbutane, $\text{C}_6\text{H}_{12}$; mw 84.16. Three
isomers are known:
2,2-Dimethyl-butane-(3) or tert-Butylethylene,
$\text{H}_2\text{C} \cdot \text{C} \cdot (\text{CH}_3)_2$; liq, bp 41.2°, d 0.6549 at
18°/4, n_D 1.37667 (Ref 1)
2,3-Dimethyl-butane-(1) or 1-Methyl-1-
isopropylethylene, $\text{(H}_3\text{C})_2\text{C} \cdot \text{C} \cdot (\text{CH}_3) \cdot \text{CH}_2$;
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-butane-(2) or Tetramethyl-
ethylene, $\text{(H}_3\text{C})_2\text{C} \cdot (\text{CH})_2$; liq, fr p 74.2°,
bp 73.2°, d 0.7076 at 20°/20 (Ref 3)

Other props & methods of prepn are given
in the Refs
Re fs: 1) Beil 1, 217, (91) & [814]
2) Beil 1, 218, (91), [195] & l816]
3) Beil 1, 218, (91), [195] & l817]

4-Nitroso-1-nitro-2,3-dimethyl-butene-(2),
ON-CH₃, C(CH₃)₂C(CH₃)₂CH₂NO₂; mw 158.16, N 17.71%; ndls (from eth acet),
bp 104–05°; sl sol in cold benz; obt'd with other products by reaction of N₂O₄ on 2,3-
dimethyl-1,3-butadiene in ether in the cold (Refs 1 & 2). Its expl props are not rep-
ported
Refs: 1) Beil 1, [l820] 2) A.A. Ivanov,
JGenChem(Russ) 16, 648 (1946) & CA 41,
1202 (1947)

1,4-Dinitro-2,3-dimethyl-butene-(2)
O₂N.CH₂-C(=CH₂)₃C(CH₃)₂CH₂NO₂; 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 obt'd with other pro-
ducts when 2,3-dimethyl-1,3-butadiene in
ether was treated with ice-cold N₂O₄ and
the liq N₂O₄ adduct was steam distilled
and cryst'd on standing over sulfuric acid
(Refs 1 & 2). Its expl props are not rep-
ported
Refs: 1) Beil 1, [l820] 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.
H₃C.CH₂.C(CH₃)₂CH₃; mw 84.16; may be
considered as the parent compd of its ni-
trated deriv although not used to prep it
(Ref 1):
3,3-Di(nitratomethyl)-butene-1 or 3,3-
Bis(nitratomethyl)-butene-1,
H₃C.CH₂.C(CH₃)₂NO₂CH₃; mw 206.16,
N 13.59%; oxygen-rich monomer; no props
are reported except IR spectrum; was prep'd
by reaction of ethyl methyl ketone with for-
maldehyde, 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 polymer-
ized, of propellants for rockets having a fa-
orable oxygen balance (Ref 3)
Refs: 1) Beil 1, 217, (191) & l814]
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-tert-butyl-benzene,
(H₃C)₃C.C₆H₃.C(CH₃)₃; mw 162.26, liq, fr p
21.5°, bp 205.5–206.5°, d 0.8619 at 30°;
α D 1.4935 at 25°(Ref 1)

2-Azido-4,6-dinitro-1,3-dimethyl-5-tert-
butylbenzene, (H₃C)₃C(NO₂)₂(NO₂)₃C(CH₃)₃,
mp 293.28, N 23.88%; wth plrts (from alc),
mp 89° & 146°; sol in org solvs; insol in w;
was prep'd by reaction of NH₃ on the 4,6-
dinitro-2-amino deriv, diazotizing and con-
verting with K petrobromide soln (Ref 2)

2,4,6-Trinitro-1,3-dimethyl-5-tert-butylbenzene,
(H₃C)₃C(NO₂)₃C(CH₃)₃; ndls (from alc),
exist in a RT stable form, mp 107°; form I,
mp 114°; and a form II, converted from form I;
was prep'd by nitrating the parent compd with
nitric acid (d 1.5) & concd sulfuric acid (Revs
3 & 4). Its expl props are not reported
Refs: 1) Beil 5, 447, (213), [339] & l1032]
2) Beil 5, 448 & [340] 3) Beil 5, 448, (213),
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,
H₃C.CH₂.C(CH₃)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,
H₃C:C(NO₂)₂-C(CH₃)₂-COOH; mw 206.16,
N 13.59%; monoclinic crystals (from alc), volatile at 100°, mp 215°(dec); sol in alc- sol in hot w; v sl sol in cold w; was prep’d by heating camphor with nitric acid; forms several crystal metallic salts, some of which are unstable on heating, for example, Barium salt, 
Ba(C₆H₅N₂O₆)₂ + 3H₂O or 5H₂O, deflyr on heating above 120° (Refs 2 & 3)
Refs: 1) Beil 2, 335, (143), [293] & [1756]
2) Beil 2, 337 (Dinitrodibutyril Acid)
3) Kullheim, Ann 163, 231 (1872) & Kachler, Ann 191, 143, 156 (1878)

Dimethyl-Cadmium or Cadmium Dimethyl,
(CH₃)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 prep’d by reaction of methyl Mg bromide & CdCl₂, or from methyl Mg iodide & CdBr₂ in ether (Refs 1 & 2). At 70° vapor explodes in air

Dimethylcamphorate (Camphorate de méthyle in Fr),
H₂C·(H₃C)₂.C·H₃·C·COOH
H₂C·CH₃·C(CH₃)₂
COO·CH₃
mw 182.25; liquid which can be distilled under reduced pressure; was prep’d 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. Ficherouille, MP 30, 283-300 (1948)

Dimethylcarbamoyl Azide, (H₃C)₂N·CON₃,
mw 114.11, N 49.10%, liq, bp 59° at 15mm press; explodes violently when heated in a free flame; was prep’d by long heating of dimethylcarbamide chloride with NaN₃ in acetone (Refs 1 & 2)

N,N-Dimethyl-carbanilide. See Centralite 2
in Vol 2 of Encycl, pp C137ff

Dimethylcoumarin and Derivatives
6,7-Dimethylcoumarin or Dimethylcumarin,

\[
\text{H}_2\text{C} - \text{CH} = \text{CH} - \text{CO} - \text{OH}
\]

mw 174.19; ndls (from alc), mp 148-49°; was prep’d by heating o-xyleneol, malic acid & concd sulfuric acid (Ref 1)

3,5,8-Trinitro-6,7-dimethylcoumarin,

\[
\text{H}_2\text{C} - \text{CH} = \text{C} \cdot \text{NO}_2
\]

mw 309.19, N 13.59%; yel prisms (from glacial ac); mp 213-16° (dec); was prep’d 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 (Trinitrotetramethylcoumarin) 3) A. Clayton, JChemSoc 97, 1399 (1910)

Dimethylcumene and Derivatives
3,5-Dimethylcumene or Dimethylpropylbenzene,
(H₃C)₂C₆H₃·CH₂·C₂H₅, mw 148.24; liq; bp 206-210°; prep’d with other products by
mixing 4 vols acetone, 2 vols methylpropylketone & 3 vols sulfuric acid (Ref 1)
2,4,6-Trinitro-3,5-dimethylcyclohexane,
\((H_2C)C_6(NO_2)_2CH_2C_6H_5\), mw 283.24, N 14.84%; crysts, mp 98.5–99°; was prep by nitrating the parent compd (Ref 3). Its expl props are not reported
Ref:s: 1) Beil 5, 440 (Dimethycumene)
2) Beil 5, not found (Trinitrodimethylcyclohexane)
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,

\[
\begin{align*}
H_2C & \quad \text{CO.CH}_2 \quad \text{C(CH}_3)_2 \\
\text{CO.CH}_2 & \quad \text{CO.CH}_2
\end{align*}
\]

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 chl, benz & eth acet; sl sol in eth; v sl sol in petr eth; can be prep 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,

\[
\begin{align*}
O_2NHC & \quad \text{CO.CH}_2 \quad \text{C(CH}_3)_2 \\
\text{CO.CH}_2 & \quad \text{CO.CH}_2
\end{align*}
\]

mw 185.18, N 7.56%; col pttts (from eth + petr eth), mp 102–105°; sl sol in cold w; v sl sol in alc, eth & benz; was prep by nitratign Dimedone with fuming nitric acid (d 1.5) while cooling with NaCl-ice mix, and by other methods (Refs 2 & 3). The product & NaCO_3 gave a Sodium salt, \((C_9H_{11}NO_4)Na\), yellow crysts; with Ag_2O a Silver salt, \((C_9H_{11}NO_4)Ag\), yellow; with PbO in alc a yel Lead salt, \((C_9H_{11}NO_4)Pb\); with a hot suspension of CuO a green Copper salt, \((C_8H_{10}NO_4)Cu\); similarly a Cobalt salt, \((C_8H_{10}NO_4)Co\); and refluxing with FeS in alc gave violet Iron salt, \((C_8H_{10}NO_4)Fe\) which on heating decomposed with a flame, characteristic of all the above salts, Silver being the least stable (Ref 3)
Ref:s: 1) Beil 7, 559, (313), [531] & [3225]
2) Beil 7, 563
4) CondChemDict (1961), 390

3,8-Dimethyl-4,6-decadiyn-3,8-dihydroperoxide.
See under Diacetelyenic Dihydroperoxides in this Vol, p D1120-R

Dimethylallylsilane,

\[
H_2C:CH.CH_2\quad \text{Si} \quad \text{CH}_2.CH:CH_2\quad \text{CH}_3
\]

mw 140.27; liq, bp 124–25° at 760mm, a
D 1.4411 at 20°; was prep by Schaffner from allylchloridedimethyl dichlorosilane 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-nitrites. 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

Ref:s: 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 No 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
**Dimethylaminoethane.** Same as Dimethyl- 
ethylenediamine

**Dimethylaminopropane.** Same as Dimethyl- 
propylenediamine

**Dimethyl-diaminotartaric Acid and Derivatives**

*N,N'-Dimethyl-diaminotartaric Acid or N,N'-Dimethyl-succinamide,*

\[ \text{H}_3\text{C}.\text{HN.CO.CH}_2\text{.CH}_2\text{.CO.NH.CH}_3 \]; mw 144.17, N 19.43%; crystals (from acet & eth), 
mp 174–75°; readily sol in alc & w; s1 sol in acet & warm eth acet; v s1 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'-dimethyl-
tartardiamide Dinitrate,*

\( (\text{H}_3\text{C})\text{(NO}_2\text{)CN.CO.CH(ONO}_2\text{).CH(ONO}_2\text{).CO.-}) \)

\( -\text{N(NO}_2\text{)}\text{(CH}_3 \); mw 356.17, N 23.60%, OB to CO\(_2\) 
minus 18%; crystals, (from CCl\(_4\)), mp 114°, bp 
exp 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'-dimethyl- 
diaminotartaric acid in acetone at -5 to 0°, 
adding slowly conc 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)

4, [169] (Nitratned) 3) P. Frankland & A. 
Slator, JCS 83, 1360(1903) 4) T. Urbański, 
Roznik:Chem 16, 334(1936) & CA 31, 1770 
(1937)

**4,4'-Dimethyl-3,3'-diazooaminofurazan,**

\[ \text{H}_3\text{C} \cdot \text{C} \longrightarrow \text{C} \longrightarrow \text{N:N.NH} \longrightarrow \text{C} \longrightarrow \text{C.CH}_3 \]; 
\( \text{mw 269.17, N 46.88%; hydrated ppt lts from dil alc), dec in light, mp} \)

\( 114° \) & decomp at higher temp; anhyd crystals 
sol in cold alc, eth, acet, hot petr eth, chlf 
& benz; forms a Silver salt, AgC\(_{4}\)H\(_{4}\)N\(_{2}\)O\(_{2}\), 
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(ter-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**

**[1,1'-Dimethyl-1,1'-dibutyl-tetrazene(2)]**

\( \text{H}_3\text{C} \longrightarrow \text{N:N:N} \longrightarrow \text{CH}_3 
\text{H}_3\text{C}_4 \text{N} \longrightarrow \text{C}_4 \text{H}_9 
\text{N} \longrightarrow \text{NH}_2 \); 
\( \text{mw 200.32, N 27.97%, oily liq, sp gr 0.8798} 
\text{at 15°, fr p – did not solidify at minus 20°; } 
bp 119–20° at 18mm pressure; deflagrates 
on rapid heating; very s1 sol in w; was prepd by 
Franchimont & van Erp by treating cooled 
etheral soln of N-methyl-N-butyl hydrazine, 
(\text{H}_3\text{C})\text{C}_4\text{H}_9\cdot\text{N.NH}_2\text{, with yel mercuric oxide} 
Refts: 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-butylenylene-dihydro-
peroxide in Vol 1, p A66-R under Acetylene 
Hydroperoxides or Peroxides

**3,6-Dimethyl-1,2-dihydro-1,2,4,5-tetrazine,**

\( \text{H}_3\text{C} \cdot \text{C} \longrightarrow \text{NH} \longrightarrow \text{NH} 
\text{N} \longrightarrow \text{N} \longrightarrow \text{C.CH}_3 
\text{mw 112.14, N 49.97%; cryst + H}_2\text{O, mp –} 
deliq at 100°; readily sol in water; insol in 
eth; prepd by heating acetoinitrile with anhyd 
hydrazine (Refts 1 & 2). No expl props are 
reported

*NOTE: No Azido, C\(_{4}\)H\(_{7}\)N\(_{3}\), Mononitro, 
C\(_{4}\)H\(_{4}\)N\(_{2}\)O\(_{2}\), or Dinitro, C\(_{4}\)H\(_{6}\)N\(_{2}\)O\(_{4}\), derivs 
were found in the literature thru 1966

*Refts: 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.

Dimethylketopiperazine and Derivatives
3,6-Dimethyl-2,5-diketopiperazine or 3,6-Dimethyl-2,5-dioxopiperazine,

\[
\begin{align*}
\text{HN} & \quad \text{CO} \cdot \text{CH(CH}_3)_2 \cdot \text{CO} \\
\text{CH(CH}_3)_2 \text{NH} & \quad \text{CH(CH}_3)_2 \text{NH}
\end{align*}
\]

mw 142.16, N 19.71%; crysts, mp 280°; was first prep'd by Franchimont & Friedmann (Ref 3). Shibata & Asahina prep'd it (Ref 4) by heating 132g of dl-alanine(2-aminopropionic 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,

\[
\begin{align*}
\text{(O}_2\text{N})_2 \text{N} & \quad \text{CO} \cdot \text{CH(CH}_3)_2 \cdot \text{CO} \\
\text{CH(CH}_3)_2 \text{N(NO}_2)_2 & \quad \text{CH(CH}_3)_2 \text{N(NO}_2)_2
\end{align*}
\]

mw 232.16, N 24.14%, OB to CO\textsubscript{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 prep'd by Franchimont & Friedmann (Ref 1 & 2). Shibata & Asahina prep'd 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 exptl comparable in sensitivity to impact to TNT; it is very hygroscopic and hydrolyzable 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).


Dimethyl-dimethylene-triamine and Derivatives
1,5-Dimethyl-2,4-dimethylene-1,3,5-triamine or 2,4,6-Triazaheptane,

\[
\begin{align*}
\text{H}_2\text{C}.\text{NH} \cdot \text{CH}_2 \cdot \text{NH} \cdot \text{CH}_2 \cdot \text{NH} \cdot \text{CH}_3 & \quad \text{mw 103.17, N 40.73%; may be considered the parent compd of its trinitro deriv, although not used to prep it:}
\end{align*}
\]

1,5-Dimethyl-2,4-dimethylene-1,3,5-trinitramine or 2,4,6-Trinitro-2,4,6-triazahexane,

\[
\begin{align*}
\text{CH}_3 \cdot \text{N(NO}_2)_2 \cdot \text{CH}_2 \cdot \text{N(NO}_2)_2 \cdot \text{CH}_2 \cdot \text{N(NO}_2)_2 \cdot \text{CH}_3 & \quad \text{mw 262.19, N 32.06%; crysts (from alc), mp 169.5-170°; was prep'd by adding (O}_2\text{NN}-\text{CH}_2\text{CH}_2)_2\text{NH.HCl at 0° to HNO}_3, allowing the temp to rise to 20° and pouring the mixt into ice-water (Refs 2 & 3). No expl props are reported.}
\end{align*}
\]


1,1-Dimethyl-2,2-dimethyl-ethane-1-hydroperoxide-nitrobenzoate,

\[
\begin{align*}
\text{(H}_2\text{C})_2\text{C} \cdot \text{CH(CH}_3)_2 \\
\text{OO} \quad \text{C} \cdot \text{C} \cdot \text{CH} = \text{C} \cdot \text{NO}_2 \quad \text{O}
\end{align*}
\]

mw 267.27, N 5.24%; colorless plts, mp 57-58°, exploding in a flame; the hydroperoxide is prep'd by treating hydrogen peroxide at 0° with small amt's of sulfuric acid and the mixt treated cautiously with dimethyl-isopropyl carbinol and allowed to stand overnight in a refrigerator. It was converted to the nitrobenzoate by interaction of the hydro-
peroxide with 4-O₂N.C₆H₄.COCl, 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(methylidinitrophenyl)-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-tetraza-cyclooctane.** See under Dimethyl-tetraza-cyclooctane and Derivatives

**Dimethylidiphenylamine and Derivatives**

3,3' Dimethylidiphenylamine or Bis(3-methylphenyl)amine, H₃C NH-CH₈

mw 197.27, N 7.10%; liq, fr p 12°. Prep and other props are in Ref 1

4,4' Dimethyl-diphenylamine or Ditolyamine, H₃C NH-CH₈

wh crysts, mp 79°; v sol in cold acet & benz and in hot alc & chl; darkens easily on exposure to light; can be prep as described in Beil (Ref 2) or by the method of Tavemier & 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 compounds seems to be explosive

**3,3'-Dimethyl-2,4,6,2',4',6'-hexanitro-diphenylamine,** H₃C NO₂ NO₂ CH₈

mw 467.27, N 20.98%; bm tablets (from alc), mp 60°. Can be prep by warming 4,6-dinitro-3,3'-dimethylidiphenylamine 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 prep by treating with fuming nitric acid, either p,p-ditolyamine or nitroso-p,p-ditolyamine. 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. Lehue, Ber 13, 1545 (1890) (Hexanitro-4,4'-dimethyl-diphenylamine) 6) L. Brady et al, JCS 125, 2404 (1924) (Hexanitro-3,3'-dimethylidiphenylamine) 7) P. Tavemier & M. Lamouroux, MP 38, 75 & 84(1956) (4,4'-Dimethylidiphenylamine)

**Dimethylidiphenylamine and Derivatives**

N,N'-Dimethyl-3,3'-diphenylidiamine or 3,3'-Dimethylamino-diphenyl

(H₃CCHN NH(CH₃))

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'-dimethylamino-diphenyl,**

(O₃N)₂C₆H₄(NH.CH₃)-C₆H₄(NH.CH₃)(NO₂)₆;

mw 392.28, N 21.43%; yel powd, mp - explodes when heated above 350°; tums reddish-bm when exposed to light; was obtd when tetranitro-3,3-dichloro-phenyl was heated with methylamine & alc for 5 hrs at 100° (Ref 3)

**N,N',4,4',6,6'-Hexanitro-N,N'-dimethyl-3,3'-diphenylidiamine or 4,4',6,6'-Tetranitro-3,3'-dimethylamino-diphenyl,**

(O₃N)₂C₆H₄(N(NO₂)₂CH₃)-C₆H₄(N(NO₂)₂CH₃)(NO₂)₆; mw 482.28, N 23.24%; yel powd, mp 210° (dec) & explodes at 225°; sol only in acet; was prep from the tetranitro deriv by dissolving in abs nitric acid at -10° & adding cold water (Ref 3)

Refs: 1) Beil - not found (Dimethylidiphenylidiamine) 2) Beil - not found (Nitrated derivs) 3) J. van Alphen, Rec 51, 364 (1932) & CA 26, 2447 (1932)
Dimethylidiphenylether and Derivatives

Dimethylidiphenylether or Dimethylphenyl

oxide, \((\text{C}_8\text{H}_7\text{C}_6\text{H}_4\text{O})_2\text{C}_6\text{H}_4\text{(CH}_3)\)\), mw 194.22.

Three derivs are known:

1. Dimethylidiphenylether, liq, bp 274°, d 1.047 at 24.3° (Ref 1)
2. Dimethylidiphenylether, liq, bp 284°, d 1.032 at 21° (Ref 2)
3. Dimethylidiphenylether, prisms (from MeOH), mp 50°, bp 285° (Ref 3)

Other props & methods of prepn are given in the Refs

Tetranitrodiphenylether,
\((\text{C}_8\text{H}_7\text{C}_6\text{H}_4(NO)\text{2})_2\text{O}\)\), mw 378.25, N 14.81%. Five isomers are known:

1. Tetranitro-2,2'-dimethylidiphenylether, powd, mp 115°; was prepd by

nitrating the 2,2'-dimethyl deriv with conc nitric and sulfuric acids (Ref 4, p180)

2. Tetranitro-2,6'-dimethylidiphenylether, hexag plts, mp 175°; sol in hot alc;

obtd with other isomers on nitrating the 3,3-dimethyl deriv with conc sulfuric acid

& fuming nitric acid in the cold (Ref 4, p194)

3. Tetranitro-3,3'-dimethylidiphenylether, wh powd, mp 203°; insol in hot alc;

prep'd by nitrating the 3,3'-dimethyl deriv with mixed acid in the cold (Ref 4, p194)

4. Tetranitro-4,4'-dimethylidiphenylether, tablets (from alc or glc acid ac),

mp 240°; sl sol in acet, v sl sol in eth, chlf & benz; insol in cold glc acid ac &

petr eth; obtd with other isomers on nitrating the 4,4'-dimethyl deriv with mixed acid at

10° (Ref 1, p392 & Ref 6)

5. Tetranitro-4,4'-dimethylidiphenylether, yell ndls, mp 84°; prep'd by warming the

4,4'-dimethyl deriv with fuming nitric acid in glc acid ac (Ref 4, p207 & Ref 5)

Their expl props were not dnd

Refs: 1) Beil 6, 353, (171) & [1250]
2) Beil 6, 377, (186), [352] & [1103]
3) Beil 6, 394, (200), [377] & [1360]
4) Beil 6, (190, 194, 207) & [392]
6) J. Reilly et al, JChemSoc 1927, 71

N,N'-Dimethyl-N,N'-diphenyl-urea. See

Centralite 2 in Vol 2 of Encycl, p C137-L and the following addnl refs: A. Apati,
MP 22, 180 (1926); H. Nomm, "Die Lösungsmittel und Weichmachungsmittel", Wissensch.
Verlags, Stuttgart (1946); CondChemDict (1961), 395-L

N,N-Dimethyl-diphenyllylamine. See N,N-

Dimethyl-aminophenixlyl in this Vol

Dimethyleneperoxidecarbamide (Dimethylene-

peroxyd-carbamide, in Ger) (Was formerly called

Tetrathoxenyl-diperoxoaryl-dicarbamide) (On p

[48] of Vol 3, Beilstein called it 4-Amino-

formyl-1,2,4-dioxozol),

\[\begin{array}{c}
\text{H}_2\text{N},\text{CO,N} \rightleftharpoons \text{CH}_2\text{O}
\end{array}\]

mw (118.09)_x, N 23.72%; crstls, mp — explodes on heating; insol in w, ethanol, methanol, chlf, pyridin and other org solvents;
dissolves in conc nitric or sulfuric acids and may be prpd from these solns by w. Was

prep'd (Reps 1 & 2) by dissolving with cooling urea (1 mole), formaldehyde (2 moles)
and 30% hydrogen peroxide in conc nitric acid (See also Ref 3)

Reps: 1) Beil 3, (27), [48] & [112]
2) C. von Gisewald & H. Siegens, Ber 47, 2464-66 (1914)
3) Ibid, Ber 54, 493 (1921)

Dimethyleneperoxide-ethylamine (Dimethylene-

peroxyd-athylamin, in Ger),

\[\begin{array}{c}
\text{C}_2\text{H}_5\text{N} \rightleftharpoons \text{CH}_2\text{O}
\end{array}\]

mw 103.2, 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

prep'd by the action of hydrogen peroxide on formaldehyde in the presence of ethylamine

Reps: 1) Beil 27, [523]
2) F.C. von Gisewald & H. Siegens, Ber 54, 492 &

497 (1921); CA 15, 2417 (1921)
3) J.F.

Walker, "Formaldehyde", Reinhold, NY (1953)
Dimethylether (Methylether or Methyloxide), CH₃.O.CH₃, mw 46.07; col gas; flammable and explosive, fr p 141.50°, bp 24.82°.
Other props & prep in Refs

Di(methylether)-peroxide or Bis(methylmethyl)-peroxide,
H₃C.O.CH₂—O—O—CH₂.O.CH₃, mw 74.12, O 21.59%; crystls; 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)

Dimethyl-ethyl-acetate and Derivatives
1,1-Dimethyl-ethylacetate,
H₃C.C(CH₃)₂COOCH₃, mw 116.16; may be considered as the parent compd of its tri-nitro deriv although not used to prep ir
1,1-Dimethyl-2,2,2-trinitroethylacetate,
(O₂N)₃C.C(CH₃)₂.COOC₃H, 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-ethylacetate) 2) Beil, not found (Dimethyl-trinitroethylacetate) 3) J.C. Conly et al, “USRubberCo Quarterly Report No 28”, Aug 1–Nov 1, 1954, p 10(below)

Dimethylethlyenediamine and Derivatives
N,N¹-Dimethylethlyenediamine; 2,5-Diazahexane or N,N¹-Dimethyl-N,N¹-diaminoethane, (H₃C)CH₂.CH₂.NH(CH₃), mw 88.15, N 31.78%. Prepn and props are given in Ref 1
N,N¹-Dimethyl-N,N¹-dinitroso-ethylenediamine or 2,5-Dinitroso-2,5-diazahexane,
(H₃C)ON.CH₂.CH₂.N(NO)(CH₃), mw 148.17, N 37.82%; It yel-gey plits from alc + eth, mp 56–61°; soln in alc; sl sol in eth; was prepd by reaction of NaN₂O₃ on a HCl soln of the parent compd (Ref 2). Its expl props are not reported
N,N¹-Dimethyl-N,N¹-dinitroethylenediamine; Me₂EDNA or N,N¹-Dimethyl-ethylenedinitramine; 2,5-Dinitro-2,5-diazahexane; N,N¹-Dimethyl-N¹-dinitroso-ethane or N,N¹-Bis(methyl)-ethylenedinitramine,
(O₂N)₂CH₂.CH₂.N(O₂N);
CH₃     CH₃
mw 178.15, N 31.46%; O Bal to CO₂ minus 80.6%; crystls mp 137.5–138°; does not expld 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 Bruceon 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)
Me₂EDNA was first prepd by Franchimont & Klobbie (Ref 4) by alklylation of ethylenedinitramine, (O₂N)₂CN.CH₂.CH₂.NHO₂N), with methylidiole, CH₃I, and also with methylsulfate, (CH₃)₂SO₄. Its structure was not definitely established until 1912 by Backer (Ref 5)
Blomquist & Fiedorek reported its prep in mixture with MeEDNA (Methylethlyenedinitramine) by treating EDNA (Ethylenedinitramine) with methylbromide, CH₃Br (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) Me₂EDNA 12, RDX 50, R-Salz 36 & unaccounted 1%
b) Me₂EDNA 2.5, RDX 96.5 & DPhA (Diphenylamine) 1% (See also Ref 11).

Note: R-Salz is Cyclotrimethylene trinitramine, which is described in Vol 3 of Encycl, p C630-R. RDX is described as Cyclotrimethylene trinitramine on pp C626ff.

Ultraviolet spectrum of Me₂EDNA was determined in Canada (Ref 8).


 asym-Dimethylacrylamide. See iso-Butanediol in Vol 2 of Encycl, p B370-L.

 sym-Dimethylacrylamide. See Butanediol in Vol 2, p B368-R.

### Dimethyl-9-fluorenone and Derivatives

1,3-Dimethylfluorenone or Diphenyleketteone, [H₃C]

mw 208.25; crysts (from MeOH), mp 112°; forms an oxime, mp 197-98°. Prep: Nitosylantranilyl chloride with m-xylene & AlC₃ gives 2,4-methyl-2-tosylaminobenzophenone which is saponified by sulfuric acid & the product is diazotized with NaN₂O₃ (Ref 3)

2,4,7-Trinitro-1,3-dimethyl-9-fluorenone, [NO₂]

mw 343.25, N 12.24%; melts (from benz or acet), mp 236°. Preparation: nitration of parent compd with KNO₃ in H₂SO₄ gives a Dinitro deriv, mp 238°; nitration of parent compd with nitric and sulfuric acids gives the Trinitro deriv. Heating the Trinitro deriv with N₂H₄ 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. Wüml, Helv 33, 1388 (1950) & CA 45, 1550 (1951)

### NN-Dimethylformamide (DMF) (Formyldimethylamine), H(C=O).N(CH₃)₂; mw 73.09, N 19.17%, OB to CO₂ minus 120.4%; colorless liquid; sp gr 0.9484 at 22.4°/4°C (duPont's 0.9683 at 0°/4°C and 0.9445 at 25°/4°C), nD 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 vaporzation 210 Brtt/lb, heat of combustn 457.5 kcal/mole (In Ref 4 Q̇ is given as 464.8 and Q̇₂ 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 re-commended 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 decomposed 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 comds, 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 propyl and expl mixtures, such as Comp C-2.


**Dimethyl-3-furoyl Azide, C₇H₅N₂O₂; mw 165.15, N 25.45%. Two isomers are known: 2,4-Dimethyl-3-furoyl Azide, C₄H₃NO₂

N(CH₃)₂

H₄C.CO.CH₂.O.C(CH₃)₂.C-CON₃

oil, n o data given; prep'd when the hydrazide in aq soln was cooled to 0–5°, NaNO₂ & excess acetic acid were added and the azide separated after 1 hr at 5–10°(Ref 2) 2,5-Dimethyl-3-furoyl Azide, CH₂(C₂H₅)₂.O.C(CH₃)₂.C-CON₃; fr p 24–25°; prep'd from the hydrazide by treating it with NaN₃ in dl acq 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 Dimethyl-ethylcarbamate,

N(CH₃)₂

CO

OC₂H₅

mw 117.15, N 11.96%; liq, bp 147°, d 0.9725 at 15°; prep'd by various methods; decomp in warm concd sulfuric acid into carbon dioxide & dimethylamine (Ref 1)

**N,N-Dimethylglycolurethane Nitrate,**

N(CH₃)₂

O.CH₂.CH₂.ONO₂

mw 178.15, N 15.73%; liq; bp 110° at 3mm; starts to decomp ca 140°. It was prep'd 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) & 1441 (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-Dimethylacetylene diurea,

NH-C(CH₃)₂-NH

OC

NH-C(CH₃)₂-NH

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., chl and benz. It can be prep'd 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-dimethylacetylene diurea,

NH-C(CH₃)₂-N(NO₂)

OC

NH-C(CH₃)₂-N(NO₂)

mw 260.17, N 32.31%; OB to CO₂ 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°C); 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 prep 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°C; 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

Shiner (Ref 5) prep'd it in 80–87% yield by nitrating DMGU with concd nitric acid in presence of acetic anhydride, while cooling the mixture to 5°C.

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°C — unsatisfactory. Attempts by Shiner to nitrate it to higher NO₂ content were unsuccessful (Refs 5 & 6).


Dimethylglyoxime and Derivatives

**Dimethylglyoxime or Butane Dioxide,**

H₃C,C.:C(=NOH).C.:C(=NOH).CH₃; mw 116.12, N 24.13%; col crystals, mp 240–42°C; very sl sol in w; sol in alc & eth. It can be prep'd by reacting methylketoxime with diazacyclonexime in dil sulfuric acid at 40°C (Ref 1). It is used in analytical chemistry, especially as a reagent for Ni.

A small yield (ca 10%) of Tetranitro-

butane could be obt'd on treating it with fuming nitric acid.

**Dimethylglyoxime Diacetate,**

H₅C.C.:C(=NOOC.CH₃).C.:C(=NOOC.CH₃).CH₃; mw 200.19, N 13.99%; no props reported, except hydrolysis rate; was prep'd by Milone (Ref 5) from dimethylglyoxime, Ac₂O & AcONa (Refs 5 & 6).

Treating it with fuming nitric acid gave a small amt of Tetranitrobutane.

**Dimethylglyoxime-bis(O-methylether),**

H₃C.C.:C(NO.CH₃).C.:C(NO.O.CH₃).CH₃; mw 144.17, N 19.43%; crystals (from alc), plts (by condensation), mp 41–44°C, bp 158.5°C at 780mm press; obt'd by reacting diacetyl-dioxime with dimethylsulfate in NaOH, 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.


**Dimethylguanidine and Derivatives**

**N,N-Dimethylguanidine,**

(H₃C)₂N.C.:NH₂, mw 87.13, N 48.23%; ndls (from alc + eth), mp -- produces gas at 144°C & melts at 172°C; forms many salts which decom on heating (Ref 1).

**N,N-Dimethyl-N¹-nitroguanidine,**

(H₃C)₂N.C.:C(=NH).NH(NO₂); mw 132.13, N 42.41%; ndls, mp 193.6–94.5°C; sol in alc; sl sol in cold w & eth; decom on heating in hot w; prep'd by reacting Nitroguanidine with a 20% soln of dimethylamine at 75–80°C (Refs 2, 3 & 4). No expl props reported.

**Dimethylguananylpyrazole and Derivatives**

3,5-Dimethylguananylpyrazole,

\[
\begin{align*}
\text{H}_2\text{C} & \quad \text{C} \quad \text{CH}_3 \\
\text{N} & \quad \text{N} \\
\text{C} & \quad \text{(NH)}(\text{NH}_2) \\
\text{C} & \quad \text{(NH)}(\text{HN}O_2)
\end{align*}
\]

mw 138.17, N 40.55%; known as the Nitrate salt, \(\text{C}_6\text{H}_1\text{O}_4\text{N}_4 + \text{HNO}_3\), crystals (from w) or ndls (from alc), mp 168°(dec); prep & other props in Ref 1.

3,5-Dimethyl-4-nitro-1-nitroguananylpyrazole,

\[
\begin{align*}
\text{O}_2\text{N}:\text{C} & \quad \text{C} \quad \text{CH}_3 \\
\text{N} & \quad \text{N} \\
\text{C} & \quad \text{(NH)}(\text{HN}O_2)
\end{align*}
\]

mw 228.17, N 36.84%; crystals, mp 126–129°; sol in aq alc; can be prepd by nitrating the 3,5-dimethyl-1-nitroguananylpyrazole 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;

\[
\begin{align*}
\text{H}_2\text{C} & \quad \text{(CH}_3\text{)}\text{CH}_2\text{CH}_2\text{C(}\text{CH}_3\text{)}\text{CH}_2 \\
\text{N} & \quad \text{N} \\
\text{O}_3 & \quad \text{O}_3
\end{align*}
\]

mw 110.19; col flammable liq; bp 114.3°; sp gr 0.740–0.760 at 25/25°; n\(_D^\circ\) 1.426–1.429 at 25°; 90% distn betw 114 & 123°; sol in hydrocarbons, insol in w. Prepn is given in Refs 1, 4 & 5.

1,5-Dimethylhexadiene Dioxonide,

\[
\begin{align*}
\text{O}_3 & \quad \text{O}_3 \\
\text{H}_2\text{C} & \quad (\text{CH}_3\text{)}\text{CH}_2\text{C(}\text{CH}_3\text{)}\text{CH}_2
\end{align*}
\]

mw 206.19, O 46.56%; yel viscous syrup, mp – very expl; decom 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) (? H. Harris & (?) Türk.


1,12-Dimethyl-2,3,5,6,10,11-hexamethylene-7-oxa-8-nitroso-9-nitro-1,4,12-trinitrimine; iso-N\(_2\),N\(_4\)-Dimethyl-triethylene-tetranitramine (Ref 2); or 2,10,13-Trinitro-7-oxa-2,5,6,10,13-pentaoxatetradec-5-ene-6-oxide (CA nomenclature),

\[
\begin{align*}
\text{O} & \quad \text{NO}_2 \\
\text{O} & \quad \text{N} \quad \text{H}_2\text{CH}_2\quad \text{H}_2\text{CH} _2 \quad \text{N} \quad \text{CH}_3 \\
\text{H}_2\text{CH}_2\quad \text{H}_2\text{CH} _2 \quad \text{N} \quad \text{CH}_3 \\
\text{NO}_2 & \quad \text{NO}_2
\end{align*}
\]

mw 354.28, N 31.63%, crystals, mp 88.5–90°; pH 6.2, expln temp 325° (ignited), at 135° – explodn in 10 mins; power 109.1% of TNT, sensitivity to impact >90 cm (TNT = 48–50 cm). 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-ethylendinitramine was alkylated, using ethylene dibromide (Ref 2). Its UV spectrum was detd by Jones & Thorn (Ref 3).

Refs:

NOTE: See following item.

1,10-Dimethyl-2,3,5,6,8,9-hexamethylene-1,4,7,10-tetranitrimine or \(\text{N}^1\cdot\text{N}^4\)-Dimethyl-triethylene-tetranitramine (Ref 2),

\[
\begin{align*}
\text{NO}_2 & \quad \text{NO}_2 \\
\text{CH}_2 & \quad \text{N} \quad \text{CH}_2\quad \text{H}_2 \quad \text{CH} _2 \quad \text{N} \quad \text{CH}_3 \\
\text{CH}_2 & \quad \text{N} \quad \text{CH}_2\quad \text{H}_2 \quad \text{CH} _2 \quad \text{N} \quad \text{CH}_3 \\
\text{NO}_2 & \quad \text{NO}_2
\end{align*}
\]

mw 354.28, N 31.63%, crystals, 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 >90 cm (TNT = 48–50 cm). It was known in Germany under code name BMT-90, one of
the by-products in the manuf of RDX. Blomquist (Ref 2) obd as it as one of two isomers when N-methyl-ethylenedinitramine was alkylated, using ethylene dibromide (Ref 2).

Re/s: 1) Beil, not found 2) A.T. Blomquist, OSRD 4134 (1944), pp 8 & 68

NOTE: See above item

Dimethylhexane and Derivatives

2,5-Dimethylhexane or Diisobutyl, (H₂C)₂CH₂CH₂CH₂CH₂-C(OOH)₂; mw 114.22; col liq, sq pr 0.694 at 20/4°, ft p minus 91.2, bp 109.1°. Prep & other props are in Ref 1

2,5-Dimethylhexane-2,5-dihydroperoxide, (H₂C)₂OC(OOH)₂-C(OOH)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 prep from [(H₂C)₂C(OH)CH₂]₂ & 75% hydrogen peroxide is given in Ref 4

2,5-Dinitro-2,5-dimethylhexane, (H₂C)₂CH₂CH₂(NO₂)₂CH₂-C(Ch₃)₂; mw 204.22, N 13.72%; crysts (from benz), mp 124-25°; sol in benz, alc & hot glacial ac acid; sl sol in petr eth & eth; insol in alkalies; was prep by treating tert-Nitroisobutyl with nitric acid (d 1.075), or by other methods (Ref 3)

x,x,x-Trinitro-2,5-dimethylhexane, C₆H₉(NO₂)₂; mw 249.22, N 16.86%; large colorless plates (from benz or petr eth), mp 91°; prep by nitrating isobutyl with fuming nitric acid (Ref 3). No expl props are reported. This compd was not found described in later Refs thru 1966

Re/s: 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-hexaoxadi-spiro-[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(Ch₃)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 & 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 aceton, K cyanate & hydrocyanic acid. Thermochem props (Ref 5). Gives on nitratation mono- and dinitro compds

1-Nitro-5,5-dimethylhydantoin or Nitro-acetonylurea, OC.NH.CO.N(NO₂)₂.C(Ch₃)₂; mw 173.13, N 24.27; col ndls; mp 139-41°; bp (dec at 155° with eoivn of gas); v sl sol in w & in benz; better sol in alc & chl; 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(NO₂)₂.CNO(NO₂)₂.C(Ch₃)₂; mw 218.13, N 25.69%, OB to CO₂ minus 51.3%; col plates, mp 98.5-99.5°, bp (puffs off and catches fire, but does not expl even at 360°). Laboratory prep, 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 chl/pet eth

It is an expl comparable in sensitivity with TNT; heat of combustion 604.2 kcal/mole, heat of formation 73.1 kcal/mole; thermal stability at 75° and 100°—unsatisfactory; not satisfactory from the point of view of hygroscopicity (Refs 3 & 4)


**Dimethylhydrazine and Derivatives**

**uns-Dimethylhydrazine (UDMH);** (1,1-Dimethylhydrazine), code name Dimazine, (H₃C)₂N.NH₂; 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 prep 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) prep'd it by catalytic reduction of dimethylnitrosamine, (CH₃)₂N.NH.NO

Eutectic mixtures of UDMH and of methylhydrazine, CH₃NH.NH₂ (col hygroscopic liq, described in Beil 4, 546) for use in proplts are listed in Ref 15

Dimazine has been used in jet & rocket proplts; as stabilizer for org peroxide fuel additives and for other purposes (Refs 9, 15 & 16)

Acording 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 proplts (based on UDMH, high energy liquid fuel), will give the Titan near instant readiness. Both the proplnt combi-

nation and the rocket engine designed to use the proplnts are being developed by Aerojet under Air Force contract (Ref 14)

Explosibility of mixt of UDMH with diethylenetriamine, (NH₂.C₂H₅)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** — 10 mg per liter, maximum

A sample of proplnt shall consist of not less than 600 ml. 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 500 ml graduate. Use this 500 ml of proplnt for particulate analysis in order to test for contamination. Use ASTM Designa-

tion D-2276-65T, Method A, with the following exceptions. The filter disc should be solvent resistant made from such materials as Millipore LSWP 04700 (Millitex-Teflon), Millipore URWP 04700 (Solvinitert), or Geiman VF-6 (Fluoride-Metricel), plain, white 10½ microns, 47 mm 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 dein of UDMH in mixtures with other substances are as follows:

Duigou (Ref 12) describes two micro-colorimetric 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 macro-colorimetric 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% “thiôdene” 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 thiôdene, but simply says that it is an iodo-metric 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 Erlen, with slight stirring, from a burette contg N/10 K bromate soln (prep by dissolving 2.78g KBrO₃ and 10g KBr in 1 liter of distilled w) until the light yellow coloration starts to be persistent. Note the number of ml added (say x) and then add quickly 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 lose 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.

\[
\% \text{UDMH} = \frac{60x(N-a)x1000x1.00}{8x10000x10p} = 7.5(N-a) \\
p
\]

where: 
N = total number of ml of K bromate used
a = number of ml of thiosulfate
p = grams of UDMH sulfate 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 (prep 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 ml vs emf to locate the point of greatest inflection. The temp through the titration should be maintained in the range –5 to +10°C, and the titration should be completed within a total time of 5 min. At the end point the soln will be yellow and the steep potential increase will be in the range 0.67 to 0.70 volts:

\[
\text{UMDH, } \% = \frac{12.02AM}{W}
\]

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 2.0mm 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°C 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.
breathe the thermometer by taking the mp of pure chl (−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 calc'd from the 90-ml distillation temp as follows:

$$\text{Wt} \% \text{H}_2\text{O} = 0.14[(\text{corrected 90-ml temp in } ^{\circ}\text{F}) - (146 ^{\circ}\text{F})]$$

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-Dimethylhydrazone, N,N'-Dimethylhydrazine or Hydrazo-methane), H₃C.NH.NH.CH₃, mw 60.10, N 46.62%, hygroscopic liq, sp gr 0.827 at 20/4°, fr p – not listed; bp 81° at 747 mm; 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,
H₉C.NH.N(NO)=CH₃; 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, decomgradually 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 nitrile in ac ac soln (Refs 2, p(561) & Ref 3). See also Ref 4


1,1-Dimethyl-5,5'-hydrazo-1H-tetrazole or 5,5'-Hydrazobis[1-methyl-1,2,3,4-tetrazole]

N-N(CH₃)=C-NH–NH–C-N(CH₃)=N H₂O

N—N — N

mw 196.19, N 71.40%; crystls, mp – decom at 158° with evolution of gas; was obtd by reduction of 1,1-Dimethyl-5,5'-azotetrazole, om-yel crystls, mp 182°(dec). Its expl props were not investigated

Dimethylhydorgenphosphate, \((\text{CH}_3\text{O})_2\text{PHO}\);
mw 110.06; col liq with mild but toxic odor;
sp gr 1.200 at 20°; bpt 72–73° at 25mm;
flash p 205°F; nD 1.400 at 25°; sol in w
and miscible with many org solvents. It
is hazardous to handle and personnel sampl-
ing or testing this material should be ade-
quately 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

Dimethylhydorgenphosphate is intended
for use as an intermediate in chemical agent
production and also as an adhesive & lubri-
cant additive. US Armed Forces require-
ments are listed in Ref 2

Refs: 1) Beil 1, 285, (142) & [1203]
2) Spec MIL-D-10396(G)(1969)  3) CondChem-
Dict (1961), 398-L

1,4-Di(methyleneimino)-p-quinone. See Benzo-
quinone-1,4-di(methyleneimine) in Vol 2, pB83-R

Dimethyliminotetrazoles, such as 1,3-Dim-
eethyl-5-iminotetrazole and 1,4-Dimethyl-
5-iminotetrazole are described under Di-
alkyliminotetrazoles in this Vol

Dimethylindazole and Derivatives

Dimethylindazole or Dimethylbenzopyrazole,
\(\text{C}_9\text{H}_8\text{N}_2\text{O}_2\), mw 146.19, N 19.16%.
Four isomers
are described in Beil (Ref 1)

4(or 6)-Azido-5,7-dimethylindazole,

\[
\text{H}_3\text{C} \begin{array}{c}
\text{CH} \\
\text{N or N}
\end{array} \text{CH}_3
\]

mw 187.20, N 37.41%; yel ndls (from benz),
mp 134–37°; sol in alc & benz; insol in
petr eth; was prep'd by diazotizing 2,4-
diaminomesitylene with nitroyl sulfonic
acid in concd sulfuric acid and treating
the diazonium salt with NaN_3 in cold water
(Refs 2 & 5)

Mononitrodimethylindazole, \(\text{C}_9\text{H}_8\text{N}_3\text{O}_2\),
mw 191.19, N 21.98%. Two isomers are
known: 4-Nitro-5,6-dimethylindazole,
plits (from petr eth + benz), mp 204°; and
7-Nitro-5,6-dimethylindazole, lt-yel ndls,
mp 180.5–181.5°. Prep & other props are
given in the Refs (Refs 3 & 4)

Dinitrodimethylindazole, \(\text{C}_9\text{H}_8\text{N}_4\text{O}_4\), mw
236.19, N 23.72%. Two isomers are known:
4,7-Dinitro-5,6-dimethylindazole,

\[
\begin{array}{c}
\text{H}_3\text{C} \\
\text{CH} \\
\text{N or N}
\end{array} \begin{array}{c}
\text{NO}_2 \\
\text{NO}_2
\end{array}
\]
greenish ndls (from anisole), mp 221–22°;
sol in boiling alc, anisole & w concg acetic
acid; sol in alkali is om; prep'd by dia-
zotizing 3,6-dinitro-2,4,5-trimethylaniline
in glc acet cl & sulfuric acid and treating
the diazonium salt with dil boiling sulfuric
acid soln (Refs 3 & 4)

4,6-Dinitro-5,7-dimethylindazole,

\[
\begin{array}{c}
\text{H}_3\text{C} \\
\text{O}_2\text{N} \\
\text{CH}_3
\end{array} \begin{array}{c}
\text{NH} \\
\text{N or N}
\end{array} \begin{array}{c}
\text{NO}_2 \\
\text{NO}_2
\end{array}
\]
yel ndls (from alc), mp 247°; prep'd by treat-
ing 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
glc ac et cl & concentrate 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, \((\text{CH}_3)_2\text{C}::\text{CO}\), mw 70.09;
yel liq, fr p minus 97.5°, bp 34° at 750mm; sol in eth; decomp by w or alc (Ref 1)

**Dimethylketene Peroxide**

$$\left[\text{CH}_3\text{C} = \text{CO}\right]_x\text{O} \cdot \text{O}$$

mw 102.09, O 47.02%; jell (from ethyl acetate) or wh powd (from ether), which when dry is exceedingly expl, detonates with great violence when touched, often even spontaneously; obt by autoxidation of dimethylketene at low temp (Refs 2 & 3)


**Dimethylmalonamide and Derivatives**

N,N'-Dimethylmalonamide, CH₂(CO.NH.CH₃)₂; mw 130.15, N 21.55%; ndls (from benz), mp 101°; sl sol in alc, chl, acet ac, acet & w; sol in benz; v sl sol in CCl₄ & CS₂; prepd by reaction of CO with methylamine in ether (Ref 1)

N-Nitro-N,N'-Dimethylmalonamide,

$$\text{CH}_2\text{CON(NO}_2\text{)CH}_3$$

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-nitrobarbituric 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',N'-Dinitro-N,N'-dimethylmalonamide,

CH₂[CO.N(NO₂)CH₃]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 chl, CS₂ & petr eth; was prepd by nitrating N,N'-dimethylmalonamide with nitric acid in the cold (Refs 3 & 4).

No expl props are reported


**Dimethylmethanediamine and Derivatives**

N,N'-Dimethyl-methanediame,

$$(\text{H}_3\text{C})\text{HN.CH}_2\text{NH(CH}_3\text{)};$$ 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',N'-Dinitro-N,N'-dimethylmethanediame or 2,4-Dinitro-2,4-diazopentane,

$$(\text{H}_3\text{C})\text{O}_2\text{N.CH}_2\text{N(NO}_2\text{)CH}_3;$$ mw 164.13, N 34.14%; crysts (from chl-hexane), mp 49-51°; was prepd by treating paraformaldehyde in 90% sulfuric acid at -2 to -5° portionwise with methylisotramine, 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)

**Dimethylmethyleneaminoquguanidine and Derivatives**

Dimethylmethyleneaminoquguianidine or Isopropylideneminoquinuamine,

H₂N.C(:NH).NH.N.C(CH₃)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'-Nitrodimethylmethyleneaminoquinuainidine,

$$(\text{O}_2\text{N})\text{HN.C(:NH).NH.N.C(CH}_3\text{)₂; mw 159.15, N 44.01\%; OB to CO}_2\text{ minus 105.6%; ndls; mp - detonates when brought in contact with a flame; diff sol in w; can be prepd by treating nitroquinuainidine, (O}_2\text{N})\text{HN.C(:NH).NH.NH}_2\text{, with acetone (Refs 1, 2 & 3).}$$

A slightly stronger explosive is obt by treating nitroaminoguainidine with aqueous formaldehyde

N'-Nitromethylenexionguainidine,

$$(\text{O}_2\text{N})\text{HN.C(:NH).NH.N.CH}_2\text{, mw 131.10, N 53.43\%; OB to CO}_2\text{ minus 30.5%; ndls, mp - explodes (Refs 1, 2 & 3).}$$

Dimethylmethylgalactoside and Derivatives

2,3-Dimethyl-α-methylgalactoside,

\[ \text{HOH}_2 \text{C} - \text{C} - \text{C} - \text{C} - \text{OCH}_3 \]

mw 267.24; clear syrup, nD 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, C₁₀H₁₄N₂O₁₀; mw 312.24, N 8.97%; crystals (from alc), mp 88–90°; can be prep'd 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,

\[ \text{H}_3\text{C} \text{OH}_2 \text{C} - \text{C} - \text{C} - \text{C} - \text{CH} \]

H OH H OCH₃ OCH₃

mw 267.24; ndls (from eth), mp 45–46.5°; prep'd by hydrolyzing 3,4-acetone-2-methyl-β-methylgalactoside (Ref 4)

2,6-Dimethyl-β-methylgalactoside-3,4-dinitrate, C₁₀H₁₆N₂O₁₀; mw 312.24, N 8.97%; ndls (from MeOH), mp 88–88.5°; prep'd 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,

\[ \text{H}_3\text{COH}_2 \text{C} - \text{C} - \text{C} - \text{C} - \text{OCH}_3 \]

H OH H OCH₃ H

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-isopropyldiene-α-methylgalactoside for 30 mins at 0° with 30% fuming nitric acid in dry chlf (Ref 5)

3,4-Dimethyl-β-methylgalactoside, C₁₀H₁₃O₆; 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 K₂CO₃ and extraction with chlf (Ref 6)

3,4-Dimethyl-β-methylgalactoside-2,6-dinitrate, C₁₀H₁₄N₂O₁₀; mw 312.24, N 8.97%; plates (from aq MeOH), mp 75–76°; obtd by repeated methylation of β-methylgalactoside with MeI & Ag₂O in dry acetone at 45° (Ref 6)

No expl props of the dinitro derivs are reported


Dimethylmethylglucoside and Derivatives

2,3-Dimethyl-β-methylglucoside,

\[ \text{HOH}_2 \text{C} - \text{C} - \text{C} - \text{C} - \text{C} - \text{H} \]

H OH H OCH₃ OCH₃

mw 222.23; col syrup, nD 1.4690; obtd from benzylidene-β-methylglucoside by methyla-tion and hydrolysis using N/20 HCl in acetone (Ref 4). See also Refs 7 & 8

Dimethylmethylglucoside-6-dinitrate, C₁₀H₁₄N₂O₆; mw 267.24, N 5.24%; props & methods of prep of two derivs are given in Refs 4 & 9

2,3-Dimethylmethylglucoside-4,6-Dinitrate, C₁₀H₁₆N₂O₁₀; 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,
Dimethylphenaphthalene and Derivatives

2,6-Dimethylphenaphthalene, (CH₃)₂C₆H₄C₁₀H₈,

mw 156.22, w h lfts, sp gr 1.142 at 0°/4°,
mp 110°-111°, bp 261°-62° at 767 mm. Prepn
& other props in Ref 1

1,4,5(?)-Trinitro-2,6-dimethylphenaphthalene,

(CH₃)₂C₆H₄(NO₃)₃; mw 291.22, N 14.93%;

pur e 1-x yl ndls (from AcOH), mp 243°;

obtd with dinitro derivs when 1-nitro-2,6-
dimethylphenaphthalene in glacial ac is ni-
trated 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 Dimethyamine
and Derivatives in this Vol

2,4-Di(methylnitramino)-3,5-dinitrotoluene.

See 2,4-Bis(methylnitramino)-3,5-dinitro-
toluene 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'-tetrani-
trotobenzophene. See 4,4'-Bis(methylnitramino)-
3,5,3',5'-tetrinitrotobenzophene in Vol 2,
p B149-L

4,4'-Di(methylnitramino)-3,5,3',5'-tetrani-
diphenylmethane. See 4,4'-Bis(methylene-
min)-3,5,3',5'-tetrinitrodiphenylmethane
in Vol 2, p B149-R

3,5-Di(methylnitramino)-2,4,6-trinitrotoluene.

See 3,5-Bis(methylnitramino)-2,4,6-trinitro-
toluene in Vol 2, p B150-L

Di(methyl-nitropyrazolyl) -triazene. See
1,3-Bis(3-methyl-4-nitro-5-pyrazolyl)-tria-
zeno in Vol 2, p B150-R

Dimethylnitrosamine. See under Dimethy-
amine and Derivatives in this Vol

2,7-Dimethyl-3,5-octadiyn-2,7-dihydroper-
oxide. See under Diacetylenic Dihydro-
peroxides 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)-formamide. See N,N'-
Bis(5-methyl-6-triazol-3-yl)-formamide in
Vol 2, p B151-L

Di(methyltrinitrophosphyl) -amine. See Bis(3-
methyl-2,4,6-trinitrophosphyl)-amine in Vol
2, p B151-R

Di(methylol)-amine and Derivatives

Di(methylol)-amine: N,N'-Bis(β-hydroxy-
methyl)-amine, or Di(methanol)-amine,
HN(CH₃OH)₂; mw 77.08, N 18.17%; may be
considered as the parent compd of its ni-
trated deriv, although not used to prep it:

Di(methylol)-nitramine Dinitrate or Nitro-
mino-dimethanol Dinitrate,

(O₃N)(CH₂ONO₂)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
NH₄OH of the filtrate from the prepn of
RDX by the method of Hale (CA 20, 40);
Di(methylamino)-ethane and Derivatives

N,N'-Dimethylyl-1,2-diamino-ethane,
HOCH₂CN₂H₂O₂H₂–NH₂CH₂OH; mw 120.15, N 23.32%; may be considered as the parent compound of its nitro derivs, although not used to prep them:

N,N'-Dimethylyl-1,2-dinitramino-ethane or 
[Ethylenebis(nitroimino)]-dimethanol,
HOCH₂C₂N₂(NO₂)₂CH₂₂C₂N₂(NO₂)₂CH₂₂CH₂OH, mw 210.15, N 22.66%; compd not isolated, but formed when 1,2-dinitramine stannane,
(CH₂NH₂NO₂)₂, was heated in 37% formalin (HCHO) to 60°C (Ref 3)

N,N'-Dimethylyl-1,4-dinitramino-ethane

Dinitrate or Ethylenebis(nitroimino)-dimethanol Dinitrate,

was added gradually to 98% nitric acid at –15°C; and after 10 mins the mix was added dropwise to anhyd ether at –40°C (Ref 4). This compd also prep'd by Simkins & Wright (Ref 5) and by Majer & Denkstein (Ref 6) is reported to be very unstable


Dimethylbenzene and Derivatives

Dimethylbenzene or Bis(hydroxymethyl)-benzene, C₈H₁₀O₂, mw 138.16, O 23.16%. Three isomers are known:

- m- or 1,2-Dimethylbenzene,
  HC—C(CH₃OH)═C(CH₃OH)
  ।
  HC—CH═CH

  Crystals (from petr eth) or tablets (from eth), mp 64–66.5°C (Ref 1)

- p- or 1,3-Dimethylbenzene,
  HC—C(CH₃OH)═CH
  ।
  HC—CH═CH—C(CH₃OH)

  Crystals (from benz) having a bitter taste, mp 46–47°C, bp 154–59°C at 13mm, d 1.161 at 18°C; sol in eth; v sl sol in w (Ref 2)

- o- or 1,4-Dimethylbenzene,
  HC—C(CH₃OH)═CH
  ।
  HC—C(CH₃OH)═CH

  Ndl's (from w) having a bitter taste, mp 114–16°C; v sol in w, alc & eth (Ref 3)

Other props & methods of prep are given in the Refs

3,6-Dinitro-1,4-dimethylbenzene or

1,4-Dinitro-2,5-dimethylbenzene,

-HC—C(NO₂)═C(CH₂OH)

  (HOCH₂)C—C(NO₂)═CH

  mw 228.16, N 12.28% crystals; mp – not reported; bp – not reported; can be prep'd from 2,5-dimethylbenzene(p-xylene) by the following series of reactions: chlorination to dichloro-p-xylene, nitration to ditnitro-p-xylene-dichloride and hydrolysis to replace chlorines by hydroxyls. It is an expl compd (Refs 5 & 6)

4,6-Dinitro-1,3-dimethylbenzene or

1,3-Dinitro-4,6-dimethylbenzene,

-HC—C(CH₂OH)═C(NO₂)

  (HOCH₂)C—C(NO₂)═CH

  mw 228.16, N 12.28% crystals, mp – not reported, bp – not reported; can be prep'd from 4,6-dimethylbenzene(m-xylene) by the following series of reactions: chlorination to dichloro-m-xylene, nitration to ditnitro-m-xylene-dichloride and hydrolysis to replace
1,4-Dinitro-2,5-dimethylbenzene Dinitrate, 
\[ \text{HC} \rightarrow \text{C(NO}_2\text{)} \rightarrow \text{C(CH}_2\text{O}_2\text{)} \]
\[ (\text{O}_2\text{NO}_2\text{)}\text{C} \rightarrow \text{C(NO}_2\text{)} \rightarrow \text{CH} \]
\[ \text{mw 318.16, N 17.61%, OB to CO}_2 \text{ minus 45.3%; crysts, mp 76}^\circ \text{C; bp – explodes at higher temp. It can be prepd by nitrating 1,4-Dinitro-2,5-dimethylbenzene. It is an exp1 slightly more powerful than Tetryl but more sensitive to impact. Its thermal stability is unsatisfactory (Refs 5 & 6) } \]
\[ 1,3,5-Dinitro-4,6-dimethylbenzene Dinitrate, \]
\[ (\text{O}_2\text{NO}_2\text{)}\text{H}_2\text{C} \rightarrow \text{C(NO}_2\text{)} \rightarrow \text{CH} \]
\[ \text{HC} \rightarrow \text{C(CH}_2\text{O}_2\text{)} \rightarrow \text{C(NO}_2\text{)} \]
\[ \text{mw 318.16, N 17.61%, OB to CO}_2 \text{ minus 45.3%; crysts, mp 114}^\circ \text{C; bp – explodes at higher temp. It can be prepd by nitrating 1,3-Dinitro-4,6-dimethylbenzene. It is an exp1 composite in power and sensiti} \]
\[ \text{ity to Tetryl but of unsatisfactory thermal stability (Refs 5 & 6) } \]
\[ \text{Refs: 1) Beil 6, 910 & 4(587) \quad 2) Beil 6, 914, (446) & 146001 \quad 3) Beil 6, 919, (446), [891] & 146081 \quad 4) Beil 6, not found (Di-}
\]
\[ \text{nitro- and Dinitrodinitrate derivs) 5) C.M.}
\]
\[ \text{Steine, USP 1309551 & 1311926 (1919) & CA 13, 2282 & 2598 (1919) \quad 6) C.M. Steine, USP 1370067 (1921) & CA 15, 1622 (1921) } \]

**Dimethylolbutanol or Bis(hydroxymethyl)-butanol and Derivatives**

2,2-Dimethylol-1-butanol or Trimethylol-ethylmethane,
\[ \text{CH}_2\text{(OH)}_2, \text{C(H}_2\text{O})_2, \text{CH}_2\text{.CH}_3; \text{ mw 134.17; crysts (from eth + acet), mp 58.8–59}^\circ \text{C; was prepd by condensation of butyaldehyde with formaldehyde in w contg Ca(OH)}_2 \text{ (Refs 1 & 4) } \]
3,3-Dimethylol-2-butanol,
\[ \text{CH}_3, \text{CH(OH)}_2, \text{C(3-CH}_2\text{O})_2, \text{CH}_3; \text{ mw 134.17; crysts, mp 59 to 66}^\circ \text{C; bp 134–37}^\circ \text{C at 3–4mm Hg; can be prepd by the oxidation of 3,3-}
\]
\[ \text{dimethyl-1-butanol. It was nitrated to dinitrate & trinitrate derivs. The dinitrate was an unstable & volatile oil and for this reason not of interest for military applica-}
\[ \text{tions (Ref 3, p3). The Trinitrate is de-}
\]
\[ \text{scribed below } \]

2,2-Dimethylol-1-butanol Trinitrate,
\[ \text{CH}_2\text{(ONO)}_2\text{).C(H}_2\text{.ONO)}_2\text{.CH}_2\text{.CH}_3; \text{ mw 269.17, N 15.61%, OB to CO}_2 \text{ minus 50.5%; crysts (from acet + alc), mp 51.2}^\circ \text{C; prepd by nitrating the 2,2-dimethylol parent compd with HNO}_3\text{.H}_2\text{SO}_4 \text{ mixed acid (Ref 2) } \]

3,3-Dimethyl-2-butanol Trinitrate,
\[ \text{CH}_3, \text{CH(OH)}_2, \text{C(3-CH}_2\text{O})_2, \text{CH}_3; \text{ mw 269.17, N 15.61%; (crude product obt as an oil by Warren had N 14.1%, OB to CO}_2 \text{ minus 50.5%. The oily product puffed off at 245}^\circ \text{C (after 5 seconds heating), and it did not ex-}
\]
\[ \text{plode on impact of 100cm fall of a 2kg wt using USButMines machine; thermal stability by 82.2}^\circ \text{C KI Test, 1 min; volatility at 27}^\circ \text{C in 48 hrs, 0.25% and hygroscopicity at 30}^\circ \text{ C & 90% RH, 41.1%. It was prepd at PicArsn Lab by Warren on adding in small portions and with agitation 25g of pulverized dimethylol-}
\]
\[ \text{butanol to cooled to 0}^\circ \text{C mixed acid consisting of nitric acid 25.3, sulfuric acid 59.6 & water 15.1% in the ratio of 1.5 parts of nitric acid to 1 part of dimethylbutanol; time of nitrat-}
\]
\[ \text{on after addn of butanol was ca 1.5 hrs. After nitratons the batch was poured on}
\]
\[ \text{cracked ice with stirring and the trinitrate dissolved in aqueous mixed acid extracted with factory ether; the ethereal soln was neutralized with Na bicarbonate soln,}
\]
\[ \text{washed with w and the separated oil decanted and dried overnight over anhyd Na sulfate, followed by evaporation of residual ether in a vacuum desiccator over Ca chloride } \]
\[ \text{Refs: 1) Beil 1, [23491] \quad 2) Beil 1, [23501] \quad 3) K.S. Warren, "Study Nitrates of Poly-}
\]
\[ \text{hydric Alcohols as Explosives", PATR 1103(1941) \quad 4) L. Méidard & M. Thomas,}
\]
\[ \text{MP 35, 158 (1953) (2,2-Dimethylol-1-butanol and its Trinitrate) \quad 5) No later Refs found thru 1966 } \]

**Dimethylolbutanone and Derivatives**

3,3-Dimethylol-2-butanone; Dimethylol-
methylethylketone or \( \beta \)-Methyl-\( \beta \)-hydroxy-
methyl-\( \gamma \)-ketobutanol,
CH\(_3\).CO.C(CH\(_2\)OH)\(_2\).CH\(_3\), mw 132.16; crystals, 
mp 54–59\(^\circ\)C, bp 121–125\(^\circ\)C at 9–14\(\mathrm{mm}\); can be 
prepd by condensation of methylthyl ketone 
with 2 moles of formaldehyde in presence of 
an alkaline catalyst, such as 2\(\mathrm{N}\) NaOH (Ref 3)
It gives on nitration an explosive di-
nitrate
3,3-Dimethylol-2-butane 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\(^\circ\) after heating for 
5 secs; it did not explode by impact of 2kg 
wt at 100\(\mathrm{mm}\); hygroscopicity at 30\(^\circ\) & 90\% RH 
0.40\%; volatility at 27\(^\circ\) in 48 hrs 0.34\%; ther-
mal stability at 82.2\(^\circ\) KI test 1 min. Warren 
prepd it at PicArsn Lab by adding in small 
portions and with agitation, pulverized di-
methylolbutanone to cooled to ca 5\(^\circ\) 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 pro-
duct 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 Dimethylol-
propane or Pentaglycol in this Vol

**Dimethylol-dimethyl-glutarate 
and Derivatives

(2,2-Dimethylol)-dimethyl Glutarate,
CH\(_2\)COO(CH\(_2\)OH)
| | | |
| CH\(_2\)COO(CH\(_2\)OH)

mw 252.22; no information available on this, 
the parent compd of its nitrated deriv 
(2,2-Dimethylol)-dimethyl-glutarate Tetra-
nitrate called by Blatt Pentaerythritol Di-
glycolate Tetranitrate,
CH\(_2\)COO(CH\(_2\)ONO\(_2\))
| | |
| CH\(_2\)COO(CH\(_2\)ONO\(_2\))

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 prep-
b by nitration of parent compd (Refs 3, 4 & 5) 
Refs: 1) Beil, not found (Parent compd) 
2) Beil, not found (Tetranitrate) 3) West-
fälisch-AnhaltscheSprengstoffAG, 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 I(1947), p105

**Dimethylol-di(oxymethylene)-peroxide or 
Bis(hydroxymethylene)peroxide,
(HO\(_2\)C.C.H\(_2\).O\(_2\).C.H\(_2\).O.C.H\(_2\)).OH; mw 
154.12, O 62.9\%; heavy oil, sp g 1.2765 
at 16.7\(^\circ\), n\(_D\) 1.4216 at 16.7\(^\circ\); it is a weak 
expl, sol in w. It can be prepd by treating 
an etheral soln of Di(methylol)peroxide, 
HO\(_2\)C.O\(_2\).C.H\(_2\).OH, with 2 mols of formal-
dehyde, HCHO (Ref 2) 
Note: On long standing, the oil gradually 
changes to a crystalline substance, C\(_2\)H\(_2\).O\(_2\), 
which partly sublimes at 140–145\(^\circ\); the 
balance melting at 152\(^\circ\). A very expl sub-
set can be prepd from it, called Cyclicdiox-
myhyleneperoxide (called Pertertiomyethylene, 
in Ger) (See Vol 3 of Encycl, p C594-L) 
Refs: 1) Beil, not found 2) A. Rieche & 
R. Meister, Bet 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, (HO\(_2\)C)\(_2\).C(H\(_2\)).C.H\(_3\), mw 90.12; no 
info on this compd was found in the litera-
ture thru 1966. It may be considered the 
parent compd of its nitrated derivs: 1,1-
Di(methylol)-2-nitroethane and 1,1-Di(methylol-
nitrate)-2-nitroethane
1,1-Di(methylol)-2-nitroethane or Bis(methylol)-nitromethylmethane,
(HO\_2C\_2-CH\_2(CH\_2(NO\_2)\_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(nitroxy-
methyl)-nitromethylmethane or 1,1-
Di(methylol)-nitromethylmethane Dinitrate,
(O\_2NO.H\_2C)\_2CH\_2CH\_2(NO\_2)\_2; mw 225.12, N
18.67%, OB to CO\_2 minus 24.88%; crysrs,
mp 39.5\(^\circ\); explodes at higher temps; was
prepd by Wyler (Ref 3) on nitrn attion of 1,1-
di(methylol)-2-nitroethane and proposed for
use as an explosive

1,2-Di(methylol)-ethane,
(HO\_2C)\_2CH\_2.CH\_2(CH\_2OH), mw 90.12; no
info on this compd was found in the liter-
ature thru 1966. It may be considered as the
parent compd of its nitrated derivs: 1,2-
Di(methylol)-1-nitroethane and 1,2-Di(methyl-
olnitrate)-1-nitroethane

1,2-Di(methylol)-1-nitroethane,
(HO\_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(nitroxyethyl)-1-nitroethane; or
1,2-Bis(nitroxyethyl)-1-nitroethane,
(O\_2NO.H\_2C).CH(NO\_2).CH\_2(CH\_2ONO\_2)\_2; mw
225.12, N 18.67%, OB to CO\_2 minus 24.88%;
no info on this compd was found in the liter-
ature thru 1966
Refs: 1) Beil, not found [Di(methylol)-
ethane] 2) Beil, not found [Nitrate derivs
of Di(methylol)-ethane] 3) J.A. Wyler, USP
2195551 (1940) & CA 34, 5283 (1940)
4) Le Vévéinaire 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)\_2.C.CH(CH\_2.OH).CH\_2.CH\_2.CH\_2. CH-
(CH\_2.OH).CH\_2.OH), mw 192.25; may be
considered as the parent compd of its nit-
trated 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-tetramethyloctane Tetra-
nitrate (called by Blatt, Ref 3)

\[ \text{CH}_2.C.(\text{NO}_2)(\text{CH}_2\text{ONO}_2)\_2 \]
\[ \text{CH}_2.C.(\text{NO}_2)(\text{CH}_2\text{ONO}_2)\_2 \]
mw 462.25, N 18.18%; OB to CO\_2 minus
31.2%; crysrs, mp 85\(^\circ\). It is an expl more
powerful than TNT (137% TNT by Ballistic
Mortar Test); temperature of explosion
(calcld) 3731\(^\circ\)K; heat of expln at const vol
498.3kcal/mol; and heat of combstn at Cy
1104.4kcal/mol. It can be prep'd by conden-
sation of 1,5-dinitropropane with formalde-
hyde, followed by nitrntion of the product
Refs: 1) Beil, not found 2) H.B. Haas,
OSRD Rep 2616 (1943) 3) A.H. Blatt,
OSRD 1941 (1944) 4) Anon, OSRD 5746
(1945) 5) ADL Pure Expl Compds, Vol
(compd No 82)

Dimethylolhexanediol or Bis(hydroxymethyl)-
hexanediol and Derivatives
2,5-Dimethylol-1,6-hexanediol,
(HO)CH\_2.CH(CH\_2.OH).CH\_2.CH\_2.CH(CH\_2.OH).
-CH\_2(OH); mw 178.22; may be considered as
the parent compd of its nitrated deriv, al-
though not used to prep it:
2,5-Dinitro-2,5-dimethylol-1,6-hexanediol
Tetranitrate; 2,5-Bis(hydroxymethyl)-2,5-
dinitro-1,6-hexanediol Tetranitrate or 1,4-
Dinitro-1,1,4,4-tetramethyloctane Tetra-
nitrate (called by Blatt, Ref 3)

\[ \text{CH}_2.C.(\text{NO}_2)(\text{CH}_2\text{ONO}_2)\_2 \]
\[ \text{CH}_2.C.(\text{NO}_2)(\text{CH}_2\text{ONO}_2)\_2 \]
mw 448.22, N 18.75%, OB to CO\_2 minus
21.4%; crysrs, mp 134\(^\circ\). A powerful expl:
Ballistic Mortar Test 147% TNT; temp of
expln (calcld) 4189\(^\circ\)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 prep'd by condens-
sation of 1,4-dinitrobutane & formaldehyde, followed by nitration of the product

Dimethylolmethane, Propanediol, Trimethylolpropaneglycol or Bis(hydroxymethyl)methane and Derivatives

Dimethylolmethane, HOH₂C.CH₂.CH₂.OH; mw 76.09; liq, fr p minus 32°, bp 214.2°, d 1.0538 at 20°, nD 1.4389–1.4397 at 20°. Other props & methods of prepn are found in Beil (Ref 1)

Dimethylolmethane Dinitrate,
(O₂N)₂CH₂.CH₂.CH₂(O₃NO₂); mw 166.09, N 16.87%, OB to CO₂ minus 28.9%, brn, oily, having a sweet taste, fr p below –20°, d 1.3952 at 20°; miscible with MeOH, eth, chl, benz & acet; v sl sol in CS₂. It was first prep by Blechta (Ref 3) by nitrating trimethylolglycol with mixed acid at 8°. This compd showed the same sensitivity 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)-methoxethane; [Bis(hydroxymethyl)-2-methoxy-ethane or 2-Methylmethoxypropane and Derivatives

1,1-Di(methylol)-2-methoxy-ethane,
HOH₂C—CH—CH₂OH

CH₂OCH₃ ; 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, (O₂N)₂H₂C.C(NO₃)₂—CH₂(O₃NO₂);

CH₂OCH₃

mw 255.15, N 16.47%, OB to CO₂ 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,

H₃C(CH₂OH)₂

H₃C—C≡C

H₃C—CH=CH

H₃C—CH=O

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,
(O₂N)C(CH₂.O₂NO₂)₂

H₃C—C≡C

H₃C—CH=O

H₃C—CH=O

mw 332.19, N 16.87%, OB to CO₂ 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-nitrophenylmethane 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,

(O₂N)₂C(CH₂.O₂NO₂)₂

H₃C—C≡C

H₃C—CH=O

(O₂N)₂C—CH=O

mw 377.19, N 18.57%, OB to CO₂ 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 combustn 1022.9 kcal/mole. It can be prepd by condensing phenylinitromethane with formaldehyde and nitrating the product (Refs 2 & 3)


**Di(methylol)-methylene diamine or Bis(hydroxymethyl)-methylene diamine and Derivatives**

$N,N'$-Di(methylol)-methylene diamine,

$$\text{(HOH}_2\text{C})\text{NH.CH}_2\text{NH(CH}_2\text{OH)};$$

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)-methylene diamine

$$\text{(HOH}_2\text{C})\text{NH.H}_2\text{N(NO}_2\text{XCH}_2\text{OH)};$$

mw 196.13, N 28.57%; prisms, mp 70–75°C (decomp), expl at high temp. It can be prepd by passing gaseous formaldehyde to saturation thru a soln of methylated diamine,

$$\text{(O}_2\text{N})\text{NH.CH}_2\text{NH(NO}_2\text{)},$$

in ethyl acetate, followed by cooling the mix to 0°C (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,

$$\text{CO.NH.CH}_2\text{OH};$$

mw 148.12, N 18.91%; crysts (from 50% acetic acid), mp 204°C; prepd by condensation of formaldehyde (40%) with oxamide, (CONH)$_2$, in the presence of K$_2$CO$_3$ (Ref 2). Welcher (Ref 3) reported a novel synthesis from (CN)$_2$, paraformaldehyde & 12N HCl at 28–32°C

Note: No nitrated derivs of $N,N'$-Di(methylol)-oxamide were found in the literature thru 1966


**Di(methylol)-oxazolidone or Bis(hydroxymethyl)-oxazolidone and Derivatives**

4,4'-Di(methylol)-1-oxazolidone, or 4,4'-Bis(hydroxymethyl)-1-oxazolidone,

$$\text{C(O)}—\text{NH}_{\text{CH}_2\text{OH}_2}\text{O}—\text{CH}_2$$

mw 147.13, N 9.52%; crysts (from methanol), mp 108–109°C (Ref 3) & 109.5–110.7°C (Ref 4). It was prepd from trimethylaminomethane, H$_2$N.CH$_2$.OH, and chlorourethane (ethyl-chlorocarbonate), CICO$_2$.C$_2$.H$_5$, in aq NaOH soln, as described on pp 50 & 128–29 of Ref 3

4,4'-Di(methylolnitrate)-1-oxazolidone or 4,4'-Bis(nitroxyethyl)-1-oxazolidone,

$$\text{C(O)}—\text{NH}_{\text{C(CH}_2\text{ONO}_2}_2}\text{O}—\text{CH}_2$$

mw 237.13, N 17.71%; crysts (from methanol), mp 106–107°C; was prepd by treating the above compd with 98% nitric acid at 5–10°C (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(nitroxyethyl)-1-oxazolidone,

$$\text{C(O)}—\text{N(NO}_2\text{)}—\text{C(CH}_2\text{ONO}_2}_2\text{O}—\text{CH}_2$$

mw 282.13, N 19.88%; crysts (from methanol), mp 122–123°C; expl at higher temps; was prepd by nitrating 4,4-di(methylol)-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


Di(methylol)-peroxide- Bis(hydroxymethyl)-peroxide; Dimethylenediol-peroxide; Di-hydroxydimethyl-peroxide or Diformalperoxidehydrate [Called in Ger Dioxym-dimethylperoxy] or Bis(oxymethylene-peroxy),

\( \text{HOH}_2\text{C}—\text{O}—(\text{CH}_3\text{OH})—\text{w} 92.08, \text{O} 69.51\%\); large prisms (from eth or chlor), mp 64–65° (with sl decomp); v sol in w & alc; less sol in cold eth; sl sol in benz & chlor. When in the dry state it is stable in storage; explodes on heating to 70° in a direct flame or when introduced into NH3 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 "Hexoxymethyleneperoxy". Nef (Ref 3) prepd di(methylol)-peroxide by treating formaldehyde with ag hydrogen peroxide and called the product "Diformalperoxyhydrat". 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 Hexamethylenetriperoxydiamin. Its structure was detd as:

\[
\begin{align*}
\text{CH}_3—\text{CHO}—\text{CH}_2—\\
\text{N—CH}_2—\text{O}—\text{CH}_2—\\
\text{CH}_2—\text{O}—\text{CH}_2——\text{N}
\end{align*}
\]

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 Hexamethylenetriperoxydiamine]

Fenton (Ref 5) prepd Di(methylol)-peroxide by evaporating equal volumes of formaldehyde and hydrogen peroxide. Wieland & Wingerl (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 \( \beta \)-butylene (Ref 1). p[642]

(See also Refs listed below)


Di(methylol)-phenylmethane or Di(methylol)-toluene. See Di(methylol) -methylbenzene in this Vol

Di(methylol)-propane; Di(methylol)-dimethylmethane; Dimethyl-propanediol; Bis(hydroxymethyl)-propane; or Pentaglycol (PGc) and Derivatives

2,2-Di(methylol)-propane or 2,2-Dimethylpropane-1,3-diol, \( \text{CH}_3\text{C}—\text{CH}_3 \)

\( (\text{CH}_3\text{OH})_2; \text{W} 104.15\) col ndls; mp 127–129°; hp 210° at 760mm and 110° in vacuo; v sol in w, alc and in hot trichloroethylene. It was first prepd in 1894 by Apel & Tollen (Ref 2) by heating dilute formaldehyde with isobutylaldehyde 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 isobutylaldehyde
2CH₂O + (CH₃)₂CH·CHO + NaOH =
HCOONa + (CH₃)₂C(CH₂OH)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 separated funnel (Ref 5)

Nitrification of pentaglycol gives explosives

2,2-Di-(methylol)-propane Dinitrate; 2,2-
Di-(nitroxyethyl)-propane; Pentaglycol
Dinitrate; 2,2-Dimethyl-1,3-propanediol
Dinitrate or Dimethylidimethylmethane
Dinitrate, abbreviated as PGcDN,
CH₃-C-CH₃

(CHO·ONO₂)₃, mw 194.15, N 14.43%,
OB to CO₂ minus 74.2%; It yel crystals, 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) prep 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 q Na carbo-
date and washed with w. Blomquist &
Fiedorek (Ref 5) prep the PGcDN by nit-
trating 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 – 715.8 kcal/mol
Heat of Formation – 97.7 kcal/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 mixtures 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, carbonous 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
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(bydroxyethyl)-ethane Trinitrate. These
mixtures were of low sensitivity and practically
nonfreezing in winter and were claimed to
be suitable replacements for mixtures based
on NG–NGc or NG–Nitrosugars
Refs: 1) Beil 1, 41391 (PGc) 2) Beil, not
found (PGcDN) 3) C. Spaeth, USP 183045
(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,
CH₂OH

CH₃CH₂OH

CH₂OH; mw 104.15; may be con-
sidered as the parent compd of its nitrate
derivs, although not used to prep them:

1,1-Di-(methylol)-1-nitropropane or 2-Ethyl-
2-nitropropane-1,3-diol,
\[
\text{CH}_2\text{OH} \\
\text{CH}_3\text{C}_2\text{H}_5\text{C:NO}_2
\]

\[
\text{CH}_2\text{OH}; \text{mw} 149.15, \text{N} 9.39\%; \text{ndls (from w)}, \text{mp} 57-58^\circ; \text{decomp when distilled at 100^\circ \text{press}; sol in alc, w \& eth; was prepd by condensation of 1-nitropropane \& formaldehyde in the presence of K}_2\text{CO}_3\text{(Ref 1)}
\]

1,1-Di(methyl)-1-nitropropane Dinitrate or 2-Ethyl-2-nitro-1,3-propanediol Dinitrate,

\[
\text{CH}_2\text{ONO}_2
\]

\[
\text{CH}_3\text{C}_2\text{H}_5\text{C:NO}_2; \text{mw} 239.15, \text{N} 17.57\%; \text{liq, d 1.443 at 20^\circ, nD 1.4734 at 20^\circ (Ref 3)}; \text{it withstands 82.2^\circ \text{Heat Test for 5 mins; power by ballistic mortar test 92\% of Blasting Gelatin; was prepd by condensing formaldehyde with 1-nitropropane in an alkaline medium, and nitrating the product. It was reported to be a poor gelatinizer for soluble NC used in blasting compns}}
\]

See also the Refs listed below

Refs: 1) Beil 1, 483 & 2199 2) Blatt, OSRD 2034(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_\text{comb} as 693.5 \text{kcal/mole}) \& CA 49, 11284(1955) 5) J. Salle, MP 36, 305 (1954) (Detd the Dinitrate to be a vasodilator, less effective than NG, but exercising a more lasting effect) \& CA 49, 16218(1955) 6) K. Szyc-Lewanska \& M. Syczewski, Nitro Compds, ProclnternaSymp, Warsaw 1963, 257 (Pub 1964 in Engl) (Detd the Dinitrate to be a good polymerizer for Me methacrylate) \& CA 63, 14989(1965)

Di(methyl)toluene. See Di(methyl)methylbenzene in this Vol

\[
\text{N,N'-Di(methyl) Urea and Derivatives}
\]

\[
\text{HO.H}_2\text{C}---\text{HN}.\text{C}.\text{NH}---\text{CH}_2\text{OH}; \text{mw} 120.11, \text{N 23.33\%; crysts, mp 126^\circ; sol in w \& methanol; insolv in eth; can be prepd by combination of urea \& formaldehyde in the presence of salts of alkaline catalysts. It is capable of polymerization to synthetic resins}
\]

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:

\[
\text{HO.H}_2\text{C}---\text{HN}.\text{C}.\text{NH}---\text{CH}_2\text{OH}+\text{HC(NO}_2)_3\rightarrow \text{O}
\]

\[
\text{HO.H}_2\text{C}---\text{HN}.\text{C}.\text{NH}---\text{CH}_2\text{C(NO}_2)_3+\text{H}_2\text{O}
\]

The resulting solid substance had mw 255.14, N 27.66\%, melted at 183-184^\circ with decomp and could be detonated with difficulty by hammer blow

The name for the above explosive was not given in US Rubber Co reports No 3 \& No 4. We suggest N-(Methyl)-N'-(2,2,2-trinitroethyl) Urea

When this substance was treated with another mol of nitroform, the following reaction was believed to take place:

\[
\text{O}
\]

\[
\text{HO.H}_2\text{C}---\text{HN}.\text{C}.\text{NH}---\text{CH}_2\text{C(NO}_2)_3+\text{HC(NO}_2)_3\rightarrow \text{O}
\]

\[
\text{(O}_2\text{N)}_3\text{C.H}_2\text{C}---\text{HN}.\text{C}.\text{NH}---\text{CH}_2\text{C(NO}_2)_3+\text{H}_2\text{O}
\]

The resulting solid substance had mw 386.16, N 29.02\%, melted at 189-90^\circ with decomp and detonated readily and sharply when struck with a hammer

The name for the above substance was not assigned in US Rubber Co reports No 3 \& No 4, but it is probably correct to name it N,N'-Bis(2,2,2-trinitroethyl) Urea


\[
\text{Dimethyloxamide and Derivatives}
\]

\[
\text{N,N'-Dimethyloxamide (abbr as DMO), H}_2\text{C.NH.CO.CO.NH.CH}_3; \text{mw} 116.12, \text{N}
\]
N,N'-Dinitroso-N,N'-dimethyloxamide (DNsODMO)
H₃C.N(NO).CO.CO.N(NO)₂.CH₃; mw 174.12, N 32.18%; crystals, mp not reported. It was prep by Reimlinger (Ref 17) by action of nitrous gases or N₂O₄ in CCl₄ on DMO. He found it stable and esp suitable as an intermediate for the prep of CH₂N₂ (Diazomethane).

Preussman (Ref 18) stated that in one of several expts in prep of DNsODMO, the chlf used as the solvent for nitrization was not distd in vacuo but removed at atm pressure on a hot water bath in a hood. An extremely violent expn 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 (DNDO)
(Code named MNO),
H₃C.N(NO)₂.CO.CO.N(NO)₂.CH₃; mw 206.12, N 27.18%, OB to CO₂ minus 38.8%; col ndls; sp gr 1.52 (crysts), mp 123-124° (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) prep 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) prep'd DNDO in 97% yield by nitrating dimethyloxamide with mixed nitric-sulfuric acid congl 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 DNDO:
Abel's Test at 80° - more than 1 hour (Ref 15)
Heat of Combustion - 511.9 kcal/mol at Cv (Ref 12)
Heat of Formation - 74.5 kcal/mol, based on heat of combustn of 508.4 kcal/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 hydrolys)
(Ref 2)
Impact Sensitivity - FI (Figure of Insensitivity) 89% of TNT, which means that it is less sensitive than TNT. Sensitivity using Brueton 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 hor concd HCl; reacts with alkalis with formation of salts of methyl-nitramine (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. 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), DNNDMO 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 DNNDMO and 70 parts of PETN yield an eutectic which melts at 100° and can be cast to form an HE insensitive to pass the Rifle Bullet Test. Its rate of deton 8500m/sec is equal to that of PETN under best conditions. The further addn of DNNDMO or incorporation of camphor lowers the mp still more; it affects the briskness only slightly, but has a significant phlegmatizing action. A mixture of PETN 60, DNNDMO 30 and dimethylxylate 10% melts at 83° and has, when cast, a vel of deton 7900m/sec which is higher than vel of cast Picric Acid

Herz & Naoum (Ref 10) reported moldable expls formed by mixing 50 parts of DNNDMO with TNT, TNNaphthalene, TNChlorobenzene, etc.

Allenby & Wright (Ref 13) reported prepn of a pourable HE with setting point 75° when mixing DNNDMO in equimolecular amt with Picric Acid. The rate of deton of this expl was 6680 to 7500m/sec, depending on the densities of 1.41 to 1.59.

Accdg to Blatt (Ref 12), DNNDMO has been evaluated as betw Tetryl & TNT in performance, but of lower stability

Refs: 1) Beil 4, 61, (330), [564] & 1131 (DMO)

Dimethyl-xylate and Derivatives

3,3-Dimethyl-xylate, Bis(methyl)-xylate or Trimethylene oxide,

\[ \text{CH}_3 - \overline{\text{O}} - \text{CH}_2 - \overline{\text{C}}(\text{CH}_3)_2 - \overline{\text{O}} - \text{CH}_2 - \overline{\text{C}}(\text{CH}_3)_2 \] [mw 86.13, O 18.58%; liq, bp 79.5–90°; obtd from 3-chloro-2,2-dimethyl-1-propanol by stirrin with powd KOH in tert-amyl alcohol at 95°, and boiling at 48° to separate the xylate (Ref 4)]

3,3-Di(or Bis)(nitroatomethyl)-xoate,

\[ \text{CH}_3 - \overline{\text{O}} - \text{CH}_2 - \overline{\text{C}}(\text{CH}_3\text{ONO}_2)_2 - \overline{\text{O}} - \text{CH}_2 - \overline{\text{C}}(\text{CH}_3\text{ONO}_2)_2 \] [mw 208.13, N 13.46%; crystals, mp gr 1.76, mp 88–91°, ignition temp 233°; somewhat sensitive to impact (34cm with 2.5kg wt); thermal sta:
bility by vacuum test, 1.12 ml gas/g at 100° in 48 hrs. It was prep at Hercules Powder Co by Kouba et al by treating Pentacythritol Trinitrate with alcoholic KOH, as described in detail in Ref 2. Elrick et al (Ref 3) prep the compd by reaction of Pentacythritol Trinitrate & Na ethylate or alcoholic NaOD at 70° for 8 hrs


3,3-Di(or Bis)[nitromethyl]oxetane,

\[ \text{CH}_3 \text{— O — CH}_2 \text{— C(CH}_3\text{NO}_2\text{)}_{3,2} \text{; 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}_3, 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


2,4-Dimethyl-1,3-pentadiene

and Derivatives

2,4-Dimethyl-1,3-pentadiene

\[ \text{CH}_3 \text{— CH}_3 \text{— CH}_3 \text{CH}_3 \text{CH}_3 \text{— O — O} = \text{ mw 128.17, O 24.97%; solid, mp — forms a dimer, C}_7\text{H}_6\text{O}_4 \text{ 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-pentadiene 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}

\[ \text{[H}_5\text{C}_3\text{CH}_2\text{CH}_2\text{CH}_3\text{]}_2 \text{O} \]


Dimethylpentane and Derivatives

Dimethylpentane or Heptane, C\text{7H}_{16}; mw 100.20.
Four isomers are described in the literature:

2,2-Dimethyl-pentane, \text{H}_3\text{C.C(CH}_3\text{)}_2\text{CH}_2\text{CH}_2\text{CH}_3; liq, bp 79.2° (Ref 1)

2,3-Dimethylpentane, \text{H}_3\text{C.CH(CH}_3\text{).CH(CH}_3\text{).CH}_2\text{CH}_3; liq, bp 89.8° (Ref 2)

2,4-Dimethyl-pentane, \text{H}_3\text{C.CH(CH}_3\text{).CH}_2\text{CH}_2\text{CH}_3; liq, bp 80.5° (Ref 3)

3,3-Dimethyl-pentane, \text{H}_3\text{C.CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3; liq, bp 86.1° (Ref 4)

3-Nitroso-3-nitro-2,4-dimethylpentane,

\[ ([\text{H}_5\text{C}_3\text{CH}_2\text{CH}_3\text{]}\text{NO}_2 \text{)}_2 \text{; mw 174.20, N 16.08%; non-freezing blue oil, bp — dec at 54°; was prepd by treating diisopropylketoxime in ether soin with NO}_2 \text{ under cooling & with exclusion of light}

Dinitrodimethylpentane, \text{C}_7\text{H}_4\text{N}_2\text{O}_4; mw 190.20, N 14.73%. Four isomers are found in Beil:

2,3-Dinitro-2,3-dimethylpentane,

\text{H}_3\text{C}_2\text{C(CH}_3\text{NO}_2\text{).C(CH}_3\text{.)NO}_2 \text{; crysts (from alc), mp 88—88.4° (Ref 6)

2,4-Dinitro-2,3-dimethylpentane,

\text{H}_3\text{C.CH(NO}_2\text{).CH(CH}_3\text{).C(CH}_3\text{.)NO}_2 \text{; pale yel oil, bp 90—92° at 0.5mm press; soin in alkali (Ref 6)

2,4-Dinitro-2,4-dimethylpentane,

\text{H}_3\text{C}_2\text{C(NO}_2\text{).CH}_2\text{.C(NO}_2\text{).CH}_3\text{.)NO}_2 \text{; prisms, mp 81—82°; soin in eth & petr ether (Ref 5)
3,3'-Dinitro-2,4-dimethylpentane,
(H₃C)₂CH₃C(NO₂)₂CH(CH₃)₂; liq, bp
203–207°C at 717 mm press (with partial de-
compo) (Ref 5)

Their methods of prep are given in Refs.
No expl props reported
Ref/s: 1) Bei/l 1, 157, [120] & [442] 2) Bei/l 1,
[120] & [445, 449] 3) Bei/l 1, 158, (59),
[121] & [449] 4) Bei/l 1, 158, (59), [121]
& [452] 5) Bei/l 1, 158 (Nitroso-nitro deriv
& Dinitro derivs) 6) Bei/l 1, [448] (Dinitro
derivs)

Dimethylpentane and Derivatives
4,4-Dimethyl-2-pentanone,

\[
\text{CH}_3
\]
\[
\text{CH}_3 \cdot \text{CO} \cdot \text{CH}_2 \cdot \text{C} \cdot \text{CH}_3
\]

CH₃ ; mw 114.18, liq, bp
125–30°C; prepd by oxidation of 2,4,4-
trimethyl-2-pentanol (Ref 1). May be con-
sidered as the parent compd of its trinitro
deriv, although not used to prep it.

4,4-Dimethyl-5,5,5-trinitro-2-pentanone,

\[
\text{CH}_3 \cdot \text{CO} \cdot \text{CH}_2 \cdot \text{C} \cdot \text{NO}_2\text{s}
\]

CH₃ ; mw 249.18, N
16.86%; ltr yel liq, bp 109–13°C at 8mm, or
92–93°C at 2mm; nD 1.4717 at 20°C; 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(NO₂)₃, and mesityl oxide,CH₃-CO.CH·CH(C(H₃)₂),
as described by Schaffner (Ref 2)
Ref/s: 1) Bei/l 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,
H₃C.COO.CH₃ ; mw 62.07, O 51.56%; gas at
RT with a sweet ethereal odor; sp gr 0.8677
0°F/4°C; fr p minus 100–105°C, bp 13.5°C
at 740mm; nD 1.3503 at 0°C; 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 coned sulfuric acid; v sol
in cold eth and alc; sol in toluene and ethyl
acetate. It was prepd by treating dimethyl-
sulfate with 10% hydrogen peroxide and K
hydroxide in nitrogen atmosphere at 15°C
(Refs 1–5)

Sensitiveness of peroxide was deter-
mained by Baker et al (Ref 6)
Ref/s: 1) Bei/l 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(tolui-
dino)-ethane. See Bis(methylphenylamino)-
ethane in Vol 2 of Encycl, p B150-R

Di(methylphenylamino)-propane or Di(tolui-
dino)-propane. See Bis(methylphenylamino)-
propane in Vol 2 p B151-L

N,N-Dimethyl-p-phenylniline. See N,N-
Dimethylanisobenphyl or N,N-Dimethy-
lenylamine

Dimethylphenylcarbamide. See Dimethyl-
phenylurea

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), H₃C.CHN.C₆H₄.NHCH₃; mw 136.19,
N 29.57%; oily liq; browns on standing; bp
165–170°C 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
dist off. The resulting viscous mass was
crystallized from benz and the crystals dis-
solved 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)

$N,N'\text{-Dimethyl-N,N'-dinitroso-m-phenylene diamine}$, $C_6H_4(N(NO)CH_3)_2$; mp 194.19, N 28.85%; phtl. (from alc), mp 109–10°C; was prp'd by nitritating the parent compd with HNO$_3$ (Refs 1 & 2). No expl props were reported.

Refs: 1) Beil 13, 53  2) O. Fischer, Ann 286, 168 (1895)

$N,N'\text{-Dimethyl-4-mononitro-m-phenylene diamine}$, $H_2N.C_6H_4(NO,N(CH_3)_2$; mw 181.19, N 23.19%; yel crystals (from alc), mp 135°C; sol in dil sulfuric acid with a yel color; was prp'd from 3,4-dinitrodimethyl-aniline & alc ammonia under prolonged heating or by heating in a tube at 120°C (Refs 1 & 2).

Refs: 1) Beil 13, 57 & [30]  2) P. van Romburgh, Rec 42, 804 (1923) & CA 18 49 (1924)

$\text{Dimethyltrinitro-m-phenylenediamine}$, $C_6H_4(NO)_3O_4$; mw 226.19, N 24.77%. Four isomers are found in Beil:

$N,N'\text{-Dimethyl-2,4-dinitro-m-phenylenediamine}$, $H_2N.C_6H_2(NO,O)NO_2N(CH_3)_2$; crysts (from acet), mp 166–62°C; was prp'd by treating 2,3,4-trinitrodimethylaniline with alcoholic ammonia at RT (Refs 1, 5 & 6)

$N,N'\text{-Dimethyl-2,4-dinitro-m-phenylenediamine}$, (O$_2$N)$_2C_6H_4(NH.CH_3)_2$; yel crystals, mp 169–70°C; was prp'd by action of an alc soln of methylamine on 2,4,5-trinitromethylaniline on 2,4,5-dinitromethylamino-dimethylaniline in a tube at 125°C (Refs 2 & 5)

$N,N'\text{-Dimethyl-4,6-dinitro-m-phenylenediamine}$, $H_2N.C_6H_2(NO,O)NO_2N(CH_3)_2$; no descript. given in CA or in Beil; was prp'd by action of alc ammonia on 2,4,6-trinitrodimethylaniline (Refs 3, 5 & 6)

$N,N'\text{-Dimethyl-4,6-dinitro-m-phenylenediamine}$, (O$_2$N)$_2C_6H_2(NH.CH_3)_2$; yel nlds (from acet), mp > 290°C (dec); obtd with other products by warming 5-chloro-1,2,4-trinitro-benzene or 5-bromo- deriv with ca 4 moles of benzaaldehyde-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]
5) P. van Romburgh & D.W. Wensink, Versl.-Akad.Amsterdam 23, 967 (1914) & CA 9, 1175 (1915)
6) A. Forster & W. Coulsion, JChem Soc 121, 1995 (1922) & CA 17, 76 (1923)
7) M. Giua, Gazz 53, 55–56 (1923) & CA 17, 2273 (1923)

$\text{Dimethyltrinitro-m-phenylenediamine}$, $C_6H_4(NO)_3O_4$; mw 271.19, N 25.83%. Three isomers are known:

$N,N'\text{-Dimethyl-N',4,6-trinitro-m-phenylenediamine}$, $H_2N.C_6H_2(NO,O)NO_2N(CH_3)_2$; crysts, mp 181.5°C; was prp'd by treating 4,6-dinitro-3-methylisimidino-phentole with an alcoholic methylamine soln; can be nitrated to form an expl pentanitro deriv (Refs 1 & 5)

$N,N'\text{-Dimethyl-2,4,6-trinitro-m-phenylenediamine}$, (O$_2$N)$_2C_6H_4(NH.CH_3)_2$; yel crystals, mp 235–40°C (dec), bp — explodes on heating to high temp; was prp'd by treating 2,3,4,6-tetranitromethylaniline with an aq soln of methylamine (Refs 2, 4, 5 & 6)

$N,N'\text{-Dimethyl-2,4,6-trinitro-m-phenylenediamine}$, $H_2N.C_6H(NO,O)NO_2N(CH_3)_2$; crysts, mp 187°C; was prp'd by action of cold aq dimethylamine soln on 2,3,4,6-tetranitro-aniline, or by treating 2,3,4,6-tetranitrodimethylaniline with cold aq ammonia (Refs 3 & 5)

5) A. Forster & W. Coulsion, 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,N-Dimethyl-2,4,6-tetranitro-m-phenylenediamine, H₃C.NH. C₉H(NO₃)₃.N(NO₂)-CH₃; mw 316.19, N 26.58%, golden-yellow crystals (from acet acid), mp 190-92°, bp - explodes at high temp; can be prep by treating 2,4,6-trinitro-3-methylaminophenotetol with an alcoholic methylamine soln and other methods (Refs 1 & 2)

Refs: 1) Beil 13, 61 & [34] 2) A. Forster & W. Coulson, JChemSoc 121, 1791 (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(methylammonio)-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-trinitro-anilino)-ethane (See Vol 2 of Encycl, p B131-R),

\[
\begin{align*}
\text{N(CH₃)NO₂} & \\
\text{O₂N.C} & \text{— C} \equiv \text{C(NO₂)} \\
\text{H.C} & \text{— C} \equiv \text{N(CH₃)NO₂} \\
\text{NO₂} & 
\end{align*}
\]

mw 361.19, N 27.15%, OB to CO₂ minus 42.1%, brown crysts (from acet + alc), mp 206°(corr) with decomp; sol in acet & in boiling glc AcOH; sl sol in boiling toulene; nearly insol in w and common org solvents such as alc, eth, etc. It was first prep by van Romburg in 1887 (Refs 1 & 2) by boiling N,N'-dimethyl-m-phenylenediamine with fuming nitric acid. The same investigator (Ref 3) prepmed it by dissolving 1 volume of DMPDA in 2 vols of concd sulfuric acid and adding this soln gradually to caled amt of concd nitric acid. This caused the mixture to turn red and to evolve copious vaps of nitrogen oxides. After the evoln of gas stopped, the lig 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) prepmed it by nitrating DMPDA with mixed nitric-sulfuric acid

Van Duin & van Lennep (Ref 6) prepmed it by treating m-Nitroteteryl-(2,4,6-Tetranitrophenyleniminitramine 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 de Paris (Ref 4) prepmed N,N',2,4,6-Pentanitrodimethyl-m-phenylenediamine by nitration with concd nitric acid of \( (O_2N)_2C_6H_4[N(NO_2)CH_3]_2 \), 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, torpedos and detonators either alone or mixed with other expls or with oxidizers such as nitrates, chlorotes 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) prepmed 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 Eq Na hydroxide

Pentanitrodimethylphenylenediamine was discussed by Médard (Ref 11) as a compd
being intermediate between Tetryl and 1,3,5-
Tris(methylnitramino)-2,4,6-trinitrobenzene,

\[
\begin{align*}
&\text{O}_2\text{N},\text{N},\text{CH}_3 \\
&\text{H}_2\text{N} \\
&\text{O}_2\text{N} \\
&\text{N} \\
&\text{O}_2\text{N} \\
&\text{CH}_3
\end{align*}
\]

- 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)
- Société Anonyme d'Explosifs, FrP 391107 (1907) & CA 4, 2735 (1910)
- 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-Nitrodimethyl-
nitro-dimethyl-m-phenylenediamine)
- A.H. Blatt, OSRD 2014 (1944) [2,4,6-
Trinitro-1,3-di(methylnitramine)-benzene]
- L. Médard, MP 33, 46 (1951)

**3,4-Dimethylphenyldrazine**, formula same as above, yel ndls (from eth), mp 57°; its Hydrochloride salt, C₆H₁₂N₂ + HCl, scales (from w), dec at 197° (Ref, p 172)
- Other props & methods of prep'n are found in the Refs
- Refs: Beil 15, 548, 549, 552 & (171, 172, 173, 175)

**4,6-Dinitro-2,5-dimethylphenyldrazine**, \(\text{O}_2\text{N},\text{C} \rightarrow \text{C}(\text{N},\text{NH}_2) \rightarrow \text{C},\text{CH}_3\)

\[
\begin{align*}
\text{H}_3\text{C},\text{C} \rightarrow \text{C}(\text{NO}_2) \rightarrow \text{CH}
\end{align*}
\]

mw 226.19, N 24.77%; yel prisms (from alc), mp 180° (dec); sol in hot benz, chlf, eth & acet; v s l sol in petr eth; the soln in alc or acet gives a brick-red soln in presence of alkali; was prep'd 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)

**Trinitrodimethylphenyldrazine**, C₆H₁₂N₂O₆; mw 271.19, N 25.83%. Two derivs are known: N,N'-Dimethyl-N-(2,4,6-trinitrophenyl)-
hydrazine or \(\alpha,\beta\)-Dimethyl-picrylhydrazine, called by Knorr *Dimethylpikrazid*,
\((\text{O}_2\text{N})_2\text{C}_6\text{H}_2\text{N}(\text{CH}_3)\)\_NH(CH₃); yel-red lftts, mp 141°; bp – puffs off with evolution of flame at higher temps; sol in chlf & ether; diff sol in water & alcohol; was prep'd 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 \(\beta,\beta\)-Dimethylpicrylhydrazine, \((\text{O}_2\text{N})_2\text{C}_6\text{H}_2\text{N}(\text{CH}_3)\)₂, red crysts (from alc), mp 136.5°, bp – explodes on heating above its mp; readily sol in benz; diff sol in eth; was prep'd from reaction of N,N-
dimethylhydrazine & picryl chloride in alcohol KOH (Refs 2 & 4)
- Refs: 1) Beil 15, 493 2) Beil 15, (147)
- L. Knorr & A. Köhler, Ber 39, 3264 (1906)
- H.J. Backer, Rec 31, 152 (1912) & CA 6, 2744 (1912)

**Dimethylphenylurea and Derivatives**

**N,N-Dimethyl-N'-phenylurea**, C₆H₅.NH.CO.N(CH₃)₂; mw 164.20, N 17.06%;
ndls (from w) or crystals (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).

N,N-Dimethyl-N'-[2-nitrophenyl]-urea,
\(O_2N-C_\text{H}_4-NH.CO.N(N(CH)_2)_3\) mw 209,20, N 20.09%; yel oil, fr p – becomes viscous at -15° w/o cryst at -30°; obtd by warming dimethylaniline & 2-nitrophenylisocyanate in ether soin (Ref)

Ref: Beil 12, 694

Dinitrodiphenyleneurea, C\(_6\)H\(_{14}\)N\(_4\)O\(_6\), not found in Beil
N,N'-Dimethyl-N-(2,4,6-trinitrophenyl)-urea or N,N'-Dimethyl-N-picrylurea,
\(O_2N)C_6H_2(N(CH)_2)CO.NH.CH\_3; mw 259,20, N 23.41%; yel ndls, mp volatile in a capillary tube at 150° & melts (w decomp) at 160°; was prepd by reaction of dimethylcarbodi-imide & Picric Acid in eth. No exp exps were reported (Refs 1 & 2)


o-Dimethylphthalate (DMePh) or Methylphthalate, C\(_6\)H\(_{4}\)(COOCH\(_2\))\(_2\), 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\(_\text{v}\) 1120.1 kcal/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 soin 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 soin to 100ml distd w
2) Specific Gravity: Det the sp gr at 15.5°/15.5° by means of a pycnometer 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 (prep 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 soin to 25°. Titrate with 0.5N HCl using 3 drops of phenolphthalein indicator [previously prepd by dissolving 1 g of phpt in 100ml of 8% (by vol) ethanol] until discoloration of soin

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
pH indicator, and neutralize the alcohol
with a few ml of 0.1N NaOH to a faint pink
color. Add to the flask an accurately mea-
sured 50ml portion of the sample and titrate
the mixture with 0.1N NaOH till reappearance
of faint pink coloration

\[
\text{Phthalic Acid} = \frac{0.1665V}{G} \text{N}
\]
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 be-
cause 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
Uns 1027-1029 6) USSpecification
JAN-D-709(1) (June 1949)

**N,N-Dimethylpicramide.** See 2,4,6-Trinitro-
N,N-dimethylphenylamine under N,N-Dimethyl-
aniline and Derivatives

\(\alpha,\beta\)-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; Neopenaite or Tetra-
methyImethane, \(\text{CH}_3\cdot\text{C}-(\text{CH}_3)\cdot\text{C}-(\text{CH}_3)\cdot\text{CH}_3\); mw 72.15;
col gas or very volatile liq; sp gr 0.603 at
\(0^\circ\)/\(0^\circ\), fr p -20\(^\circ\), bp 9.5\(^\circ\); sol in alc, insol
in w; present in small amts in natural gas;
can be prepd by reaction of tert-butyl iodide
& Zn dimethyl, or from butyl Mg iodide &
dimethylsulfate in ether (Refs 1 & 14)

2,2-Dimethyl-1-nitro-propane or Nitrope-
ntane, \((\text{O}_2\text{N})\cdot\text{CH}_2\cdot\text{C}-(\text{CH}_3)\cdot\text{C}-(\text{CH}_3)\cdot\text{CH}_3\); mw 117.15,
N 11.96%; liq, fr p 13-14\(^\circ\), bp 146-51\(^\circ\),
d 0.9494 at 20\(^\circ\), n\(_D\) 1.4099 at 30\(^\circ\); obtd with other products by treating 1-nitro-2-methyl-
1-propane with methyl-Mg-iodide in ether at
0-10\(^\circ\); by action of NaOH on 3-nitro-2,2,4-
trimethyl-4-pentanol, or by nitration of neo-
pentane at 410\(^\circ\) in the vapor phase (Refs 2,
6 & 7)

2,2-Dimethyl-1,3-dinitro-propane or Dinitro-
neopentane, \((\text{O}_2\text{N})\cdot\text{CH}_2\cdot\text{C}-(\text{CH}_3)\cdot\text{C}-(\text{CH}_3)\cdot\text{NO}_2\); mw 162.15, N 17.28\%, OB to CO\(_2\) minus 109%
crysts, mp 93-94\(^\circ\), bp 120\(^\circ\) 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\(^\circ\)

**Heat of Formation** – 72.0 kcal/mol

**Hygroscopicity** – no gain of wt at 25\(^\circ\)
& 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\(^\circ\) – not acid and no
expln after 300 mins

(See also Refs 3, 9, 11, 12 & 13)

2,2-Dimethyl-1,1,1-trinitropropane or Tri-
nitroneopentene, \((\text{O}_2\text{N})\cdot\text{HC}-(\text{CH}_3)\cdot\text{C}-(\text{CH}_3)\cdot\text{CH}_3\); mw
207.15, N 20.29\%; solid, mp 139-41\(^\circ\), burned
rapidly but did not detonate from hammer blow.
It was prepd in the lab of US Rubber Co
Pasaic, NJ (Ref 10) from nitroform and iso-
butylene. 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,
\((\text{O}_2\text{N})\cdot\text{HC}-(\text{CH}_3)\cdot\text{CH}_3\cdot\text{NO}_2\); crysts from
(from eth + petr eth), mp 121-22\(^\circ\); this compd
and other polynitrocompds were prepd by
nitration of mono- & dinitro- compds with
nitrite ions (from sodium nitrite) & Ag nit-
trate in the presence of alk or neutral aque-
ous media (Refs 16 & 17). Its expl props
were not reported

**Dimethylpropanediol.** See Di(methylol)-propane

**Dimethylpropyleneimidae and Derivatives**

2,2-Dimethyl-propylene-1,3-diamine or 2,2-Dime-thyl-1,3-diaminopropane,

\[ \text{H}_3\text{C}-\text{COCH}_3 + 2\text{CH}_3\text{NO}_2 \rightarrow (\text{H}_3\text{C})_2\text{C}(\text{CH}_2\text{NO}_2)_2 \]

**Blomquist & Fiedorek** (Ref 7) did not consider the method as promising and proposed the synthesis represented by the following equation:

\[
\begin{align*}
(\text{H}_3\text{C})_2\text{CHCHO} + \text{CH}_3\text{CHO} & \rightarrow (\text{H}_3\text{C})_2\text{C}(\text{CH}_2\text{OH})_2 \\
(\text{H}_3\text{C})_2\text{C}(\text{CH}_2\text{Br})_2 & \rightarrow (\text{H}_3\text{C})_2\text{C}(\text{CH}_2\text{NH})_2 \\
(\text{O}_2\text{N.HN})_2\text{H}_3\text{C}_2\text{C}(\text{CH}_2\text{NH})_2 \rightarrow (\text{NH}_3) \text{Cl}_2
\end{align*}
\]

**Pentaglycol Dinitramine,**

\[
\begin{align*}
(\text{O}_2\text{N.HN})_2\text{H}_3\text{C}_2\text{C}(\text{CH}_2\text{NH})_2 \rightarrow (\text{NH}_3) \text{Cl}_2
\end{align*}
\]

As the direct nitration of dimethylpropyleneimidae was not promising, Blomquist & Fiedorek (Ref 7) proposed the following reactions:

\[
\begin{align*}
(\text{H}_3\text{C})_2\text{C}(\text{CH}_2\text{NH})_2 & \rightarrow (\text{H}_3\text{C})_2\text{C}(\text{CH}_2\text{NH})_2 \\
(\text{H}_3\text{C})_2\text{C}(\text{CH}_2\text{NH})_2 & \rightarrow (\text{H}_3\text{C})_2\text{C}(\text{CH}_2\text{NH})_2 \\
(\text{H}_3\text{C})_2\text{C}(\text{CH}_2\text{NH})_2 & \rightarrow (\text{H}_3\text{C})_2\text{C}(\text{CH}_2\text{NH})_2
\end{align*}
\]

It is an explosive, but its props have not been reported in the literature

**2,3-Dimethylpropylene-1,3-diamo-diperchlorate,**

\[
\begin{align*}
\text{O}_4\text{ClH.H}_3\text{N.CH} \cdots \text{CH} \cdots \text{CH}_2\text{NH}_2 \text{HClO}_4
\end{align*}
\]

**Meyersberg** (Ref 4) prep'd it in small yield by condensation of acetone & Nitromethane, followed by reduction of resulting dinitrodimethylpropane; and Haas (Ref 5) outlined this procedure as:

\[
\begin{align*}
\text{H}_3\text{C}.\text{COCH}_3 + 2\text{CH}_3\text{NO}_2 \rightarrow (\text{H}_3\text{C})_2\text{C}(\text{CH}_2\text{NO}_2)_2
\end{align*}
\]

It is an explosive which cannot be satisfactorily detonated by MF (Ref 6)

**Ref:s:**

1) Beil, not found (Dimethylpropyleneimidae) 2) Beil, not found (Dimethylpropylenedinitramine) 3) Beil, not found (Dimethylpropylenediamino-diperoxide)


Di(methylpropyl)-maleate or Bis(methylpropyl)

maleate and Derivatives

2,2-Di(methylpropyl)-maleate (called Diisobu
ylmalenein in Ger),

\[ \text{H}_2\text{C} \cdot \text{COO} \cdot \text{CH}_2 \cdot \text{CH} \cdot (\text{CH}_3)_2 \]

\[ \text{H}_2\text{C} \cdot \text{COO} \cdot \text{CH}_2 \cdot \text{CH} \cdot (\text{CH}_3)_2 \; \text{mw} = 228.28, \text{O} = 28.04\%;
\text{liq. bp} = 125.6^\circ \text{C at 6 mm press, d} = 0.9820 \text{ at 20}\%
\text{and} 0.9265 \text{ at 86.5}\%; \text{n} = 1.4454 \text{ at 20}\% \text{ was prepd by reac. of maleic acid, isobutyl alcohol & concd sulfuric acid in boiling benz}
\text{(Ref 1)}

Di(2-nitro-2-methyl)-maleate,

\[ \text{H}_2\text{C} \cdot \text{CH}_2 \cdot \text{C} \cdot (\text{NO}_2) \cdot (\text{CH}_3)_2 \]

\[ \text{H}_2\text{C} \cdot \text{CH}_2 \cdot \text{C} \cdot (\text{NO}_2) \cdot (\text{CH}_3)_2 \; \text{mw} = 318.28, \text{N} = 8.80\%; \text{crystals (from alc), mp} = 75.5-79\% \text{ in benz, toluene, dioxane & acetone; insol in w & benz. 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-Tech Rept of Div 8, NDRC, Vol 1 (1946), p 119

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-butyne-2-ol in CA; and Dimethyl-acetylenecarbimol or Dimethyl-ethylcarbinol in Ger,\n
\[ \text{H}_2\text{C} \cdot \text{C} \cdot (\text{CH}_2)_2 \cdot \text{OH} \; \text{mw} = 84.11, \text{O} = 19.02\% \text{ very mobile liq of characteristic penetrating odor, fr} \text{ p} \text{ = } 3.01\existent}{, \text{bp} 104^\circ \text{C; d} = 0.8624 \text{ at } 20^\circ \text{C, n} = 1.4212 \text{ at } 20^\circ \text{C; 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 crystals; Mercury salt, plts, dec >240\% and Silver salt, voluminous pp, 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-butyne-2-ol in CA, HC:CC(CH)_3_2:ONO; \text{mw} = 113.11, N = 12.38\%; liq, bp 80-82\% at 750mm press; n = 1.3926 at 20\%; was prepd when NOCl & ether were added to a stirred soln of 3-methyl-1-butyne-3-ol (parent compd) & C_5H_4N at 20-30\% This compd was patented as a propellant fuel and for other uses (Ref 3)


Dimethylpyrazole and Derivatives

3,5-Dimethylpyrazole,

\[ \text{H}_2\text{C} \cdot \text{C} \cdot \text{NH} \cdot \text{N} \]

\[ \text{H}_2\text{C} \cdot \text{C} \cdot \text{NH} \cdot \text{N} \; \text{mw} = 96.13, \text{N} = 29.14\% \text{ plates, sp gr 0.864 at } 20^\circ/4\%
\text{mp} 106-107\%
\text{bp} 218^\circ \text{at 759mm; sol in w, alc, eth, benz & chll; can be prepd by heating 3,5-dimethylpyrazole-carboxylic acid-1-
nilide with water or alc, and by other methods (Ref 1)}

3,5-Dimethyl-4-azido-pyrazole,

\[ \text{H}_2\text{C} \cdot \text{C} \cdot \text{NH} \cdot \text{N} \]

\[ \text{N} \cdot \text{C} \cdot \text{CH}_3 \; \text{mw} = 137.15, \text{N} = 51.07\% \text{ lfts (from benz & petr eth); mp 81^\circ \text{dec); bp - expl at higher temp; dec explosively on contact with concd sulfuric acid; easily sol in org solvants. It can be prepd from 3,5-dimethylpyrazole-4-diazonium chloride and Na nitrite in acid soln (Refs 2 & 3)}

3,5-Dimethyl-4-nitro-pyrazole,

\[ \text{H}_2\text{C} \cdot \text{C} \cdot \text{NH} \cdot \text{N} \]

\[ \text{O}_2\text{N} \cdot \text{C} \cdot \text{CH}_3 \; \text{mw} = 141.13, \text{N} = 29.87\% \text{ crystals, mp 124-128\%; bp 325^\circ \text{at 749mm press; readily sol in alc,}
eth & warm water; prepd by nitrating 3,5-
dimethylpyrazole with mixed nitric and sul-
furic acids, and by other methods (Refs 3
& 5). This compd dec on heating at high temp
3,5-Dimethyl-1,4-dinitropyrazole,
(H₃C)C—N(NO₂)—N
\[(O₂N)C———C(CH₃)\]; mw 186.13,
N 30.10%; crystals, mp 66–67⁰; was obtd to-
gether with 3,5-dimethyl-1-nitropyrazole when
3,5-dimethylpyrazole in acetic acid was ni-
trated 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) (Dimethyl-
pyrazole) 2) Beil 23, (25) (Azidomethyl-
pyrazole) 3) Beil 23, (25) & (691)
(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) (Dinitrodimethylpy-
razole)

2,4-Dimethylpyridine or α,γ-Lutidine and Derivatives

2,4-Dimethylpyridine,
HC—N———C(CH₃)
\[(HC₃C)C———C\]; mw 107.15, N
13.07%; liq, sp gr 0.938 at 0⁰; 0.9331 at
20⁰; bp 157.3⁰; sol in w, alc or eth; can be
prep'd by the dry distillation of binisourn
coal and by other methods; forms crys compds
with mineral acids (Refs 1 & 4)

2,4-Dimethylpyridino-diazido-copper (II)
Complex, [CuC₃H₃N(N₃)]₂; mw 278.76, N
35.18%; solid, mp — exp liq at 208–209⁰, with
a loud report, on preheated block; exp liq under
light blow; was prepd by Cirulis & Straumanis
(Ref 3) by dissolving Cu(N₃)₂ in 2,4-dimethyl-
pyridine and add aq acid followed by MeOH
or ether to ppt the product, or by addg NaN₃
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-bm 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, (H₃C)C—NH———CO
HC—C(CH₃)——CH; mw 123.15,
N 11.37%; ndls (from alc), mp 180–81⁰, bp
305–307⁰; sol in alc; sl sol in w & chl;
v sl sol in petr eth, eth & benz; was prepd
by heating 6-hydroxy-2,4-dimethyl-5-cyan-
pyridine with concd HCl and by other methods
(Ref 1)

4,6-Dimethyl-3,5-dinitro-2(1H)-pyridone,
(H₃C)C—NH———CO
\[(O₂N)C———C(CH₃)———C(NO₂); mw 213.15, N
19.72%; yel crys (from 95% alc), mp 238⁰
(dec), darkens 10⁰ before mp & finally dec
to a black liq; was prepd by addg a mix of
acetic anhydride & fuming nitric acid at 0⁰
to 4,6-dinitro-5-nitro-2-pyridinol,
\[(H₃C)C——N———COH
\[(O₂N)C———C(CH₃)———CH\] in Ac₂O at -5⁰
for 10 sec, stirring, allowing the temp to
rise to 40⁰, addg cracked ice to ppt the pro-
duct (Ref 2)

Heating the dinitro deriv with PCl₅ for
4 hrs at 180⁰ and pouring onto ice gives
4,6-Dimethyl-3,5-dinitro-2-chloropyridine,
mp 84⁰ & distilling at 120cc/min (Ref 2)

No expl props of the compds are re-
ported
Refs: 1) Beil 21, 51 (204) & (36)
2) R.P. Mariella, JOrgChem 20, 1726 (1955)

2,6-Dimethyl)-4-pyrones,
HC—CO———CH
\[(H₃C)C———O———C(CH₃); mw 124.13; wh
crys, mp 132⁰, bp 248–50⁰; was prepd by
Collie & Tickle (Refs 1 & 2) by rapidly
heating diaacetylaceetone with PCl₅ &
HCl on a steam bath
It was tried in France before WWII (Ref 3) as a possible gelatinizer for NC in prep 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,  
\[ (\text{C}_6\text{H}_{11}\text{O}_2)\text{ClO}_4 \cdot \text{mw} 238.55; \text{crysts, mp} \]  
\[ 190^\circ; \text{sl sol in aic & acet}; v \text{sl sol in cold w}; \text{was prep by Piccard by warming methylpyrone with dimethyl sulfate \& MeOH at 50}^\circ, \text{cooling \& treating with 20}\% \text{HClO}_4 \]  
It was suggested as a component in flash compn for blasting caps

**Refs:** 1) Beil 17, (133)  
2) J. Piccard, USP 1964077 (1934) \& CA 28, 5242(1934)

2,4-Dimethylpyrrole-3,5-dicarbonyl Azide,  
\[ \text{N}_3\cdot\text{OC} \cdot \text{C} \cdot \text{NH} \cdot \text{C} \cdot (\text{CH}_3) \]  
\[ (\text{H}_3\text{C})\text{C} \cdot \text{C} \cdot \text{CO} \cdot \text{N}_3 \cdot \text{mg} 233.20, \text{N} \]  
\[ 42.05\%; \text{solid, mp – dec explosively at 119^\circ}; \text{was prep by treating the hydrazine deriv of 2,4-dimethyl-3,5-dicarboxylic acid with HCl \& sodium nitrite (Ref 2)} \]  

**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),  
\[ (\text{CH}_3)_2\text{O} \cdot (\text{COOCH}_3)_2 \cdot \text{mw} 230.30; \text{ndls or plts, sp gr 0.9896 at 20/20, n_D 1.4376 at 20^\circ}; \text{mp} \]  
\[ 38^\circ (26^\circ) \text{(Ref 1)}; 24.5^\circ \text{(Ref 4)}; \text{bp} \]  
\[ 293-294^\circ \text{at 754mm; flash point } 145^\circ (293^\circ F); \text{sol in aic, benz, eth, ligroin; heat of combustion } 16.4\text{kcal/mol at C_T. It can be prep by heating sebacic acid (CH}_3)_2\text{O} \cdot \text{COOH}_2 \text{with methanol and hydrochloric acid. It has been used as a plasticizer for NC in proplnts, as for example Unit No 398 listed in Ref 5. Davis (Ref 3) stated that for complete gelatinization of 100 parts of Pyrocellulose are required 80 parts of DMeSeb if in alcoholic soln, 70 in benzene and 105 in lignin. Note: There exists also a monomethyl-sebacate listed in Beil 2, [608] \]  

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) Cond-ChemDict (1961), 399-L  
5) Proplnt Manual SP1A/M2 (1962) (Conf)

**Dimethylsulfamide or Dimethylsulfonamide and Derivatives**

\[ \text{N,N'-Dimethylsulfamide,} \]  
\[ \text{H}_3\text{C-} \text{NH}_2\text{SO}_2-\text{NH}_2\text{-CH}_3; \text{mw} 124.16, \text{N} 22.56%; \text{mp} 77.6^\circ \text{(Grasselli). It was first prep by} \]  

Franchimont (Refs 1 \& 3) by interaction of sulfuryl chloride with methylene. Shriner dissolved them in chl (Ref 7)

\[ \text{N,N'-Dinitro-}N,N'-\text{dimethylsulfamide or} \]  
\[ \text{N,N'-Dinitro-}N,N'-\text{dimethylsulfonamide,} \]  
\[ \text{H}_3\text{C} \cdot \text{N(NO}_2)_2 \cdot \text{SO}_2 \cdot \text{N(NO}_2)_2 \cdot \text{-CH}_3; \text{mw} 214.16, \text{N} 26.16%; \text{Ob to CO} \text{ \& SO}_2 \text{ minus } 21.4\%; \text{crysts (from benz); mp } 88.5-90^\circ; \text{bp ca } \]  
\[ 160^\circ \text{and its vapor expl above } 160^\circ \text{(Franchimont); accdg to Aarsonon (Ref 6) it decompd at } 170^\circ \text{ (in 5 sec) and detonated at } 180^\circ, \text{while below } 180^\circ \text{ only light grey smoke was observed. Accdg to Shriner (Ref 7) it decompd at } 135^\circ \text{ in 8 mins. It is sl sol in w; very sol in benz, chl \& hot alc; sol in eth \& petr eth; was first prep by} \]  

Franchimont (Ref 3) by nitrating of dimethylsulfamide with 98% nitric acid. Aarsonon prep 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 nitrating 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 mix filtered thru a Büchner. After evaporating the filtrate to a small volume, the crop of wh, lustrous crystals (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 recrystalized from a mix 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 conc 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 chased by 0.2g Tetryl
Heat of Combustion - 472.1 kcal/mol (Ref 5)
Hygroscopicity - nil
Impact Sensitivity (by BusMinesMachine) 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 decomp temp (170°). He recommends instead to evaluate the properties of its higher homologue, N,N'-Dimethyln,N'-diethyl-sulfamide (See under Dinitro compounds in this Vol)

Naomé (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 1, 289, [277] & [1217] 2) Beil 4, 86 (Dinitrodimethyl-sulfamide) 3) A.P. Franchimont, Rec 3, 419 (1884) 4) P. Naomé (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), pp 10 & 14

**Dimethylsulfate** or **Methylsulfate**, (CH₃)₂SO₄; 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 prep’d by adding fuming sulfuric acid to ethanol and distilling in vacuo

It is a blister gas known in France before WWII as **Rationite** and in Ger as **D-Stoß** (Refs 2 & 4)


**N,N'-Dimethylsulfonamide.** See **N,N'-Dimethylsulfamide** in this Vol

**Dimethylsulfoxide (DMSO)**, (CH₃)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 prep’d by the liquid phase oxidation of dimethylsulfide using nitrogen oxide or concd nitric acid (Refs 1 & 2)

An exln can be produced on contact of DMSO with 70% perchloric acid as was reported by Kharasch (Ref 3). Vacuum dist’ 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 exln, 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 comds. 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

N,N'-Dimethyltartramide or N,N'-Dimethyl-diaminotartaric Acid and Derivatives

H₃C.H₂.NH.CH(OH).CH.OH·CO·NH·CH₃;
mw 176.17, N 15.89% cryps, mp 189°C
(Ref 3); sol in warm alc, w & pyridine, insol in cold alc; was prep'd by Frankland & Slator (Ref 3) by introducing methylamine into a soln of methyl-d-tartarate in MeOH and cooling.

Urbanski (Ref 4) also prep'd N,N'-Dimethyl-diaminotartaric Acid, but its mp 213–214°C did not agree with the mp of the compd prep'd by Frankland & Slator. Urbanski also prep'd N-Methylaminotartaric Acid and its mp was reported as 198°C (Ref 1).

N,N'-Dimethyl-N,N'-dinitro-tartramide Dinitrate; 1,6-Dimethyl-3,4-dinitroxy-tartor-1,6-dinitramide or 1,6-Dimethyl-dinitramino-3,4-dinitroxy-tartaric Acid;

H₃C·(O₂N)·N·OC·CH(OH)·CH·(ONO₂)·CO·N·(ONO₂)·CH₃; mw 536.17, N 23.60%, OB to CO₂ minus 18%; cryps, mp 114°C; bp expl at 117°C when heated at the rate of 20°C per minute; sol in acet, benz, chlf, CCl₄, and alc. It can be prep'd by dissolving N,N'-dimethyl-tartramide in acet at below 0°C and adding to it concd nitric acid, followed by acetic anhydride.

It is a very powerful expl with sensibility to shock between that of MF & LA and stable below 110°C; was proposed as a component in primer mixtures.


1,5-Dimethyl-1,2,3,4-tetrazole or 1,5-Dimethyl-1H-tetrazole;

(H₃C)C—N(CH₃)—N

N

H₂C—NH—CH₂—N(CH₃)
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-tetraazacyclooctane, (H₃C)N—CH₂—N(CH₃)

H₂C—NH—CH₂—N(CH₃)
mw 234.22, N 33.88%; ndls (from acet), mp 124°C & 154–155°C (Ref 6); sol in acet, alc, chlf & 10% eq NaOH; insol in eth & petr eth; was obtd by Chute et al (Ref 4) by condensing MeNH₂ with a formalin-nitramide soln. Chapman et al (Ref 6) also prep'd this compd (mp 125–26°C, dec) when a soln of methylene dinitramine in ethyl acetate was saturated with dry gaseous formaldehyde at 0°C and a 33% methylamine in alc was added. See also Ref 5.

1,5-Dimethyl-1,2,3,4-tetrazole or 1,5-Dimethyl-1H-tetrazole,

(H₃C)C—N(CH₃)—N

N

N

H₂C—NH—CH₂—N(CH₃)
mw 98.11, N 57.11%; cryps (from petr eth), mp 71–74°C; was obtd when acetone was slowly added to a mix of hydrazoic acid, benz & sulfuric acid (Ref 1). This compd was also prep'd by Harvill et al (Ref 2). McEwan & Rigg deter its Qcomb sm as 532.19kcal/mol & calculated its Qformation as 45.08kcal/mol at 25°C. Its X-ray diffraction pattern is also reported.

Di[1-methyl-tetrazoly-5]-diazene; 1,1-Dimethyl-5,5'-azotetrazole or Azido-di(1-methyltetrazole-5),
N,N(CH₃)₂.C——N.N——C.N(CH₃)₂.N
N——N   N——N;
N; mw 194.17, N 72.14%; orn-yel crysts, mp 182°C (dec); bp pulls off with a loud report on rapid heating; easily sol in hot w; sl sol in hot alc; can be prep'd by shaking and then warming on a water bath, the 1-methyl-5-amino-tetrazole, H₂N.C——N(CH₃)₂——N
N——N; with an aq soln of Ca hypochlorite.
Ref's: 1) Beil, not found 2) R. Stolle, J Prakt.Chem 134, 284 & 287 (1932) & CA 26, 5565 (1932)

Dimethyltoluidines and Derivatives
N,N-Dimethyl-m-toluidine,
H₃.C₆.H₄.N(CH₃)₂; mw 135.20, N 10.36%; yel oil, sp gr 0.941 at 20/4; bp 213–215°C; insol in w; sol in alc & eth; was prep'd by van Romburch (Ref 3) by heating for 24 hrs m-toluidine, methyl alcohol & HCl at 200°C. Its nitration did not produce the expected "Dinitrodimethyltoluidine", but the following compd:
2,4,6-Trinitro-3-methyl-nitraminotoluene, code named MethyTetryl (USA) or MethyICE (Gt Britain),
H₃.C₆.H₄.N(CH₃)₂.N(NO₂)
O₂.N.C——C——C(NO₂)
H.C——C(NO₂)——N(CH₃)
₃   2   1
₆   5   4
mw 301.18, N 23.26%, OB to CO₂ minus 61.1%; pale yel crysts, mp 100–102°C; expl at higher temp. It was first obt'd in 1884 by van Romburch (Ref 3), instead of expected Dinitrodimehtyltoluidine, on treating dimethyltoluidine with mixed nitric-sulfuric acid. Its structure was not established until 1902 when Blanksma (Ref 4) prep'd a compd identical to van Romburch compd from 2,4,6-Trinitro-methylaminotoluene. It was also prep'd by Garner & Abemethy (Ref 5). Davis (Ref 6) and Clift & Fedoroff (Ref 7) described prep of MeTetryl from beta- and gamma-Trinitrotoluene isomers always present as impurities on the surface of crystals of crude alpha-TNT. The β and γ isomers can be removed by washing the crystals 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:
(O₂N)₂C₆.H₄——CH₃
SO₂ONa + CH₃.NH₂ —> 
O₂N)₂C₆.H₄——NHCH₃

Nitration of this product with mixed nitric-sulfuric acid gives Methyltetryl,
(O₂N)₂C₆.H₄——CH₃
N,N-Dimethyl-dinitro-m-toluidine,
H₃.C₆.H₄(NO₂)₂.N(CH₃)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 crystal granules, mp 168°C; was prep'd from

Heat of Combustion: 1009.3 kcal/mol
Impact Sensitivity expressed as Fl 67% of Picric Acid
Power by Ballistic Mortar 110% TNT and by Trauzl Test 103% PA
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 prepbd 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


N,N-Dimethylos-toluidine,
H₃C₆H₄N(CH₃)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 prepbd 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,
H₃C₆H₄(NO₂)₂,N(CH₃)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, H₃C₆H₄,N(CH₃)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 prepbd by reaction of methylchloride on hot p-toluidine & methyl-p-toluidine, and by several other methods (Ref 1)

N,N-Dimethyl-dinitro-p-toluidine,
H₃C₆H₄(NO₂)₂,N(CH₃)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 pltls (from alc), mp 103–04°; readily sol in eth, chl, ethyl acetate, hot alc & hot petr eth; sl sol in cold alc & cold petr eth; was prepbd by nitrating 2-nitro-4-dimethylaminotoluene with a soln of nitric-sulfuric acid and pouring intro w (Ref 2, pp 1009 & 442)

N,N-Dimethyl-2,6-dinitro-p-toluidine, yel ndls (from acet ac), mp 192°; was prepbd by heating 2,6-dinitro-4-aminotoluene with dimethysulfate 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 prepbd 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]

N,N-Dimethyl-2,4,6-trinitro-p-toluidine
(called 2,3,6-Trinitro-4-dimethylaminotoluol in Ger),
O₂N,C—C(CH₃) == C.NO₂
\[\text{HC—C} \quad \text{== C.NO₂} \]
\[\text{N(CH₃)₂} \]
mw 272.0, N 20.74%; red ndls or tablets (from alc), mp 137°; was prepbd 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


N,N-Dimethyl-tetranitro-p-toluidine,
C₃H₃N₆O₈; not found in the literature thru 1966

N,N'-Dimethyltriazen; 1,3-Dimethyltriazen or Diazooaminomethane, H₃C₅N₆NH.CH₃;
mw 73.10, N 57.49%; col liquid, fr p minus 12°; bp 92° (with decomp) and exploded with a sharp report when heated in a capillary tube; sol in w, imparting a sweet taste
to it; was prep'd by treating methylazide with methyl-magnesium iodide:

$$\text{CH}_3\text{MgI} + \text{N}_3\text{CH}_3 \rightarrow \text{CH}_3\text{-N:}\text{N(Mgl)}\text{-CH}_3$$

and then decomposing the resulting product with water:

$$\text{CH}_3\text{-N:}\text{N(Mgl)}\text{-CH}_3 + \text{H}_2\text{O} \rightarrow \text{CH}_3\text{-N:}\text{NH.CH}_3$$

Several salts were prep'd by Dimroth (Ref 2), such as the copper, CuC_2H_4N_3, mp 185586° and the silver, AgC_2H_4N_3, mp – decmp 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 comds or with their addn complexes, heating above 110° is to be carefully avoided. Their prep & props are described in great detail (Ref 4).


**Di(methyltriazolyl)-formamide.** See N,N'-Bist(methyl-s-triazol-3-y1)-formamide in Vol 2 of Encycl, p B151-L.

**Dimethylthiethylentetramine and Derivatives 1,10 or N,N'-Dimethylthiethylentetramine,\)**

$$\text{H}_3\text{C.NH.CH}_2\text{NH.CH}_2\text{NH.CH}_2\text{NH.CH}_2\text{NH.CH}_3; \text{mw} 174.29, \text{N} 32.15\%; \text{may be considered as the parent compd of its nitrated衍生s, although not used to prep them. It is a liq of bp 152° at 17mm press and np 1.4718 at 25° (Ref 13). Prep is given in Ref 14.**

**N,N'-Dimethylthiethylene-tetranitramine or 1,10-Dimethyl-1,4,7,10-tetranitro-thiethylentetramine,\)**

$$\text{H}_3\text{C.N(NO}_2\text{)CH}_2\text{N(NO}_2\text{)CH}_2\text{N(NO}_2\text{)CH}_2\text{N(NO}_2\text{)CH}_3; \text{mw} 354.29, \text{N} 31.63\%; \text{OB to CO}_2 \text{minus 76.8%}. \text{Crysts, mp 211.5-213°(decomp); bp – did not boil and did not expl on being heated to 360°, was prep'd by Blomquist & Fiedorek (See Ref 5) by refluxing a mixture of N-methyl-ethylenedinitramine, (CH}_3\text{O(NO}_2\text{)N.CH}_2\text{CH}_2\text{N(NO}_2\text{) with ethylene dibromide, Br.CH}_2\text{CH}_2\text{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.N:N.CH_2.CH_2.N(NO_2).CH_3, \{\}

$$\text{O}

mw 354.29, \text{N} 31.63\%; \text{OB to CO}_2 \text{minus 76.8%}; \text{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 obtld:

<table>
<thead>
<tr>
<th>Compound</th>
<th>mp or n</th>
<th>Analyses</th>
<th>Data Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-Methyl-N-(2-nitroethy1)-ethylenedinitramine</td>
<td>165.11</td>
<td>N 25.45%, OB to CO₂ minus 43.6%</td>
<td>Ref 1</td>
</tr>
<tr>
<td>2-(Nitroethy1)-methylene nitramine, called M-NEN</td>
<td>165.11</td>
<td>N 25.45%, OB to CO₂ minus 43.6%</td>
<td>Ref 1</td>
</tr>
<tr>
<td>(O₂NO)CH₂.CH₂.N(NO₂)CH₃</td>
<td>165.11</td>
<td>N 25.45%, OB to CO₂ minus 43.6%</td>
<td>Ref 1</td>
</tr>
<tr>
<td><em>Note:</em>* When compd of mp 88.5-90° was treated with 98% nitric acid, a mixture of the following products was obtld:</td>
<td>165.11</td>
<td>N 25.45%, OB to CO₂ minus 43.6%</td>
<td>Ref 1</td>
</tr>
</tbody>
</table>
mp 39-40.5°. Besides obtg it by the method of Blomquist & Fiedorek outlined in Ref 3, it could be prep'd by treating N-methyl-
ethanolamine, \( H_2C.NH.CH_2.CH_2O.H \), with nitric acid, acetic anhydride and Zn chloride, as described in NDRC Div 8, IntRepts PT-5, Dec 15, 1942 to Jan 15, 1943, p25. 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 Bruceon 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 (Dimethyltrichloro-
tetramine) 2) Beil, not found (Dimethyltri-
bis(α-methylaminoacetamide) with LiAlH₄ in anhyd tetrahydrofuran)

**Dimethyleurea and Derivatives**

**N,N'-Dimethyleurea**; **sym-Dimethyleurea** or **1,3-Dimethyleurea**, \( H_2C.NH.CO.NH.CH_2.CH_2O.H \), 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 prep'd by heating urea with met hylamino hydrochloride at 160-170° and by other methods (Ref 1)

Attempts by Shriner (Ref 3) to use it as an ingredient of expls and proplns 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,

\[ H_2C.N(NO_2)_2.CO.N(NO_2)_2.CH_2.CH_2O.H \]

mw 162.11, N 34.56%; liq, cap sensitive but insensitive to shock & friction; was prep'd by nitrating the parent compd below 30° with concd nitric acid or with a mixt of HNO₃ & Ac₂O

Its expln compns consist of: AN 60, NH₄ClO₄ 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 1 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'-diphenyl-
urea, called Centralite 2, described in Vol 2 of Encycl, pC137-1)


**N,N-Dimethyl-xenylamine or N,N-Dimethyl-
p-biphenylamine**. See N,N-Dimethylaminobiphenyl in this Vol, pD1308-R

**Dim Igniters**. See under Dark and Dim Ig-

iters in Vol 3 of Encycl, pD16-L.
Diminution of Sensitivity of Dynamites.
See under DYNAMITES in this Vol, p

Dina. Ger designation for Dinitronaphthalene. It was used during WWII, together with Tri-
nitronaphthaline at Semtin Fabrik, Pardubice
(Czechoslovakia) and used in some composite
explosives
Ref: Fedoroff et al, PATR 2510(1958),
pp Ger 36 & 37

DINA. Code name for Diethyolyt-nitramine
Dinitrate or Di(nitroxyethyl)-nitramine, de-
scribed under Diethyloxyamine in this Vol
p D1240-R

Dinafitaliti. Russian nonpermisible exps, such as
Dinafitalit Zernnnymi (Granular
Naphthalite) listed in Vol 3 of Encycl, p
C441-R, under COAL MINING EXPLOSIVES
and in Gorst (1957), p104

Dinamitas. Spanish Dynamites. Nonpermis-
sible types (Explosivos de no seguridad)
are listed in Vol 3, pp C441-R & C442-L.
Permissible types (Explosivos antgrisú)
are listed on pp C445-R & C455-L

Dinamiti. Italian Dynamites. Nonpermis-
sible types are listed in Vol 3, p C440-L,
and permissible types on p C451-L. See also
Giua, Trattato 6(1)(1959), 338–45

Dynamity, Nepredokhranitel'niy. Russian
nonpermisble Dynamites are listed in Vol
3, pC441-R

Dynamity Predokhranitel'niy. Russian
permisible Dynamates are listed in Vol
3, pC454-R

Dynamoni 1º and 5. Italian nonpermisible
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 Belgano (1952), p153 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

Dynamony. Russian nonpermisible explo-
sives of which four formulations are listed
in Vol 3, pC441-R and several other formu-
lations are given in this Vol, under DYNAM-
MONS

Dimaphylylamine and Derivatives
Dimaphthylamine (β,β'), C18H11NH,C1H7;
mw 269.33, N 5.20%; ltrs (from benz), mp
171–72º, bp 471º. 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'-dimaphthylamine,
C20H11N(NO2)4; mw 449.33, N 15.59%; yel
cryst granules (from hot Nitrobenz), mp
285–86º; v sl sol in benz, cumol, alc &
eth; sol in hot nitrobenzene; was prepd by
nitrating the parent compd in acetic acid with
conc acid (Ref 2). No expl props are
reported

Hexanitro-β,β'-dimaphthylamine,
C16H12(NO2)3.C1H7; 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

Ref: 1) Beil 12, 1278, (536) & (717)
2) Beil 12, 1279 (Tetranitro) 3) Beil 12,
1279 (Hexanitro)

Dimaphthyazo-tetrazole and Derivatives
1,1-Di(α-naphthyl)-5',5'-azotetrazole or
Di[1-α-naphthyl]-tetrazole-5]-diazene,
N–N(α-C10H7)–C–C=N:¢–N–N(α-C10H7)–N;
N–N–N–N)

N mw 418.42, N 33.48%; red ndls, mp – puffs
off at ca 180º; insol in w; sl sol in acet;
fairely sol in AcOH; v sl sol in alc. & eth;
can be prepd by warming an aqueous sus-
pension of α-naphthyl-1-amino-5-tetrazole
with a conc soln of Ca hypochlorite. It is
considered as a mild explosive (Refs
3 & 4)
1'-1-Di-(β-naphthyl)-5,5'-azotetrazole or
D\(_{\text{II}}\)-(β-naphthyl)-tetrazole-5)-diazine,
\[
\text{N} \rightarrow \text{N}(-\text{C} \rightarrow \text{H}) \rightarrow \text{C} \rightarrow \text{N}(-\text{N} \rightarrow \text{C} \rightarrow \text{N}(-\text{C} \rightarrow \text{H}) \rightarrow \text{N}
\]
mw 418.42; N 33.48%; red ndls, mp - explodes at 204°C; 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 prep'd by warming an aqueous suspension of β-naphthyl-1-amine-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.....)

Dinaphthyl-succinamide. See Bis(α-naphthyl) -succinamide in Vol 2, pB151-R

1,3-Di-1-naphthyltriazene. See Diaoamino-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 propelt rocket and is capable of carrying a nuclear warhead
Ref: Glossary of Ord(1959), pp96-R & 135-R

Dicnamic Acid and Derivatives

Dicnamic Acid or 3,5-Pyridinedicarboxylic Acid, NC\(_6\)H\(_4\)(COOH)\(_2\); mw 167.12, N 8.38%; col crysrs, mp 314°C(Hackh's); 323°C(decomp); bp - subl with decomp; v sl sol in w, alc or eth; sol in HCl). Prep'n & other props in Beil (Ref 1)

Dicnamic Acid Dizaide (Dinikotinsäurediaziid in Ger), NC\(_6\)H\(_4\)(CO.N\(_2\))\(_2\); mw 217.15, N 45.16%; pIIts (from eth), mp 83°C, bp - explodes on rapidly heating; was obt'd 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)

Dinitro 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, PicArms; 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-L

Di(β-nitraminoethyl) -amine. See Bis(β-nitraminoethyl)-amine or 1,7-Dinitro-1,4,7-triazahexane 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'-dinitrorea. See N,N'-Bis(β-nitraminoethyl) -N,N'- dinitrorea 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-propanedinitra-
mine. 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-benz-
aldehydedeazaine. See Vol 2, pB36-L
4,6-Dinitranino-s-triazine-2-ol. See Dinitroammeline in Vol 1 of Encycl, pA274-L

1-(2,4-Dinitranilino)-benzene-4'-diazonium Nitro. See Vol 1, pA421-R

3,3-Di(nitratotetethyl)-butene-1. See under Dimethyl-butene in this Vol, pD1331-L

3,3-Di(or Bis)-(nitratotetethyl)-oxetane or 3,3-Oxetanediemethanol Dinitrotete

\[
\begin{align*}
\text{O} & \quad \text{CH}_2 \\
\text{CH}_2 & \quad \text{CH}_2\text{ONO}_2 \\
\text{CH}_2 & \quad \text{CH}_2\text{ONO}_2
\end{align*}
\]

mw 208.13, N 13.46%; wh cake, mp 88-90°; d 1.76, ignition temp 233°, thermal stability 1.12 cc gas/g at 100° in 48 hrs; impact sensitivity 3.4 cm with 2/3 kg wt; was prep'd when PETriN (Pentaerythritoltriminotetrate) in abs alc was heated with KOH at 73° for 4 1/2 hrs with stirring and the mix cooled to 0° (Ref 2)

See also Refs 3 & 4


1,2-Di(or Bis)-(2-nitrimino)-3-nitro-1-imidazolidinyl)-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 -NO₂, two -ONO₂ or two -NO groups. As a rule, they are described in this Encyclopedia under corresponding parent compds. For example, Dinitrotoluenes are described under Benzene and Dinitrobenzenes are described 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 di-hydroxy-dinitrates

Following list gives location of explosive dinitro-, dinitroso- and dinitroxy- compounds not found in this Volume. Some non-explosion compds, such as Dinitrotoluene, are included because they were proposed as ingredients of expl compositions:

Dinitroabiotic Acid. See Vol 1 of Encycl, p A3-L

Dinitrocetophenones. See Vol 1, p A48-L

1,6-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

Dinitroaminobenzenes. 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)-benzalamino- guanidinium-biguanidinium benzalhydrzone. See Benzalaminoguanidinium-1,6-dinitro-2-(aminoguanyl) -biguanidinium benzalhydrzone 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 Dinitroamino-diazacyclpentane. See 1-Nitro-2-nitramino-Δ2-imidazoline in Vol 1, p A220-L

5,7-Dinitro-6-amino-1-azacyclonaphthalene. 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-aminophenylarsionic 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-Dinitro-isopropylamine. 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

2,6(D, or 3,6)-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-diazonium Nitrate or 2',4'-Dinitrodiphenylamine-diazonium Nitrate. See Vol 1, p A421-R

2-(2',4'-Dinitroaniline)-1,3-propanediol. See Vol 1, p A435-L

2,4-Dinitroanisol. 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

Dinitroazoazobenzene. 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 Azoamidine (nitroformamidine) in Vol 1, p A652-L
Dinitroazonaphthalenes. See Vol 1, p A656

Dinitroazobenzene. See Vol 1, p A657-L

Dinitroazosulbenes or Dinitro-distyrylazo-benzene. See Vol 1, p A659

Dinitroazotoluenes or Dinitrodimethylazo-benzene. 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

Dinitroazobenzene. 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 Dinitrodimethylazoxybenzene. 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 α-) Dinitrobenzene. See Vol 2, p B46-L

1,3-(or κ-) Dinitrobenzene. See Vol 2, p B46-R

1,4-(or β-) Dinitrobenzene. See Vol 2, p B46-R

Dinitrobenzene, Commercial. See Vol 2, p B47-L

Dinitrobenzeneazonitronaphthal. See in Vol 2, p B54-L

3,5-Dinitrobenzenazo-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 Dino. See in Vol 2, p B59-R

2,6-Dinitrobenzene-4-diazo-1-oxide or 2,6-Dinitro-4-diazophenol. See Vol 2, p B59-R

2,4-Dinitrobenzenesulfoxamide. 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

Dinitrobenzadoxan. See in Vol 2, p B67-L

Dinitrobenzodioxan. 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 Hydroxide or Dinitrobenzoylhydroxide. 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 expts, such as RDX. Its addition to some high melting HE's lowered their mp's, thus rendering them suitable for case-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°C 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:

Density: 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-Nitrocyclohexyldene Ester. See 1,1'-Bis(p-nitrobenzoyl-peroxy)-cyclohexane in Vol 2, p B135-L.

2,5-Di(p-nitrobenzoperoxy)-2,5-dimethyl-hexane 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-benzquinone-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-oxadiazo. One of the names for Diazodinitrophenol, described in Vol 2, p B59-L as 2,6-Dinitrobenzene-2-diazio-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

Dinitrobenzyloxyde. 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
Dinitrobi(bicyclo)-1,5-ethylidene-2,4,6,8-tetramethylenone-1,3,5,7-tetramine; or 3,7-Dinitro-1,5-endothylidene-1,3,5,7-tetraazao-2,4,6,8-cyclooctane; CA name 3,7-Dinitro-1,3,5,7-tetraazabicyclo[3.3.1] nonane. See under 1,5-Endoethyldiene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane.

3,7-Dinitro(bicyclo)-2,4,6,8,9,10-hexamethylenone-1,3,5,7-tetramine or 3,7-Dinitro-1,5-endothylidene-1,3,5,7-tetraazao-2,4,6,8-cyclooctane. CA name 3,7-Dinitro-1,3,5,7-tetraazabicyclo[3.3.2] deca
cane (Code name homo-DPT). See under 1,5-Endoethyldiene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane.

1,6-Dinitrobouguanidine. 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

Dinitro(bis[3-nitrobenzylidenediamino]-guanidine.) See N,N'-Bis[3-nitrobenzylidenediamino]-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 CO2 -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-nitrobenzylidenediamino)-guanidine in Vol 2, p B136-L

Dinitrobi(bicyclo) 3,7-Dinitro(bicyclo)-1,5-ethylidene-2,4,6,8-tetramethylenone-1,3,5,7-tetramine; or 3,7-Dinitro-1,5-endothylidene-1,3,5,7-tetraazao-2,4,6,8-cyclooctane; CA name 3,7-Dinitro-1,3,5,7-tetraazabicyclo [3.3.1] nonane. See under 1,5-Endoethyldiene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane.

Dinitrobenzyls. See Vol 2, p B110

3,7-Dinitro(bicyclo)-1,5-ethylidene-2,4,6,8-tetramethylenone-1,3,5,7-tetramine; or 3,7-Dinitro-1,5-endothylidene-1,3,5,7-tetraazao-2,4,6,8-cyclooctane; CA name 3,7-Dinitro-1,3,5,7-tetraazabicyclo [3.3.2] deca
cane (Code name homo-DPT). See under 1,5-Endoethyldiene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane.

3,7-Dinitro(bicyclo)-2,4,6,8,9,10-hexamethylenone-1,3,5,7-tetramine; Code named DPT or DNPT (abbr for Dinitropentamethylethenetetramine); 3,7-Dinitro-1,5-endothylidene-1,3,5,7-tetraazao-2,4,6,8-cyclooctane; CA name 3,7-Dinitro-1,3,5,7-tetraazabicyclo[3.3.2] nonane. See under 1,5-Endoethyldiene-1,3,5,7-tetraazao-2,4,6,8-cyclooctane.

1,6-Dinitroguanidine. 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

Dinitro(bis[ethylendiamine]cobalt(III)) Nitrates. See under Di(ethylendiamine) and Derivatives in this Volume, p D1231

N,N'-Dinitro-N,N'-bis(2,4,6-trinitrophenyl)-ethylendiamine; N,N'-Dipriceylethylene-dinitramine; or 2,4,6,2',4',6'-Hexanitrodiphenethylendiamine (Code named Bitetrel or Ditetril (US) and Octyl (British). See in Vol 2, p B131-R under Bis(anilino)ethane and Derivatives

Dinitrobitotol or Dinitro-dimethylbiphenyl. See Vol 2, p B163-R

1,5-Dinitrobiuret, Salts 1,5-Dinitrobiuret, O$_2$N.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$_2$C$_2$H$_5$N$_2$O$_5$, was mentioned in Encycl, while the Lead dinitrobiuret, PbC$_2$H$_5$N$_2$O$_5$, 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 100º Heat Test, this salt exploded in the first 48 hrs.

The Silver salt, AgC$_2$H$_5$N$_2$O$_4$, wh. nlds (from w + alc) also exploded on heating (Ref 2)

Refs: 1) Beil 3, 126 (K salt, KC$_2$H$_5$N$_2$O$_4$) 2) Beil 3, 126 (Ag salt) 3) S. Helf, L. 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)

Dinitroammonine. See Vol 2, p B307-R

Dinitroazobenzene. See Vol 2, p B308-L

Dinitroazoxybenzene. See Vol 2, p B308-R
Dinitrobenzobenzalone. See Vol 2, p B309-L

Dinitrobenzobenzonyl Azide. See Vol 2, p B311-L

1,1-Dinitropropane. See Vol 2, p B311-R

Dinitropropanes. See Vol 2, p B312-R

Dinitropropanoic acid. See Vol 2, p B312-R

1,3-Dinitropropanoic Acid. See Vol 2, p B312-R

Dinitropropanoic acid. See Vol 2, p B312-R

Dinitropropanoic acid. See Vol 2, p B312-R

Dinitrothiophene. See Vol 2, p B312-R

Dinitrothiophene. See Vol 2, p B312-R

Dinitro-iso-butane. See Vol 2, p B315-R

1,4-Dinitro-2,3-butanediol. See Vol 2, p B367-R

Dinitrobutanol. See Vol 2, p B367-R

Dinitrobutane. See Vol 2, p B367-R

Dinitro-isobutane. 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

Dinitrobutane. See Vol 2, p B376-L

Dinitro-N-butylaniline. See Vol 2, p B378-R

Dinitrobutyric Acid. See Vol 2, p B392

Dinitrobutyric Acid. See Vol 2, p B392

Dinitrocarbanilide. See Vol 2, p C44-L

Dinitrocarbazoles. See Vol 2, pp C47-R & C48-L

Dinitrocoumarine. See Vol 3, p C574-L

Dinitrocyanides. See Vol 3, p C637-R

3,7-Dinitro-3,7-diaza-1,5-dioxacyclooctane

O₂CH₂N(NO₂)₂ CH₂

CH₂N(NO₂)CH₂O; mw 252.14, N 38.11%;
crystals (from acet), mp 263–64°C; sol in acet;
sol 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.: 1) Beil, not found 2) W. J. Chute et al,
Canadian J. Res. 27B, 225 & 235–36 (1949) &
CA 43, 8355 (1949)

3,5-Dinitro-2,5-diazopiperidinium Nitrate

(Code name PCX)

H₃C — NH·HNO₃ — CH₂

O₂N.N — CH₂ — N.NO₂; crystals, mp
98–103°C; was isolated by Berman et al (Ref
4) in the nitromethyl of hexamine dinitrate with
97% nitric acid at −40°C 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

Ref.: 1) Beil, not found 2) W. J. Chute et
al. Canadian J. Res. 27B, 506 (1949) 3) A.H.
Vroom & C.A. Winkler, Canadian J. Res. 28B,
701 (1950) 4) L. Berman et al. Canadian Chem.
29, 767 & 773 (1951) & CA 46, 2084 (1952)

Dinitrochloroanilinopropanediols. See Vol 3,
p C247-R

Dinitrochlorobenzones. See Vol 3, p C249-R

Dinitrochlorobenzoic Acid. See Vol 3, p C252-L

Dinitrochloroazobenzoic Acid Azide or Dinitro-
chlorobenzoic Azide. See Vol 3, p C252-L
Dinitrochlorobenzonitrile. See Vol 3, p C252-R

α-Dinitrochlorohydryn. See 3-Chloropropene-1,2-diol-1,2-dinitrate in Vol 3, p C265-R

β-Dinitrochlorohydryn. See 2-Chloropropene-1,3-diol-1,2-dinitrate, Vol 3, p C266

Dinitrochloromethane. See Vol 3, p C259

Dinitrochloromethylaniline. See Vol 3, p C260-R

Dinitrochloropropene. See Vol 3, p C265-L

Dinitrochloropyridine. See Vol 3, p C267

1,5-Dinitro-2,6-diazidooanthraquinone. See Vol 1, p A460-L

4,6-Dinitro-1,3-diazidobenzene. See Vol 2, p B43-L

2',4'-Dinitro-4-diazo-diphenylamine. See 4-(2',4'-Dinitro-phenylamino)-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-diazoctane. 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' nitraminoethyl)-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-Dinitrodihydroxybenzene. See Bis-(β-nitraminocetyl)-amine in Vol 2, p B128-R

Dinitrodihydroxybenzene Dinitrate; Dinitrodiethanol-oxamide Dinitrate; or N,N'-Dinitrodi(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, H₈C₂·N(NO₂)₂·SO₂·N(NO₂)₂·C₆H₅; mw 242.22, N 23.13%. Not found in the literature thru 1966

It evidently can be prepd by nitration with 95% nitric acid, its parent compd: N,N'-Diethyl-sulfamide,
H₈C₂·NH·SO₂·NH·C₆H₅; 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 preps 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-dihydroxynitrobenzene. See 1,3-Bis(hydrazino)-4,6-dinitrobenzene in Vol 2, p B143-R

4,6-Dinitro-3,5-dihydroxynitrobenzene (called by von Herz Dinitro-azo-dihydroxy-quinonanhydrid),

\[ \text{O}_2\text{N.C} \rightarrow \text{CO} \rightarrow \text{C} \rightarrow \text{N} \]

\[ \text{H.O.C.C(NO}_2)\text{C.OH} \text{; mw 242.11, N 23.14%}. \text{This compd and its expl salts were claimed to be prepd by von Herz and recommended for use in initiators. It can be prepd by treating mono- or di-nitroaminophloroglucinol with hot} \]
strong sulfamic-nitric acid (Ref 2)
Ref:s: 1) Beil, not found 2) E. von Herz, BritP 207563(1923) & CA 18, 1573-74(1924)

9,9'-Dinitro-9,9'-dihydroxy-10,10'-dihydroantracene. See Bis(nitrohydroanthranol) in Vol 2, p B144-L

N,N'-Dinitro-N,N'-dihydroxyethyl-oxamide Dinitrate. See Dinitrodiethylyl-oxamide Dinitrate under Diethylol-oxamide in this Volume, p D1244-L

3,6-Dinitro-2,5-dihydroxyquinone Di azide. See under Dihydroxybenzoquinone and Derivatives

1,4-Dinitro-3,6-diketopiperazine. See under Diketopiperazine in this Vol

Dinitrodimethyline. See under Dimethylamine in this Vol

2,2'-Dinitro-2,2'-dimethoxylaxethane. See 2,2'-Dinitro-2,2'-azoxypropane in Vol 1, p A671-R

Dinitrodimethylbiphenyl. See Dinitrobitoyle 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

Dinitrodimethylolebenzene. See under Dimethylolebenzene in this Vol

Dinitromethylol-oxamide or Dinitroxydimethyl-oxamide. See under Dimethyloxamide 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-nitraminop-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

Dinitrodimethyltartrazamide Dinitrate. See under Dimethyltartrazine in this Vol

N,N'.Dinitro-N,N'-dimethyl-3,5,3',5'-tetrano nitrobenzidine. See Vol 2, p B64-R

1,3-Dinitro-5,7-dimethyl-1,3,5,7-tetraazacyclooctane. See under Dimethy1-tetraazacyclooctane in this Vol

Dinitrodimethylurea. See under Dimethy lurea in this Vol

Dinitro-dinitrosobenzenes. See Vol 2, p B45-R

Dinitro-di(β-nitroxyethyl)-oxamide (Code name NENO). See under Diethylyl-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'-Dinitrodiphenyloamino-4-diazonium Nitrate. See 1-(2,4-Dinitroanilino)-benzene-4-diazonium Nitrate in Vol 1, p A421-R

N,N'-Dinitro-N,N'-dipropyl-3,5,5',5'-tetranitrobenzidine. See Vol 2, p B64-R.

3,7-Dinitro-1,5-endoethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane. See under 1,5-Endoethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane (Code named homo-DPT)

3,7-Dinitro-1,5-endoethylidene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane. See under 1,5-Endoethylidene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane (Code named DPT)

Dinitroethane. It is described under Ethane

Dinitroethyleneglycol or Nitroglycol (NGc). See under Ethyleneglycol and Derivatives

Dinitroethylenurea. See under Ethylene Urea and Derivatives

Dinitroethyloamin. See 2-Amino-2,2-dinitroethanol in Vol 1, p A201-R

Dinitroethoxamide. See under Ethyloxamide and Derivatives

Dinitroformin or Formyl-dinitroglycerin. See under Formin or Formic Acid Esters

Dinitrofuran. See under Furan and Derivatives

Dinitrogen Pentoxide, N₂O₅. Described under Dinitrogen Tetroxide, N₂O₄. NITROGEN OXIDES

Dinitroglycerin or Glycerol Dinitrate and Trinitroglycerin or Nitroglycerin (NG). It is described under GLYCERIN AND DERIVATIVES

Dinitroglycerin Picrate or Glycerin-2,4,6-trinitropheny lethere Dinitrate. See under Glycerin and Derivatives

Dinitroglycerol; 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

\[ \text{N(NO}_2\text{).CH.N(NO}_2\text{)} \]

\[ \text{OC} \]

\[ \text{NH} \text{—CH.NH} \]

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 prepared 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-diozo-benzoquinone, called by von Herz Dinitroresorcinodiazooanhydrid,

\[ \text{O}_2\text{N.C—CO—C}\text{N} \]

\[ \text{HC.C(NO}_2\text{).C.OH} \]

mw 226.11, N 24.78%; yel ndls; obd as free diazium compound by treating the K salt with concd sulfuric acid (Ref 1)

Its Potassium Salt,

\[ \text{C}_6\text{H}(\text{OK}.\text{NO}_2)_2(\text{N})_(2)(\text{O}) \]

yel ndls (from concd aq soln) was prepared by von Herz on treating with an excess of K nitrite the mononitro-aminoresorcinol, H₂N.C₆H₅(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 crystals 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-chinon-diazi(1); or 3,5-Dinitro-4-oxy-chinon-diazi(1) in Ger],

\[ H\text{C} = C(N_2) = C.\text{OH} \]

O\text{N} = C(\text{:O}) = C.\text{NO}_2; \text{mw} 226.11, N 24.78%; golden yel plts, very expl contd; was obtd by dissolving 2,3,6-trinitro-4-diazophenol (2,3,5-trinitro-p-quinone-1-diazi) in boiling NaOAc, collecting and washing the orange Na salt, dissolving in water, and acidifying withaq HCl. The product combines, when moist, with β-naphth (Refs 1 & 2)


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,

![Diagram]

mw 452.22, N 24.78%; bm-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)


\( β,β\)-Dinitro-iso-propylamine. See 1,1-Dinitro-2-aminopropane in Vol 1, p A250-R

1,3-Dinitro-2-keto-5-hydroxyhexahydroxypyrindine Nitrate,

\[ \text{H}_2\text{C}.\text{N(NO}_2)_2\text{.CO} - (\text{O}_2\text{NO})\text{.CH} \cdot \text{CH}_2 \cdot \text{N(NO}_2)_2; \text{mw} 251.12, \text{N} 27.89%; \text{OB to CO}_2 \text{ 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\text{N.CH}_2\text{.CH(OH).CH}_2\text{.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]


Dinitromethane. It is described under Methane and Derivatives

Dinitromethoxyethoxyquinol-Nitric Acid, Potassium Salt of (Dinitromethoxyäthoxy-chinolnitosäures Kaliunm in German), C\text{H}_3\text{N}_3\text{O}_4\text{K} (its probable structural formula is given in Ref 1; p 290, lines 12 & 13); mw 327.29, N 12.84%; OB to CO\text{2} and K\text{2}O 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
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 A253-R as 3,5-Dinitro-4-methyl-2-nitrimino-4-thiazoline

Dinitromethoxyxamid. It is described under Methylxamid and Derivatives

1,2-Dinitro-2-methyl-propane. This compd was described in Vol 2, p B368-R as Dinitroisobutane, but its expl props, such as given by A.H. Blatt in OSRD Rept 1941 (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°C - 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°C - no brown fumes evolved after 5 hrs
* Vacuum Stability at 100°C - 2.05cc evolved in 40 hrs

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)

\[
\begin{align*}
H_2C & \longrightarrow N\text{CH}_3 \longrightarrow CH_2 \\
O_2N & \longrightarrow CH_2 \longrightarrow CH_2 \longrightarrow N\text{NO}_2; \text{ mw 205.18, N 34.14%; crysts, mp 159°C, was prep when ethylenedinitramine, (CH}_3\text{N}_2\text{H}_2\text{N}_2\text{O}_2\text{, in 37% fomalin was treated dropwise at 0°C with 30% aq methylamine soln (Refs 1 & 2). An HCl salt, mp 138-40°C was also prep} \\
\text{Its UV absorption spectrum was detd by Jones & Thorn (Ref 2)}
\end{align*}
\]


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

Dinitronitraminotoluene. See Vol 1, p A265-R

Dinitronitraminoxylene. 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-chloromethy laniline. See Vol 3, p C261-L

1-(2',4'-Dinitrophenoxy)-2-nitroxy-ethylol or β-(2,4-Dinitrophenoxo)-ethyl Nitrate,
\[
\text{H}_2\text{C} - \text{CH}_2\text{ONO}_2
\]
\[
\text{NO}_2
\]
crysts, mp 70°; dec at 145° & delivr at 300°; was prep'd by action of anhyd nitric acid on 2,4-dinitrophenoxethanol (Ref 2). Méard & Thomas (Ref 3) detd its heat of combustion as const vol = 927.4 kcal/mol & heat of formation 64.9 kcal/mol; Tavener & Lamouroux (Ref 4) reported values of 930.1 & 62.2 kcal/g, respectively. A patent was issued (Ref 5) for the prep' of this comp'd by nitrating 2-(2,4-dinitrophenoxo) ethyl nitrate with concd sulfuric acid & 60% nitric acid at −10°.

Blanksm & Fohr (Ref 2) reported prep' of the 2,4-g-binitro deriv, mp 104°, bitter taste, by nitrating the dinitro comp'd with anhyd nitric acid & sulfuric acid at 0° for 30 mins


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-L

2-(2',4'-Dinitrophenylamino)-1,3-dihydroxy-propane. See 2-(2',4'-Dinitroanilino)-1,3-propanediol in Vol 1, p A435-L

2,4-Dinitrophenylamino-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-hydroxyethoxy)-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-phenyliminom)-1-diazonium-1,4-benzoquinone or 2,4-Dinitrophenyl-p-phenylenediazomine. 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-nitroethyl-nitrate. See under Phenyl-ethylol or Phenyl-hydroxyethyl 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), H₃C.C(NO₂)₃.CH₂.O.CO.CH₂.CH.C(NO₂)₃; mw 355.18, N 19.72%; OB to CO₂ minus 29%; wh crystals, sp gr 1.68; mp 110°C (Form I), 95°C (Form II) and 59°C (Form III); bp exp at 300°C 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, CH₃C(NO₂)₂.CH₂OH, trinitrobutyl chloride, Cl.CO.CH₂.CH₂.C(NO₂)₃ and Al chloride in carbon tetrachloride at 60°C and refluxing the mixture at 75°C for 2 hrs. After the completion of reaction, the slurry was cooled and the crystalline product purified. It had a mp of 95°C to 96°C

DNPTB is one of the Navy's experimental expls. Its deton velocity was 7600m/sec and vacuum stability value 0.5m1 at 100°C in 40 hrs (Ref 3) (See also Refs 3, 4, & 6-9)


4,6-Dinitroresorcinol-bis( or di)(2-hydroxy-ethyl)-ether. See 1,3-Bis(2-hydroxyethoxy)-4,6-dinitrobenzene in Vol 2, p B145-R

Dinitrosobenzamide; Benzenyldioxynitrozolic Acid or Dinitrosobenzynamidine, C₆H₅.C(C:NO),NH(NO) or

C₆H₅.C\[\text{N:NOH}\]

mw 178.16, N 31.47%; OB to CO₂ minus 134.6%. It is an unstable expd 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 ag sol of benzamidine (benzenyldiamidine) hydrochloride, C₆H₅.C(NH),NH₂.HCl, (described in Beil 9, 280) with an ag 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, KC₄H₄N₂O, was recrystallized in the form of ndis or lfts, 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, \( \text{NH}_4\text{C}_2\text{H}_5\text{N}_4\text{O}_2 \); prisms de
going at 137°

Benzenamide Salt, \( \text{C}_6\text{H}_5\text{N}_2+\text{C}_2\text{H}_6\text{N}_4\text{O}_2 \); lfts
(from alc); prisms (from w) deflagrating at
178° on rapid heating

Silver Salt, \( \text{AgC}_2\text{H}_5\text{N}_4\text{O}_2 \); 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. Raths-
sburg, Brit P 185555 (1923) & CA 17, 1147
(1923) 4) A.H. Blatt, ORSD 2014(1944)

Dinitrosobenzene. See Vol 2, pp B44-R &
B45-L

Dinitrosobenzenylamidine. See Dinitroso-
benzamidine in this Vol

2,3-Dinitrosobenzoquinone-1,4-dioxide.
HC.C(=NOH).C:NO

HC.C(=NOH).C:NO; mw 196.12, N 28.57%;
dk ycl nds (from alc), mp 68°; sol in alka
ilies which soln on exposure to air is oxi-
dized to 1,2,3,4-tetranitrosobenzene; was
prepd by adding dropwise nitric acid (d 1.40)
to aq diquinonyl tetraoxide 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-dioxide and
Derivatives in Vol 2 of Encycl, pp B83-B84

3,7-Dinitroso(bicyclo)-2,4,6,8,9-penta-
methylen-1,3,5,7-tetramine; or 3,7-Dinitroso-
1,5-endomethylene-1,3,5,7-tetraza-2,4,6,8-
cyclooctane; 3,7-Dinitroso-1,3,5,7-tetra-
aza(bicyclo)[3.3.1]nonane (CA name). See
under 1,5-Endomethylene-1,3,5,7-tetraaza-
2,4,6,8-cyclooctane and Derivatives

Dinitrosoclaromethyline. See Vol 3,
p C260-L

3,7-Dinitroso-1,5-endomethylene-1,3,5,7-
tetraza-2,4,6,8-cyclooctane. See under 1,5-
Endomethylene-1,3,5,7-tetraza-2,4,6,8-
cyclooctane and Derivatives

3,7-Dinitrosopentamethylene-1,3,5,7-tetra-
mine. See 3,7-Dinitroso-1,5-endomethylene-
1,3,5,7-tetraza-2,4,6,8-cyclooctane under
1,5-Endomethylene-1,3,5,7-tetraza-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-tetraza-2,4,6,8-
cyclooctane, described under 1,5-Endo-
methylene-1,3,5,7-tetraza-2,4,6,8-cyclo-
octane and Derivatives

3,7-Dinitroso-1,3,5,7-tetraazabicyclo[3.3.2]-
decane. CA name for 3,7-Dinitroso-1,5-endom-
eylene-1,3,5,7-tetraza-2,4,6,8-cyclooctane,
described under 1,5-Endomethylene-1,3,5,7-
tetraza-2,4,6,8-cyclooctane and Derivatives

3,7-Dinitroso-1,3,5,7-tetraazabicyclo[3.3.1]-
nonane. CA name for 3,7-Dinitroso-1,5-endom-
eylene-1,3,5,7-tetraza-2,4,6,8-cyclo-
octane, described under 1,5-Endomethylene-
1,3,5,7-tetraza-2,4,6,8-cyclooctane and Derivatives

1,4-Dinitro-1,1,4,4-tetramethyloxobutane
Tetranitrate,\( \left( \text{O}_2\text{N} \right)_2\text{CH}_2\text{CH}_2\text{C}(\text{NO}_2) -
\left[ \text{CH}_2\text{ON}(\text{NO}_2) \right]_2 \); 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) BurMinasHigh-
ExplResDivRept No 25, June 9, 1942
3) H.B. Haas, ORSD Rept 1964(1943)
4) A.H. Blatt, ORSD 2014(1944)

1,5-Dinitroso-1,1,5,5-tetramethylxolpentane
Tetranitrate, \( \left( \text{O}_2\text{N} \right)_2\text{CH}_2\text{C}(\text{NO}_2)\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{C}(\text{NO}_2)\cdot\text{CH}_2\text{O}_2\text{ON}(\text{NO}_2) \); 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-dinitrotetra-pentene with formaldehyde

It is an explosive more powerful than Tetryl and more sensitive than TNT

1,10-Dinitro-1,4,7,10-tetrapicrylhexamethylene-1,4,7,10-tetramine or n-ω-Dinitrotetrapicryltetraethylenetetramine; 
\[
\begin{align*}
\text{C}_4\text{H}_2\text{(NO}_2\text{)}_5 & | \quad \text{C}_6\text{H}_5\text{(NO}_2\text{)}_5 \\
\text{C}_6\text{H}_5\text{(NO}_2\text{)}_5 & \quad \text{C}_6\text{H}_5\text{(NO}_2\text{)}_5
\end{align*}
\]

MW 1080.64, N 23.33%, OB to CO₂ minus 62.2%; solid, mp 159°(dec); bp — expl at higher temp; can be prepd by nitration of product obtd by condensation of triethylenetetramine with Rcryl 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 1944(1944)

1,7-Dinitro-1,4,7,10-tetraazadecane. See 1,2-Bis(2'-aminoethylnitramino)-ethane in Vol 2, p B129-L

Dinitrotoluenes. They are described under TOLUENE and DERIVATIVES

2,4-Dinitro-1,3,5-triaminobenzene. See under Triaminobenzene and Derivatives

1,7-Dinitro-1,4,7-triazahopentane. 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-dinitrooxamide or Bis(β-nitroxyethyl)-dinitrooxamide (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,
\[
\text{C}_2\text{H}_5\text{ON}(\text{NO}_2\text{)}_2\text{CH}_2\text{(ONO}_2\text{)}}_2; \text{mw 212.08}, \text{N 26.41%; OB to CO}_2 +15.1%; \text{crysts, mp 59-60°; bp — expl at higher temp; sol in methanol, eth, et acet, acetone, NMe and doxane; sl sol in benz & chlor; insol in w; was obtd by Chute et al on treating with 99% nitric acid some of the products isolated on nitrolysis of hexamine}
\]

It is a powerful, impact-sensitive expl, which unfortunately decomposes on standing when exposed to air

1,5-Dinitroxy-2,4-dinitro-2,4-diazapentane or 2,4-Dinitro-2,4-diazapentanediol-1,5-dinitrate (Code name COX). See under Dihydroxytriaminomethylenediamine

Di(β-nitroxyethyl)-nitramine. See under Diethylolamine, p D1240-R

Di(β-nitroxyethyl)-N,N'-dinitrooxamide or N,N'-Dinitroxyethol-oxamide (Code name ...
NENO). See under Diethyol-oxamide, p Di244-L

Dinitroxyethyl -nitramine or Diethylolnitramine Dinitrate. See under Diethyloamine or Diethanolamine, p D1240-R

Dinitro-xylene; Dinitro-xylene-diazide and Dinitro-xylene-dinitrate. They are described under XYLENE and DERIVATIVES

Dinitroxyaminodiethane or Diethylolnitramine Dinitrate. See under Diethylamine, 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-tetrazaanone

1,8-Dinitroxy-1,3,5,6,8-pentamethylene-2,4,7-trinitramine. See 1,8-Dinitroxy-2,4,7-trinitro-2,4,7-triazaoctane

Dinitroxy(propylamine). See under Amino-propanediols and Derivatives in Vol 1 of Encycl, p A251-L

Dinitroxy(propylamine). Same as above

Dinitroxy(propylamine) Nitrate, H₂N.C₃H₇(ONO₂)₂ + HNO₃, is described in Vol 1, p A251-R as the: Nitrate Salt of 1-Amino-2,3-propanediol Dinitrate

This compound 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

Dinitroxy(propyl)-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 Dihydroxytetramethylene-tetramine and Derivatives

1,9-Dinitroxy-2,4,6,8-tetranitro-2,4,6,8-tetraazaanone (Code designation 106) or 1,9-Dinitroxy-pentamethylene-2,4,6,8-tetranitramine (called in CA 2,4,6,8-Tetranitro-2,4,6,8-tetraazaanone-1,9-diol Dinitrate)

(⁡_NO₂)H₂C.C.N(⁡NO₂)₂.CH₂.N(⁡NO₂)₂.CH₂.N(⁡NO₂)₂. — CH₂.N(⁡NO₂)₂.CH₂(⁡NO₂); mw 434.21, N 32.26%, OB to CO₂ minus 3.7%; solid, mp 201° (on rapid heating), bp = exp at higher temp. It can be prepared either by nitration of 1,9-Diacetoxy-pentamethylene-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-tetraazaanone) 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-cyclooctane and described here under 1,5-Endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane and derivatives

Its explosive properties are:
Impact Sensitivity — Bruceon 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: 730 m/sec at sp gr 1.265 g/cc and 7895 at 1.35
1,5-Dinitroytrimethylene-2,4-dinitramine
(Code named COX). See under Dihydroxytrimethylenediamine

1,7-Dinitroxy-2,4,6-trinitro-2,4,6-triazahexane 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-triazooctane
or 1,8-Dinitroxy-1,3,5,6,8-pentamethyleno-
2,4,7-trinitramine (CA name 2,4,7-Trinitro-
2,4,7-triazooctane-1,8-diol Dinitrate).

(O_3NO)_2H_2C=NC(NO_2)_2.CH_2.N(NO_2)_2.CH_2.CH_2.-
N(NO_2)_2.CH_3(ONO_2) ; mw 374.19, N 29.94%;
wh solid, could not be purified from dioxane,
CCl_4 or acet; unstable product which decomp
after a few mins at RT; was obtd when 1,8-
acetoxy-2,4,7-trinitro-2,4,7-triazooctane was
nitrated with 98% nitric acid at 0^\circ for four
mins and the soln kept at 0^\circ 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,
Can J Res 27 B, 499 (1949) & CA 43, 9074
(1949) 3) Not found in later Refs thru 1966

Dinitryl. Same as Glycerin α-2,4-dinitro-
phenylether Dinitrate or 1-(2,4-Dinitrophenoxoy)
-2,3-propane Dinitrate, described under
GLYCERIN AND DERIVATIVES

Dinol. See 2,2,8,8-Tetranitro-4,6-dioxo-
1,9-nonanediol which is listed under 4,6-
Dioxo-1,9-nonanediol as a conf compd

Dinol. Commercial name for Diazodinitro-
phenol, 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 ineffectif 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 effective-
ness 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.
Re: Fedoroff et al, PATR 2510 (1958), p
Ger 37-L

Dinitüré. Accdg to Sidgwick (Ref), the name
"dinitüré" applies to compds contg the group
>C(NO_2)_2<, which can be isolated
among the products of reactions of nitrogen
oxides with unsaturated hydrocarbons

For example, when stilbene,
C_6H_5.CH:CH:C_6H_5, is treated with nitrogen
pentoxide, N_2O_5, the resulting compd is
C_6H_5.CH(NO_2)_2.CH(NO_2)_2.C_6H_5, named 1,2-
dinitro-1,2-diphenyl-ethane. The group,
-CH(NO_2)_2.CH(NO_2)_2-, is a "dinitüré"

Some "dinitürés" are explosive
Re: N.V. Sidgwick, "Organic Chemistry
of Nitrogen", OxfordUnivPress, London
(1937), pp 225 & 244
Dioctyl Adipate and Derivatives

N,N'-Dioctyl-adipamide or Bis(2-ethylhexyl)-adipamide,

\[ C_8H_{17}CH=NH\cdot CO\cdot CH_2\cdot CH_2\cdot CH_2\cdot CO\cdot NH\cdot CH\cdot C_8H_{17} \]

\[ C_8H_{17}CH=NH\cdot CO\cdot CH_2\cdot CH_2\cdot CH_2\cdot CO\cdot NH\cdot CH\cdot C_8H_{17} \]

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

\[ C_8H_{17}CH=NH\cdot CO\cdot CH_2\cdot CH_2\cdot CH_2\cdot CO\cdot NH\cdot CH\cdot C_8H_{17} \]

\[ C_8H_{17}CH=NH\cdot CO\cdot CH_2\cdot CH_2\cdot CH_2\cdot CO\cdot NH\cdot CH\cdot C_8H_{17} \]

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, US Rubber Co Quarterly Progress Rept 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 cntrs. One of the rocket proplnts, Unit No 547, using DOA is described in prep "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

Property

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Color</td>
<td>No darker than color</td>
</tr>
<tr>
<td>1) Sp Gr at 20⁰/20⁰C</td>
<td>0.927±0.002/g/c</td>
</tr>
<tr>
<td>2) Refractive Index at 20⁰C</td>
<td>1.447±0.0010</td>
</tr>
<tr>
<td>3) F1 Pt, min</td>
<td>385⁰F</td>
</tr>
<tr>
<td>4) F1 Pt, min</td>
<td>420⁰F</td>
</tr>
<tr>
<td>5) F1 Pt, max</td>
<td>0.10</td>
</tr>
<tr>
<td>6) Acid Pt, max</td>
<td>8.46±0.30 centistokes</td>
</tr>
<tr>
<td>7) Viscosity at 100⁰F</td>
<td>302±2</td>
</tr>
<tr>
<td>8) Saponification Number</td>
<td></td>
</tr>
</tbody>
</table>

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 & 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 Abbe (See SGA Scientific Inc, Bloomingfield, 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 vapor 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 sec's [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:

\[
\text{Acid Number} = \frac{56.1 \times (AN)}{Bd}
\]

where: \(A = \text{mL of KOH soln required to titrate the sample}\)

\(N = \text{normality of KOH soln (usually 0.01N)}\)

\(B = \text{volume of sample titrated (50mL)}\)

\(d = \text{specific gravity of the material} \quad (\text{See 4.4.2})\)

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:

\[
\text{Saponification Number} = \frac{56.1 \times (A-B)N}{W}
\]

where: \(A = \text{mL of HCl required to titrate the} \)
sample
B = ml of HCl required for blank
titration
N = normality of hydrochloric acid soln
W = weight of the sample (ca 2g)

Diocetyl Azelate (DOZ). See Di(2-ethylhexyl) azelate in this Vol, p D1237-L

N,N'-Diocetyl-p-phenylenediamine (DOPDA) or Bis(2-ethylhexyl) -p-phenylenediamine,
(H2N)2C6H2[COOCH2CH(C6H5)CH2]2; mw 420.58, N 6.66%; description of compd & props
are given below under specification requirements

E. J. Bicek [JSP 320930 (1965); CA 64, 3415 (1966)] prepared this compd by passing H and
a soln of p-O2NC6H5NH2 in C6H5COC6H5, downward over a sulfided-Pt catalyst in a
jacketed reaction tube at the rate of 100 cc/hr at 850°F/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 elastomers. 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, par-
ticularly tires. This chemical functions as an antioxidant, 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 24 hrs at a temp no higher than 42°F
and then turn the tube upside down. The material shall be capable of being poured

Diocetyl Phthalate (DOP) or Bis(2-ethylhexyl) phthalate, C6H4(OCOOCH2CH(CH2)6)C6H52;
mw 390.54; It col liq, sp gr 0.986 at 20/20°,
np 1.4830 to 1.485 at 20° ft p minus 50 to 55°,
bp 231° at 5mm, bp 385° at 760mm, flash p
410°F, vapor pressure 1.3mm Hg at 200° vis-
cosity 81.4cps at 20°; insol in w, miscible
with mineral oil; can be prep'd 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 Unir Nos 1024, 1026, 1058,
1059 & 1067 described in ConfProp-Prop-
Manual SP1A/M2(1969)

US Ammd 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 25,000ml of 0.5N al-
coholic 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 ppt K phthalate
and reflux for an addnl 45–60 mins. Cool the
flask to RT; disconnect the reflux condenser,
left 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 phpt indicator until disappearance of pink
color. Run a blank concurrently with the
sample

\[
\text{%DOP} = \frac{19.53 \text{(A-B)N}}{\text{W}}
\]

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 phpt 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 5ml 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 determined as described below:

The APHA (American Public Health Association) color standard (stock solution) is prepared by dissolving 1.245g K2PtCl6 (contains 500mg Pt) & 1.000g CoCl2.6H2O (contains 248mg Co) in distilled water, then diluting with 1000ml of HCl and diluting with water to 1000ml. This soln has a color intensity equivalent to 500ppm of platinic cobalt chloride.

Note: According to Bofors laboratory manual (Ref 6, p30) this soln, known as APHA500 contains 500mg Pt and by diluting its 10ml with 90ml of water in 100ml Nessler tube, the APHA50 contains 0.5mg of Pt.

For determination, fill the mark to a 100ml Nessler tube with the sample as received and compare the intensity of color with that of APHA50 contained in another Nessler tube by looking vertically downward through 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 oven 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 ml's are poured into 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 sproplnts. 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-yellow 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°C

3) Ester Content, as DOP. Transfer ca 2g sample weighed on analytical balance, to 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 phpt as an indicator until discoloration of soln.

Run a blank det at the same time

\[ \% \text{Diocetylphthalate} = \frac{19.52(b-a)/w}{a} \]

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 Diarylphthalate and Diarylphthalate, described on pp82-3 of Ref 5, except that the formulas for calcn are different, namely

\[ \% \text{Diarylphthalate} = \frac{15.13(b-a)/w}{w} \]
% 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 phpt indicator (using a microburet) until the appearance of faint pink coloration.

% 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 pp 15-18 of Ref 5.


Dioctyl Sebacate (DOS). See Di(2-ethylhexyl)sebacate in this Vol and the Conf Proplnt Manual SPIA/MZ (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°C = 0.913±0.002.

Determine by a pycometer as described in Vol 3 of Encycl, p D69.

3) Refractive Index at 20°C = 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% phpt 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.

\[
\text{Acid Number} = \frac{56.1\text{AN}}{\text{VG}}
\]

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 Viscometer 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% phpt 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.
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

-dione. A suffix indicating the presence of two keto-groups: -OC.CO-, such as in compds known as diketones: 2,3-butanedione(2,3-butane) or diacetyl, H$_2$C.COH.COH, or 2,4-pentadiadione(2,4-pentane) or acetylacetone, H$_3$C.COH.COH.COH, 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 (1922), p206

-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 ethanediol (or glycol), propandiol, butandiol, etc

Ref: Hackh’s Diction (1944), pp278-L & 383-L

Diolefine. An unsaturated aliphatic hydrocarbon with the general formula C$_n$H$_{2n+2}$ and two double bonds. E.g: diallyl (1,5-hexadiene), CH$_2$:CH.CH$_2$.CH$_2$.CH:CH$_2$

Ref: Hackh’s Dict (1944), p278-L

3,5-Diols-triazine, HOC==N-N

HO C==N C0H;

mw 113.08, N 37.16%; white compd (sublimation in vacuo, 200°, 1 mm), 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

Re/s: 1) Belal, nor found 2) W. Seibert, ChemBer 80, 494(1947) & CA 43, 123(1949)
3) E.A. Balco & al, J ACS 78, 1940(1956) & CA 50, 13952(1956)

Dioxane or Dioxan-1,4. (Diethylene-1,4-dioxe),

\[
\begin{align*}
\text{CH}_2 &\quad \text{CH}_2 \\
\text{CH}_2 &\quad \text{CH}_2 \\
\end{align*}
\]

mw 88.10; col, very flammable liq, sp gr 1.034
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 PictArsn (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


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 insoluble compound 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.

At 20/4°C, bp 118°C, nD 1.4221 at 20°C, hP 18°C (65°F) (ASTM open cup), vapor pressure 29.0mm Hg at 20°C, viscosity 0.0131 poise at 20°C, heat of combustion 581 kcal/mole, miscible with water & alcohol; sol in eth and in most organic 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 (Ref 11, 12, 13 & 14)

Vapors of dioxane are of pleasant odor but extremely toxic to inhale. In the book of Cumee & 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, LD50 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 prep'd in 1863 by Laurenco (Ref 1 & 2) by heating ethylene glycol with ethylene dibromide in sealed tubes for several days at 160°C. In the same year Wurtz (Ref 3) prep'd it independently by treating dioxane dibromide (obt by him in 1862) with hydrogen sulfide or Hg at RT. Several industrial methods of prep'n are listed in the book of Cumee & Johnston (Ref 13). Among them may be mentioned a method patented by scientists of IG Farbenind (Ref 4), consisting of dehydration of ethylene glycol, polyethylene glycol 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 condensation of formaldehyde or acetaldehyde over catalysts favoring esterification reactions, such as sulfuric acid or ferric chloride.

In Cond-ChemDict (Ref 14) are listed two methods of prep'n: a) Treating ethylene glycol with acids and b) Treating B,B'-dichloroethyl ether (CH₂C·CH₂Cl)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.
Dioxones, Monocyclic are described by Elderfield 6(1957), pp 1–58

4,6-Dioxo-1,9-nonanedial and Derivatives
4,6-Dioxo-1,9-nonanedial,
\[ \text{OCH}_2\text{CH}_2\text{CH}_2\text{OH} \]
\[ \text{CH}_2 \]
\[ \text{OCH}_2\text{CH}_2\text{CH}_2\text{OH} \]
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-Tetranitrodervative is described by T.N. Hall & K.G. Shipp in confidential report, MOLTR 61–6 (March 1961)
\[ \text{OCH}_2\text{C(NO}_2\text{)}_2\text{CH}_2\text{OH} \]
\[ \text{CH}_2 \]
\[ \text{OCH}_2\text{C(NO}_2\text{)}_2\text{CH}_2\text{OH} \]
mw 294.20, N 19.05%. This compd, designated as DINOLO, is described in detail, with its method of prep in the above report.

Dioxotetrazopentadecane and Derivatives
3,13-Dioxo-5,7,9,11-tetrazopentadecane,
H\(_5\)C.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\(_9\)H\(_{12}\)N\(_2\)O\(_8\), and Trinitro, C\(_9\)H\(_{12}\)N\(_3\)O\(_8\), derivs were not found in the literature thru 1966

3,13-Dioxo-5,7,9,11-tetranitro-5,7,9,11-tetrazopentadecane, (called 1,9-Diethylenicly-2,4,6,8-tetranitro-2,4,6,8-tetrazelazone) by Chute et al., H\(_5\)C.CH\(_2\)O.CH\(_2\)N(NO\(_2\))\(_2\).CH\(_2\)N(NO\(_2\))\(_2\).CH\(_2\)N(NO\(_2\))\(_2\).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-Dioxo-5,7,9,11-tetranitro-5,7,9,11-tetrazopentadecane-1,15-diol,
HO\(_2\)CH\(_2\)O(CH\(_2\)N(NO\(_2\))\(_2\))\(_4\).CH\(_2\)O.CH\(_2\).CH\(_2\)OH; mw 432.31, N 25.92%; no props are given in the patent abstract; was prep'd by treating the tetranitro linear amine having terminal nitroxy or halogen groups with dialcohols with or

Dioxotetrazatriadecane and Derivatives
2,12-Dioxo-4,6,8,10-tetrazotetradecane,
H\(_5\)C.O.CH\(_2\)NH.CH\(_2\)NH.CH\(_2\)NH.CH\(_2\)OH; mw 192.27, N 29.14%; its tetranitro deriv:

2,12-Dioxc-4,6,8,10-tetrazotetradecane, H\(_5\)C.O.CH\(_2\)N(NO\(_2\))\(_2\).CH\(_2\)N(NO\(_2\))\(_2\).CH\(_2\)O.CH\(_3\); mw 372.26, N 30.91%; is described in this Volume, p D1304-L, under 1,9-Dimethoxy-2,4,6,8-tetranitro-2,4,6,8-tetrazanone.

Dioxotriazohendecane or Dioxotriazaundecane and Derivatives
2,10-Dioxo-4,6,8-triazohendecane;
H\(_5\)C.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-Dioxo-4,6,8-trinitro-4,6,8-triazohendecane, or 2,10-Dioxo-4,6,8-trinitro-4,6,8-triazauandecane, H\(_5\)C.O.CH\(_2\)N(NO\(_2\))\(_2\).CH\(_2\)N(NO\(_2\))\(_2\).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-triazahyepane.

Dioxime. The monovalent –HC:NOH radical occuring in two isomeric forms:

<table>
<thead>
<tr>
<th>Configuration</th>
<th>Structural Formulation</th>
</tr>
</thead>
<tbody>
<tr>
<td>anti</td>
<td>-CH</td>
</tr>
<tr>
<td>syn</td>
<td>-CH</td>
</tr>
<tr>
<td>H(_2)N</td>
<td>NOH</td>
</tr>
</tbody>
</table>

Dioximes. A group of compds contg two dioxide radicals: alpha – Glyoximes; beta – Glyoxime Peroxides and gamma – Compounds in which the two oxime radicals are separated by the ethylene radical, such as in succinaldehyde-dioxime, H\(_2\)N.CH.CH\(_2\)CH\(_2\)CH:NOH

1,3-Dioxolane or Glycolmethylene Ether,
(Spelled 1,3-Dioxolane in CA and by Elderfield)
or Glycolformal, O.CH₂•CH₂

CH₂—O ; mw 74.08; flammable col liq. sp gr 1.050 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 prep'd by warming ethylene-
glycol with polyoxyethylene 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

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],

\[\text{CO.NH} \]
N₂.CO.C₆H₃
\[\text{CO.NH} \] ; mw 231.17, N 30.30%;
powd, mp — explodes on heating; insol in ether; was prep'd from the hydrazide,
\[\text{C₆H₃N₂O₃} \] by reaction with NaNO₂ 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

Dioxotetramethylenetetrazolecarboxamide
and Derivatives

1,3-Dioxotetramethylenetetrazole-2-carbox-
amide or 5,7-Dioxo-5,6,7,8-tetrahydro-pyrido-
tetrazole-6-carboxamide (CA name),

\[\text{C(O)=CH₂.C=N} \]
\[\text{N} \] ; mw 195.14,
N 35.89%; no description of compd is given in
abstract; was prep'd 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-acetonyl tetrazole,
\[\text{H₃C.CO.C₆H₃.N:N.N.H} \] which does not
undergo acid or ketone cleavage. The amidic
deriv of parent compd forms an ether,
\[\text{C₆H₅N₂O₂} \] 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-acetonyl tetrazole
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 or-
ganic compound

Dioxy-. An Amer or Brit prefix indicating the
presence of two additional oxygen atoms in an
organic compound

Dioxymonoethylene Perchlorate. See Ethylene-
dioxyamine Perchlorate (Abbrn EDAP)

Dioxybenzene. One of the names for Benzo-
quinone, which is described in Vol 2, pB79-R

Dioxydiphenyl. A Ger name for Biphenol, which
is described in Vol 2, pB122

1,1'-Di o xy-[dixetrazolyl-(5,5')]. A Ger name for 1,1'-Dihydroxy-(5,5'-bis-tetrazole), de-
scribed in Vol 2, pB148-L
DIPAM. Navy code name for 3,3′-Diamino-2,4,6,2′,4′,6′-hexanitrophenyl described in this Vol, pD1133-R. See also S.A. Moses, Ordn 56, 357 (1972)

Dipenta. Code name for Dipentaerythritol Hexanitrato, abbr as DPEHN

Dipentoborylmethane, B₆H₆CH₂B₆H₆; mw 138.35; obtd in a yield of less than 1% by reaction of pentaborane with dichloromethane in the presence of AlCl₃. 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.H₂C)₃.C.H₂.O.C.H₂.C(CH₂OH)₃. It is actually a dehydrated combination of two mol of PE (pentaerythritol), (HO.H₂C)₃.C.H₂.O.H.O.H₂C.C(CH₂OH)₃; mw 254.28; white crs, sp gr 1.365 at 15°, mp 221°; insol in acet, benz, paraffin oils and fats; sol in w = 0.12g in 100g at 15° and 11.64g at 100°; sol 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(CH₂OH)₄, by condensation of formaldehyde with acetaldehyde in the presence of Ca hydroxide

The following reactions take place:

CH₃CHO + HCHO → C(CH₂OH)₃CHO
C(CH₂OH)₃CHO + CH₂O + (½Ca(OH)₂) → C(CH₂OH)₄ + (½Ca(HCOO)₂)

The highest yields (ca 15%) of DPE can be obtd if 3 mol of formaldehyde are reacted with 1 mol of acetaldehyde and 0.5 mol of Ca hydroxide

The procedure of prepn of PE contg DPE was small quantities of polyentaerythrithols, such as TPE (triptentaerythritol) are essentially as follows:

In a stainless steel "reactor" introduced 3 mol 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 mol acetaldehyde, while vigorously stirring and maintaining the temp at 20-25°. After addition of all acetaldehyde and allowing the mix 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 (wring) to separate the crystals of impure PE from the mother liquor, which must be saved because it contains DPE and other PolyPE's such as TPE (triptentaerythritol) and TePE (tretapentaerythritol)

The material left in the Nutsch or in a wringer known as "filter cake" is removed, dried and its mp determined. It is ussly 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 (triptentaerythritol) is in smaller proportion. This product was isolated by Burrell (Ref 9). Its formula is (HO.H₂C)₃.C.H₂.O.C.H₂.C.CH₂.O.CH₂.C(CH₂OH)₃,

\((CH₂OH)₂\) white crs, mp 242-248°. It gives on nitration TPEON (Triententaerythritol Octanitrate), (O₂NO.H₂C)₃.C.H₂.O.CH₂.C(CH₂.O.NO₂)₂ → CH₂.C(CH₂.O.NO₂)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), $\text{C}([\text{CH}_2\text{ONO}_2])_4$, or PETN (Pentaerythritol Trinitrate), called Petrin,

\[
\text{C}([\text{CH}_2\text{OH}])_4
\]

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 crystals of pure PETN (1st crop) which are separated by filtration. The resulting filtrate is concentrated and then cooled. This produced the 2nd crop of crystals. In a similar manner a 3rd crop of crystals could be obtb

The "mother liquor", obtb 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 obtb

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 nitratd 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 Polypentaerythrils:

Wyler & Wemett (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 crystals 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 crystals of double-compound formed. After separating the crystals by filtration, followed by washing with warm w and drying, the product was analyzed and proved to correspond to the formula:

\[
[(\text{CH}_2\text{OH})_3\cdot\text{CH}_2\cdot\text{O}\cdot\text{CH}_2\cdot\text{C}((\text{CH}_2\text{OH})_3)]\sim [4\text{C}((\text{CH}_2\text{OH})_4]
\]

This stable compd was found to be suitable for prepn of esters and resinous compositions for plastics, or it could be nitratd 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$_2$CHO, HCHO and Ca hydroxide. On dehydrating this liquor by adding butyl alcohol, followed by distilling off the azeotropic mixture of BuOH + H$_2$O, a thick syrup was obtb. On cooling it, a large crop of fairly pure crystals 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 nitratd 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 condition of re-
action 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 prep'd, 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 obt'd by nitration of PolyPE's for use in commercial explo's

Burrell & Neidig (Ref 11) described various uses of pentaerythritols

Remensnyder et al (Ref 12) described prep'n 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 crystals was collected. The filtrate was concentrated to sp gr 1.270, cooled and filtered to obtain the 2nd crop of crystals

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 prep'n is as a by-product of the manuf of PE

Marrian & McLean (Ref 16) patented in GB Britain a method of prep'n of DPE from PE

Berlow et al (Ref 20) described prep'n and some props of DPE

Heyden Chem Corp (Ref 17) described props of their technical DPE called Dipentek

Méard & Thomas (Ref 19) listed for DPE heat of combustion and heat of formation (See above)

CondChemDict (Ref 21) gives a brief description of Dipentek

Urban'ski (Ref 22) discusses the prep'n & props of DPE, its hexanitro deriv and its eutectic mixt with PE

Walker (Ref 18) briefly mentions DPE, its eutectic with PE, and a process for separating PE from mixts with DPE, developed by Wyler & Wemett

Accdg to information obt'd from the late Mr George D. Clift, the Trojan Powder Co of Allentown, Penn. 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 am't 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 props of mixtures on knowing their mp's

DPE gives on nitration a powerful expl DPEHN (Dipentaerythritol Hexanitrate), which is described below

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 180C and allow it to remain overnight.

d) Check to be certain that 60% of water are present.

e) Place flask in a water bath at 250, gently shake, remove stopper, and stir by means of a glass rod, the separated DPE crystals, keeping the temp at 250.

f) When the crystals are broken and the mixt has been kept at 250 for at least 10 mins, transfer the crystals 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 crystals with 10ml of ethyl ether, dry at 1100 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 crystals obt'd
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)
3) J.A. Wyler, IEC (Anal Ed.), 18, 777-78 (1946) & CA 41, 863(1946)
6) Evelyn Berlow, R.H. Barth & J.E. Snow, "The Pentaerythritols", Reinhold, NY (1958), 5-6, 16-17, 33-34, 39, 126 & others

Dipentaerythritol Hexanitrate (DPEHN) or Nitrodipenta,

\[(\text{O}_2\text{NO})\text{H}_{3}\text{C}_{5}\text{C.H}_2\text{O.CH}_2\text{O.CH}_2\text{(ONO}_2\text{)}_3\]

mw 524.28, N 16.03%, OB to CO\textsubscript{2} minus 27.5%.

wh crystals, sp gr of crystals 1.63 at 15°, density of cast 1.613 at 15°, loading density at 3000-4000psi 1.59, and of pressed at 2000kgs/cm\textsuperscript{2} 1.51; mp 73.7-75°; Davis gave 72°; bp - explodes above 250°; was first prep'd 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 prep of PE and PETN, isolated DPE of mp 221° and prep'd 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 prep 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 **Nitrate Double Compound of DPE and PE**, \([\text{O}_3\text{NO}\cdot\text{H}_2\text{C}_3\text{C}_2\cdot\text{O}\cdot\text{CH}_2\cdot\text{C}(\text{CH}_3\text{ONO}_2)\text{O}_2\text{H}_2\text{C}_3\text{C}_2\cdot\text{O} \cdot\text{CH}_2\cdot\text{C}(\text{CH}_3\text{ONO}_2)\text{O}_2]\), mw 1788.86, N 17.23%; white solid, prep 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, nitrification 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 min, 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 2.05 parts of DPEHN for 1 part DPE.

DPEHN can also be obtb by nitrating crude PE rich in DPE using either concd nitric acid alone or a mixed nitric-sulfuri acid, followed by extraction of DPEHN by acetone.

Following method of nitrification 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 460 lbs of 98% nitric acid and cooled to 10° (50°F) by circulating brine thru the jacket.

While the acid was stirred, 100 lbs of pulverized coarse PE was introduced slowly by means of a vibrator feeder, while maintaining the temp at 10°. The addition took about 50 min 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 &quot;Nutsch&quot; tilted with acid-resistant material (chemical ware or stainless steel funnel, provided with a stainless steel perforated plate and a fine mesh screen made of chrome steel or fiberglass). 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 &quot;Acid Recovery House&quot;, while the &quot;filter cake&quot; was removed by a operator (wearing a gas mask) using a nonspark metal shovel, into the &quot;Wash Tank&quot; 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 fiberglass 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 &quot;Neutralizer and Crystallizer House&quot;.

The &quot;Neutralizer&quot; 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 DPEHN and 2) the lower consisting of a saturated soln of DPEHN in acetone contg a little water. The bottom layer is removed and treated with an excess of alcohol. Almost pure DPEHN 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 obtb 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 Stetterbacher (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 offenses: 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 offenses and 238.6kcal/mol at C offenses (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); Bureau Mines Apparatus, 2 kg wt 14cm; Picastiny Arsenal Apparatus, 2 kg wt 4 inches for 10mg sample (Ref 24)

Initiation Sensitivity — less sensitive to initia by I.A 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)

Dipentaerythritol Trinitrate Sebacate,
\[ \left( \text{CH}_2 \right)_8 \text{C}(\text{O})\text{OCH}_2\text{C}(\text{CH}_2\text{ONO}_2)_3 \]
\[ \left( \text{CH}_2 \right)_8 \text{C}(\text{O})\text{OCH}_2\text{C}(\text{CH}_2\text{ONO}_2)_3 \text{; mw 708.51,} \]
\[ \text{N 11.86%; viscous clear syrup; was obd when sebatic acid chloride & PE trinitrate were} \]
\[ \text{mixed and heated to } 30^\circ \text{C. There was a fairly} \]
\[ \text{vigorous evolution of HCl. Reaction mixt was} \]
\[ \text{then heated } 4 \text{ hrs at } 50-60^\circ \text{C and allowed to} \]
\[ \text{stand overnight. The material was dissolved in} \]
\[ \text{acetone & repptd with water as an oil which was} \]
\[ \text{separated, dissolved in methylene chloride,} \]
\[ \text{and dried over calcium sulfate. Pentane was} \]
\[ \text{added and the soln alternately chilled & warmed} \]
\[ \text{but no crystal product was obd. Finally, the} \]
\[ \text{soln was evaporated under a stream of air} \]
\[ \text{(Ref 2)} \]
\[ \text{This deriv of PE may have application in the} \]
\[ \text{field of expls} \]
\[ \text{Refs: 1) Beil, not found 2) D.L. Kouba et al,} \]
\[ \text{Hercules Monthly Prog Rept, "High Explosives",} \]
\[ \text{4 Jan 1952 (Navy Contract NOrd-11280, Task A)} \]
\[ \text{3) Not found in later refs thru 1966} \]

Di(pentfluorosulfur)-peroxide. See Bis(pentafluorosulfur)-peroxide in Vol 2, pB152-R

\[ \text{Di(2-pentanone)-cyanohydrazone and Derivatives} \]
\[ \text{Di(2-pentanone)-cyanohydrazone or Bis(methylpropylketone)-cyanohydrazone,} \]
\[ \text{H}_3\text{C.CH}_2\cdot\text{C}:\text{C}:\text{N}:\text{N}:\text{H}:\text{G}:\text{H}:\text{N}:\text{N}:\text{C}:\text{C}:\text{CH}_2\cdot\text{CH}_2: \text{CH}_3; \]
\[ \text{CH}_3 \]
\[ \text{mw 252.36, N 33.30%; may be considered as} \]
\[ \text{the parent compd of its hexanitro deriv, al} \]
\[ \text{though not used to prep it:} \]
\[ \text{Di(or Bis)(5,5,5-trinitro-2-pentanone)-cyanohydrazo} \]
\[ \text{ne or Diminoxalic Acid Bis(5,5,5-} \]
\[ \text{trinitro-2-pentylidene Dihydrazide,} \]
\[ \left( \text{O}_2\text{N} \right)_3\text{C}:\text{CH}_2\text{CH}_2\cdot\text{C}:\text{N}:\text{N}:\text{H}:\text{C}:\text{(NH)}- \]
\[ \text{CH}_3 \]
\[ \text{CH}_3 \]
\[ \text{CH}_3 \]

Dipentaerythritol Hexanitrate

Analytical Procedures and Physical Tests.
There are no US Specifications for analyzing DPEHN.

It is suggested that physical tests described in Ref I and in Vol 1 of Encycl, pp 847ff be used when necessary to study DPEHN

Refs - Analytical Procedures for DPEHN:
2) P. Aubertin & H. Pascal, MP 40, 123 (1958)
3) W.E. Frederick & Frank Prissner, "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) SciMethodsChemAnalysis
28, 6th ed (1963), not found
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 crys, 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

Diphenylacetamide and Derivatives

N,N'-Diphenylacetamide or Ethenydiphenylamide, CH₃C(NH.C₆H₅):N.C₆H₅, mw 210.27, N 13.32%; ndls (from alc), mp 132–33°; insol in w; sol in eth & hot alc. It can be prep'd by treating a cold mixt of 6 moles aniline & 3 moles acetic acid with 2 moles of PCl₃ and heating to 160°. This compd forms crystalline org & inorg salts (Ref 1). See also Ref 6 for prep

Azido, C₄H₁₃N₃, and Diazido, C₄H₁₂N₈, derivs were not found in the literature thru 1966

N,N'-Bis(4-nitrophenyl)-acetamide or N,N'-Di(p-nitrophenyl)-acetamide, O₂N.C₆H₄.N.C(CH₃).NH.C₆H₄.NO₂, mw 300.27, N 18.66%; yol 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 200–100° (Refs 2 & 3). See Refs 4 & 6

It forms a Nitrate salt, C₄H₁₃N₄O₄.HNO₃, powd which dec at 182° without melting; insol in w, alc, acids & alkalis

at 252°); prepd when p-nitroacetanilide was heated 2 hrs at 120° with PCl₅ and p-nitroaniline added and heated at 140° for 3 hrs]
NOTE: No higher nitrated derivs of N,N'-Bis (4-nitrophenyl)-acetamidine were found in the literature thru 1966

N,N-Diphenylacetamidoacetamide or Acetoacetamidoacetamide, C₆H₅—N—C₆H₅
| CO.CH₂.CO.CH₃; mw 253.29, N 5.93%; col. crysts (turning brown in the light), mp 81.5°; very sol in acet., et acet., benz., chl & Cs₂; sl sol in alc & gives a whitish emulsion with w. It can be prepd by interaction of diphenylamine, C₆H₅.NH.C₆H₅, and acetylketene, CH₃CO:CH:CO (Ref 2)

Its heat of combustion at Cᵥ 1955.06 kcal/mol and at Cᵥ 1956.36; heat of formation at Cᵥ 53.25 and Cᵥ 58.57 (Ref 3)
Was suggested by Tavernier & Lamouroux as an ingredient of propellants (Ref 3)

Di(or Bis)phenyl-acetone and Derivatives a,a'-Di(or Bis)-phenyl-acetone or Dibenzylketone, C₆H₅.CH₂.CO.C₆H₅, 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 a-phenyl-a'-benzyl-ethylene glycol with aq sulfuric acid, or by heating a-phenyl-a'-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,
N₂O.C₆H₅.CH₂.CO.C₆H₅,NO₂; mw 300.26, N 9.33%; yellow 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-5,5-bis(4-nitrophenyl)-2-(4-nitrobenzyl)-5,6-dihydrpyran 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,
N₂O₃C₆H₅CH₂.CO.C₆H₅; mw 390.26, N 14.36%; yel ndls (from ethyl acetate), mp — dec 183°; sl sol in alc, glact ac & chl; 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)

Diphenylacetonitrile and Derivatives
Diphenylacetonitrile or Dphenenitrile, (C₆H₅)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,
N₂O.C₆H₅.CH-CN.C₆H₅; 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, (C₆H₅)₂C(NO₂)₂-CN; mw 283.24, N 11.76%; crysts (from MeOH), mp 44-45°; dec on heating above 70°; was prepd by treating a soln of tetraphenylsuccinic acid dinitrile in chl with nitrogen dioxide under anhydrous conditions (Ref 2)
NOTE: No higher nitrated derivs of Diphenylacetonitrile were found in the literature thru 1966
Diphényleactéylene et Dérivés

Diphényleactéylene ou Tolane, C₆H₅C:C.C₆H₅; mw 178.22; monoc. prism, sp gr 0.966 at 100°/4, mp 60–62°, bp 300°; insol. in w; v sol in eth; v sol in hot alc; can be prep. by heating α,α-dichloro-α,α-diphenylethylene with 2 moles of Na methyleate, and by other methods (Ref 1)

Dinitrodiphenyleactéylene, C₁₄H₈N₂O₄; mw 268.22, N 10.45%. The following isomers are found in the literature:

2,2′-Dinitro-diphenyleactéylene,
O₂N.C₆H₄-C:C.C₆H₄N₂O₂; yel. nds (from alc or glc ac atc), mp 189–93°; sol in alc, benz., pyridine & acet.; mod sol in glc ac et & aromatic hydrocarbons; sl sol in hot alc & petr. eth (Ref 2, p330)

3,4′-Dinitro-diphenyleactéylene,
O₂N.C₆H₄-C:C.C₆H₄N₂O₂; crs (from alc), mp 173–174°; sol in hot benz., nitrobenz., acet. & ethyl acetate; sl sol in glc ac et & chlor. & alc; v sl sol in Cl₄, CS₂, eth. & petr. eth (Ref 2, p569)

4,4′-Dinitro-diphenyleactéylene,
O₂N.C₆H₄-C:C.C₆H₄N₂O₂; yel. prlts or nds (from glc ac et or alc), mp 207–11°; sol in concd sulfuric acid with a yel soln; sl sol in hot alc & glc ac et; v sl sol in eth (Ref 2, pp 657 & 320). See Ref 3

2,4-Dinitrophenyl-phenyl-acetéylene,
C₆H₅-C:C.C₆H₄N₂O₂; yel. nds (from glc ac et), mp 112–112.5°; sol in pyridine, chlor., benz., hot glc ac et & hot alc; its soln in concd sulfuric acid gives a blue-violet color (Ref 2, p320). See Ref 5

X-X-Dinitro-diphenyleactéylene, C₁₄H₈N₂O₄, crs (from glc ac et), mp 234° (Ref 2, p569)

3,3′-Dinitro-diphenyleactéylene ou Bis(m-nitro-phenyl)-acetylene, O₂N.C₆H₄-C:C.C₆H₄N₂O₂; no description of compd is given in abstract (Ref 4)

Other props & methods of prep of the Dinitro derivs are found in the Refs. No expl props are reported


DIPHENYLAMINE AND DERIVATIVES

Diphenylamine (DPhA or DPA) or Phenylaniline, C₆H₅-NH.C₆H₅, mw 169.22, N 8.28%, OB to CO₂ minus 27.9%; coll. monocryl crs (from lignin) with an aromatic odor and bitter taste; sp gr 1.150 at 20° & 1.054 at 61°; mp 52.9°, bp 302°; sl sol in w; sol in alc, benz. & lignin. 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

Acd of Refs 1 & 22, DPhA was first prepd in 1864 by A.W. Hofmann on heating rosaniline (fuchsain carbinol base), (CH₃.C₆H₅.NH₂).COH(C₆H₅.NH₂)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°:

C₆H₅.NH₂.H₂O + C₆H₅.NH.C₆H₅ + (ZnCl₂) →

H₂O + C₆H₅.NH.C₆H₅ + (ZnCl₂)

Lab method of prep, 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 disdi 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 successsive recrystallizations from a mixture of ethanol & ligroin, 1:12 by volume, followed by drying in vacuo over phosphorus pentoxide for 5hrs
CondChenDict (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 prep consisting of heating 100 kg aniline with 105 kg aniline hydrochloride in an autoclave at 230°C 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 btw 100 and 200 psi, in the presence of ferric chloride catalyst in an amount btw 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 160 psi.

DPHA is a non-explosive compound 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 propellants. Accdg to Steibach (Ref 13, p197), A. Nobel proposed using DPHA in German propellants 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 Zentralanstelle für Wissenschaftliche Untersuchungen zu Neubabelsberg (near Berlin) (Ref 9). These compounds 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 mixing expls.

Action of DPHA on NC and on other organic nitrates was examined in France by several investigators such as Demougin & Landon (Ref 14), Muravour (Ref 15) and others. Muravour 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 Muravour preferred Centralite to DPHA.

Reactions of DPHA with nitric esters in presence of acetic acid were examined by Parpaillon (Ref 27).

Muravour (Ref 15) showed that pure NC is comparatively stable, but if, for some reason (for instance by action of heat), the decomp starts, the reaction is further accelerated by the products of decomp (N oxides). The role of a stabilizer, like DPHA, is to maintain the rate of decomp within certain limits and to prevent its acceleration. If a stabilizer is not properly chosen or if it is not well mixed in propnl, it might form cmpds with NC, which instead of arresting the decomp will accelerate it. Marqueyról's tests of propellants with various stabilizers have shown that deceptive results may be obt when different stabilizers are compared just at one temp. He recommends that propellants 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°C) of a double-base propnl stabilized with 8.75% Centralite with two other propellants 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 & Crb.

Stability of French propellants using DPHA was also examined by Leclercq (Ref 26a).

Pauling (Ref 23) examined changes in the percentages of DPHA in single and double-base propellants, which have been subjected to vacuum stability tests at 80, 90, 100 and 110°C. Reactions and transformation of DPHA during storage and aging of smokeless propellants 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), Marqueyról & Maravour (Ref 5) and Marqueyról & Lariette (Ref 6) during their studies of DPHA derivatives which impart a dark color to old propellants, that the color is produced by impurities in ether (used in manuf of propnl), or by the oxidizing action of air during drying and storage. Their presence is not an evidence that the propellant has decomposed, but
indicates that a certain amount of DHPhA 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 DHPhAcontg propilins is the formation of Diphenyl-N-nitrosamine,

\[
\text{C}_8\text{H}_4\text{(NO)}\text{C}_8\text{H}_4
\]

The presence of this compound (which is as good a stabilizer as DHPhA itself) was proven in all propilins which were not too old or which had not suffered profound decomposes.

If any mineral acid is present it undergoes a tautomeric change with the formation of p-

Nitrosodiphenylamine, \(\text{C}_8\text{H}_4\text{NH} \cdot \text{C}_8\text{H}_4\text{NO}\).

This compound, a stabilizer itself, reacts with nitric acid and oxygen with the formation of 2,4'- and 4,4'-Dinitrophenylamines.

\[
(\text{O}_2\text{N})\text{C}_8\text{H}_4\cdot \text{NH} \cdot \text{C}_8\text{H}_4\text{(NO)}_2
\]

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'-Trinitrophenylamine,

\[
(\text{O}_2\text{N})_2\text{C}_8\text{H}_4\cdot \text{NH} \cdot \text{C}_8\text{H}_4\text{(NO)}_2
\]

Besides the above compounds, the presence of the following were also reported, especially when propilins were stored at elevated temperatures, such as at 100°C:

- 4-Nitrophenylamine, \(\text{C}_8\text{H}_4\text{NH} \cdot \text{C}_8\text{H}_4\)
- 4-Nitrophenyl-N-nitrosamine,

\[
(\text{O}_2\text{N})\text{C}_8\text{H}_4\cdot \text{NH} \cdot \text{C}_8\text{H}_4\text{(NO)}_2
\]

- 4,4'-Dinitrophenyl-N-nitrosamine,

\[
(\text{O}_2\text{N})\text{C}_8\text{H}_4\cdot \text{NH} \cdot \text{C}_8\text{H}_4\text{(NO)}_2
\]

- 2-Nitrophenylamine, \(\text{C}_8\text{H}_4\text{NH} \cdot \text{C}_8\text{H}_4\)
- 2-Nitrophenyl-N-nitrosamine,

\[
(\text{O}_2\text{N})\text{C}_8\text{H}_4\cdot \text{NH} \cdot \text{C}_8\text{H}_4\text{(NO)}_2
\]

- 2,4-Dinitrophenyl-N-nitrosamine,

\[
(\text{O}_2\text{N})\text{C}_8\text{H}_4\cdot \text{NH} \cdot \text{C}_8\text{H}_4\text{(NO)}_2
\]

All of these compounds were detected colorimetrically and more recently their presence was verified by the Chromatographic Method of Tawett, which is described under Chro-

MATOGRAPHY in Vol 3 of Encycl., pp C289-R to C298-L.

According to Davis and Ashdown (Refs 2 & 7), the propilin is usually nearing the end of its stable life when a Dinitrophenylamine is present and the propilin is practically worthless (or soon becomes so) when a Trinitrophenylamine is present.

The use of DHPhA in propilins containing NG is not recommended on account of the formation of tarry oxidation products (Ref 8) but, accdg to Mr E.F. Rees, formerly of PicArsn, some double-base propilins examined in his lab contd DHPhA.

Owing to the basic character of DHPhA, it must not be used in large quantities (Ref 19). It was proven by Marqueyr (Ref 10) and also by Demougin & Landon (Ref 14) that DHPhA attacks NC slightly even at a tempe as low as 40°C but at 75°C and higher temps the reaction is quite perceptible.

It should be noted that DHPhA used in smokeless propellants must be fairly pure and practically free of primary amines, because they attack NC more readily than DHPhA.

Presence of DHPhA may be detected by any colorimetric tests described under Analytical Procedures (See next item).

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 decompos.
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. Diphenylinitrosamine 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 propuln gives these tests.

Pérez Apr (Ref 19, p438) describes identical tests and states that if a propIn originally stabilized with DPHA gives negative results for the presence of both DPHA and DPHNitrosamine, such a propIn should be considered unfit for 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 propIn 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 de-

scribed 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 (prefd 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 obtb 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 K₂Cr₂O₇.** 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 sec.

**Test 3. Color Test with Selenious Acid in Concentrated HCl.** This test was developed at PicAtsn (Ref 13) to make it suitable for testing foreign propulns which contain stabilizers interfering in tests 1 & 2. Test 3 is rather specific for DPHA because Acardite, Centralite, DPHUrethane, EtPHUrethane, NG, DEGN 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 obtb when DPHA is present.

**Quantitative Tests.**

**Test 1. DPHA Nitration Method.** Cut a sample of propIn 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 glc 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\(^{\circ} \) 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\(^{\circ} \) 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\(^{\circ} \) for 1 hr and weigh within 0.2mg.

\[
\text{% DPhA} = \frac{\text{Wt of ppt on crucible} \times 42.59}{\text{Wt of sample (ca 5g)}}
\]

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 dgrep DPhA content of newly manufd propnts 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 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\(^{\circ} \) 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 tetrabromodiphenylamine (0.6ml of Br\(_2\) for each percent of DPhA in propnt). A slight excess of Br\(_2\) 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\(^{\circ} \) 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: \( A = \text{increase in wt of crucible and} \)
\( W = \text{wt of sample (ca 5g) corrected for} \)
\( \text{total volatiles} \)

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 dgrep the DPhA content of newly manufd single-base or double-base propnts contg no other brominatable material (such as salicylates, phenols, 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 glacial 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 glacial 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 glacial 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 glacial 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 distilled water 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 glacial AcOH, and exactly the same vol of the potassium bromate-bromide soln

\[
\frac{W}{N} = 2.115(A-B)
\]

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 Diphenylaminemalonic acid. 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 50μg 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 apparatus 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 saturated with HCl from flask (7). Dry the stick and the flask first by suction and then in a 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 (W₂). If the wt of sample is W₁ (ca 5g), then:

\[
\% \text{DPhA} = \frac{82.3W₂}{W₁}
\]

Note: If the sample contains a large amt of Nitrosodiphenylamine, a small amt of it ppt 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 16
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 prep'd 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 pet ether and remove the bulk of ether 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 pet ether. Pour the soxl on the column and apply gentle suction. Wash beaker with two small successive portions of 1:4-benz-pet ether 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-pet ether mixt on the column and then wash the benz from the column with a final wash of 30ml of pet ether.

Remove enough of pet ether 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 indensation 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 ether, 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 tetrabromodiphenylamime in an accurately tared sintered glass crucible. Dry the ppt for 1 hr at 100°C, 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 derg the DPhA content of newly manuf'd 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 30ml of ether alone.

Place both flasks on the steam bath, maintained at ca 75°C 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.
$W = \text{Weight of sample (ca 5g) corrected for total volatiles}$

**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:

$$\text{O}_3\text{S} \xrightarrow{\text{N}_2} \text{NH} \rightarrow \text{O}_3\text{SH} \xrightarrow{\text{N} = \text{N}} \text{Orange IV (Tropeolin)}$$

Some primary amines (aniline, toluidine) do not interfere, others (o-anaphthylamine) do.

The reagent (a soln of diazosulfanilic acid in aq sulfuric acid) was prepared in the following manner;

a) In the 1st operation, the commercial sulfanilic acid was purified by suspending 1 g of acid in 6 ml of w and heating the mix 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 sulfanilic acid was pptd in the form of small white grains.

b) After cooling the above mixture, the acid was diazotized by adding 3 ml 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) Conc. 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 100 ml volumetric flask. After filling the flask to the mark and mixing, measured quantities of this “standard” were placed in several 50 ml flasks. After adding to each flask 5 ml of w, 2 ml 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 50 ml mark and, after mixing, the colorimetric readings taken. Quantities as low as 0.02 mg of DPHA in 50 ml may be detected by this method.

**Note:** Low readings might result if the time of standing at or temp was less than 2 hrs. If, however, the soln in 50 ml flask was heated to 50°C 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 by 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 0.09 mg DPHA per 50 ml 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 described in this specification:
1) Setting Point = 51.7 to 53.0°
2) Insoluble Matter (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 distilled water. Stopper (loosely) immediately and shake vigorously for 10 minutes. Cool to 25° and filter the supernatant liquid, retaining the DPHA in the flask. Repeat the extraction with 50ml of boiling distilled water exactly as above. To the combined filtrates add a few drops of phenolphthalein indicator and observe the color. If the solution remains colorless, titrate it with 0.1N alkali and if it turns red, titrate with 0.1N acid. Make a blank titration on the distillate used and correct the titration readings for any acidity or alkalinity found in it. Calculate the acidity to HCl and alkalinity to NaOH. Save this solution for the next test.
6) Oxidizable Material (Aniline and its Salts). Pipet 25ml of 0.1N bromide-bromate solution into the Erlen flask containing the solution 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 0.1N Na thiosulfate soln using starch as indicator. Make blank determination on 25ml of 0.1N bromide-bromate soln exactly as above and calc the aniline from the formula listed below:

\[
\% \text{Aniline} = \frac{[(V_N - v_N) - 0.033] 	imes 1.551}{w}
\]

where: \( V = \text{ml Na thiosulfate used in blank} \)
\( v = \text{ml Na thiosulfate used in titration of sample} \)
\( N = \text{Normality of Na thiosulfate} \)
\( 0.033 = \text{Correction for DPHA dissolved in w} \)
\( w = \text{Weight of sample (20g)} \)

Refs for DPHA Analytical Procedures
1) O. Turek, Chemickyí Obzor 1, 295 (1927) & CA 22, 4819 (1928) (Volumetric detn of DPHA in smokeless propulnts) 2) H. Levenson, JIEC (Anal Ed) 2, 246 (1930) (Volumetric bromination determination of DPHA in NG propulnts in presence of Centr) 3) O.C. El-lington & H.B. Beard, JSCI 50, 1517 (1931) (Royal Arsenal Method of volumetric bromination for detn of DPHA in propulnts) 4) S.G. Cook, IEC (Anal Ed) 7, 250-55 (1935) (Detn of DPHA in smokeless propulnts) 5) A. Bresser, SS 30, 42-3 (1935) (Detn of DPHA in propulnts) 5a) G.C. Hale, Army Ordn 5, 674-75 (1935) 6) F. Deschaux, Rev Gen Mat Plastiques 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 propulnts) 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 Idib 2 (1943), p D11 (Description of the following procedure which was recommended by Buission in "Le Problème des Poudres", Paris (1913), p126 for identification of different products of decompn of propulnts: "Digest for 24hrs 10g of finely divided propulnt 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
DIPHENYLAMINE DERIVATIVES

Diphenylamine-4-oxide; 4-Azidodiphenylamine
or N-Phenyl-4-azido-diphenylamine, \( \text{C}_8\text{H}_8\text{N} = \text{NO}, \text{C}_8\text{H}_8\),
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 interd of hydroxylamine-hydrochloride, \( \text{NH}_2\text{OH}, \text{HCl} \), with 4-nitrosodiphenyl-
amine, \( \text{ON}, \text{C}_8\text{H}_4, \text{NH}, \text{C}_8\text{H}_4 \), in Na carbonate
sln (Refs 1 & 2)

Forms various explosive salts and nitroso
compd:

4-Azidodiphenylaminitrosamine or N-Nitroso-4-
azido-diphenylamine, \( \text{N}_2\text{C}_8\text{H}_4\text{N(NO)}\cdot \text{C}_8\text{H}_8 \),
mw 239.24; N 29.28%; yel leaflets (from alc);
mp 55° (decomp slightly above mp); bp —
explodes on heating on 
A Fe foil; can be prepd
by treating the acetic acid soln of 4-azidoDPhA
with Na nitrate at 0° (Refs 1 & 2)
Refs: 1) Beil 12, [429] 2) A. Angel & A. 
Pieroni, Atti Accad Lincei Mem [5], 31, 152 & 
453-54 (1923); JCS 124, 1, 612 & 1026 (1923);
CA 18, 1119-20 (1924)

Diphenylamine, Diazocompounds and Their
Nitroated Derivatives. See under Diazodiphenyl-
amine and Derivatives in this Vol and also:
F. Steppes, GerP 291156 (1915) & CA 11, 899
(1917) (Several expl compgs contg nitratd dia-
zo compounds of DPhA were proposed)

Diphenyldiazonium Hydroxide and Deriva-
tives. See Anilinobenzodiazonium Hydroxide
and Derivatives in Vol 1 of Encycl, p A421

Diphenylaminonitroso Derivatives:

Diphenylaminitrosamine or N-Nitroso-diphenylamine,
\( \text{C}_8\text{H}_8\text{N} = \text{NO}, \text{C}_8\text{H}_8 \), mw 198.22, N 14.13% yel
or om crysts; sp gr 1.23; mp 66-67°; insol in w;
sol in benz, alc, acet and ethylene di-
chloride; can be prepd by treating DPA with
N dioxide in abs ether and cooling the re-
action mix (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) CondChemDirt (1961), 807-R
\( \rho \)-Nitrosodiphenylamine, \( \text{ON}, \text{C}_8\text{H}_4, \text{NH}, \text{C}_8\text{H}_4 \),
mw 198.22, N 14.13%; gen plts (from benz); mp
144-145.9°; sl sol in w & ligroin; very sol
in alc, eth & chlor; sol in benz; can be prepd
by transposition of Diphenylaminitrosamine fol-
lowing the method of Goldschmidt and Wurz-
schmidt (Refs 1 & 2), using hydrogen chloride
in alc + ether

\( \rho \)-NitrosodPPhA 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 \( \alpha \)-naphthylamine, but an orange
color is produced if soln is heated. One-
tenth of a mg of nitroso imparts an intense blue color-
ation to a few mls of concd sulfuric acid (Ref
3 and Ref 4, p.312). Méard & Thomas (Ref 5)
detd its heat of formation at \( \text{C} \), 49.2 kcal/mol
and heat of combustion as 1520.8 kcal/mol
3) T.L. Davis & A.A. Ashdown, JACS 46,
1052(1924) 4) Davis (1943), 312 5) L.
Méard & M. Thomas, JACS 38, 50 (1956) & CA
50, 3763 (1956)

Mononitrodiphenylamines, 
\( \text{N}_2\text{C}_8\text{H}_4\text{NH}, \text{C}_8\text{H}_4 \), 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 & Rabassier (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 & Tranqueta (Ref 4) detd its pres-
ence in exps by extracting with methylene
chloride, dissolving the residue in gl gl acet
ac, brominating in the presence of \( \text{CCl}_4 \) and
rattinating the excess Br with thiosulfate

Tavemir & Lamouroux (Ref 5) detd its
\( Q^\circ = 1488.33 \text{kcal/mol} \) and \( Q^\circ = \text{minus 16.71}
\text{kcal/mol} \)

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 re-

requirements in MIL-N-3399A (Ref 7)
This method is used for deter the 2-NDPhA content of proplts contg nitrate esters or nitrocompds. Other brominatable compds must be absent. Prepare the proplt and extract a 5g portion as described in Vol 2 of Encycl, p C131. Dissolve the dried residue in the extraction flask contg 10ml glc 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 CCl₄ 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 distilled 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, glc AcOH and carbon tetrachloride.

% 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 proplts contg no other nitrocompds.
Prepare and extract a 5g sample of proplt 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 glc AcOH.
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.
Titratre 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.

% 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 propplts 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 propplts.

**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 vol volume with ethanol in each flask  

Fill absorption cells of spectrophotometer or colorimeter with above solns, measure the absorp 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 propplt soln in relation to reference and calc the percentage of 2-NDPhA in the propplt  

US Military requirements and tests for 2-Nitrodiphenylamine intended for use in the manuf of propplts 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 73.0º 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 95º for 15 mins, and transfer the liquid to the tube C (See Fig on p D1473) filling it to within ca 1.5 inches of the top. Close the tube with stopper (a), provided with standard thermometer T, correction thermometer T1 and stirring rod R. Immerse the stoppered tube in a water bath at 85-90º 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 0.2000g sample to a dry 500ml titration flask, add 40ml of glacial AcOH and connect with a CO2 generator, similar to H2 generator shown on p A416-L of Vol 2. After displacing air in the flask with CO2, add slowly, with constant swirling, 25ml of soln contg 1 g of CH3COONa.3H2O per ml, followed by exactly 50.00ml of ca 0.2N Titanous chloride soln. Mix thoroughly by swirling for at least 15 secs, add 5ml of 20% NH4SCN soln and titrate with ca 0.15N ferric almn soln until an amber color, which persists at least 30 secs, is produced. Determine concurrently a blank prepby using the same ingredients as above, except the sample  

\[
Purity(\% 2\text{-NDPhA}) = \frac{3.570(V-v)N}{W}
\]

where:  
V = ml of ferric almn used for blank  
v = ml of ferric almn used for the sample  
N = normality of ferric almn  
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 glacial 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.

\[
Purity(\text{% 2-NDPhA}) = \frac{3.570(V-v)N}{W}
\]

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\(^\circ\)C 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\(^\circ\)C 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\(^\circ\)) 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\(^\circ\). 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% phpt indicator and titrate to a light pink end point with 0.1N Na (or K) hydroxide soln.

\[
Acidity (mg KOH per g) = \frac{56.1VN}{W}
\]

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


Other mononitro derivatives are not of such importance. They are:

3-(or meta)-Nitrodiphenylamine, red lfts, mp 112-14\(^\circ\)

Ref: Beil 12, 702, (346) & (377)

4-(or para)-Nitrodiphenylamine, pale-yel ndls, mp 132-33\(^\circ\); was isolated by Becker & Hunold (Ref 2) as one of the products formed during aging of NC proplts stabilized with DPhA. It was proposed as a stabilizer in smokeless proplts.


Nitrodiphenyllnitrosamines,
\(\text{O}_2\text{N-C}_6\text{H}_4\text{N(NO)C}_6\text{H}_4\); mw 243.22, N 17.28%.

Two isomers were isolated by Becker & Hunold (Ref 3), three are known:

2-Nitrodiphenyllnitrosamine, tablets (from
MeOH), or plts (from alc), mp 99–101.1°C; sol in glacial ac, acet, toluene, 95% alc and in 95% MeOH (Ref 1). Methods of prep & other props are found in Refs 3, 4-Nitrodiphenylmethanamine, ndls, mp 89–90°C; readily sol in chl & acet; sl sol in alc & glacial ac; soln in concd sulfuric acid is violet in color (Ref 2).

4-Nitro-phenylnitroaniline, crystals (from chl) or prisms (ndls) (from glacial ac or xylene), mp 130–33°C; sol in cold salvs (Ref 3).

Other props & methods of prep are found in the Refs:

Refs: 1) Beil 12, 679 & [374]; 2) Beil 12, [383]; 3) Beil 12, 728, (335) & [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, Anal Chem 23, 1740 (1951) & CA 46, 5434 (1952) (UV & visible absorption spectra in alc of derivs of DPA & others).

Dinitrosodiphenylamines, C_{15}H_{12}N_{2}O_{4}, 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-gm plts (from eth), mp ca 98°C (dec); sol in alc, eth & benz; insol in w; was prep by treating a soln of 4-nitrosodiphenylamine in alc & concd HCl with qaq NaNO_3 soln (Refs 1 & 2). Refs: 1) Beil 12, 686; 2) P.W. Neber & H. Rauscher, Acta Chem Scand 5, 3411 (1943); 3) American Cyanamid Co, Brit P 824627 (1959) & CA 54, 9345 (1960) (Improves dispersion of C black in rubber vulcanizates by incorporating 0.5–2.0% of the dinitroso compound). 4) C.C. Tung, USP 3626425 (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.

Dinitrophenylamines, C_{18}H_{12}N_{2}O_{4}, mw 259.22, N 16.21%. Following isomers are listed in the literature:

2,2'-Dinitrophenylamine,

O_2N.C_6H_4-NH.C_6H_4.NO_2; golden-yel lfts, mp 166–69°C. 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°C. Refs: 1) Beil 12, 690 & [341]; 2) P. Juillard, Bull(Fr) 33, 1175 & 1179 (1905).

**2,4-Dinitrophenylamine,**

(O_2N)_2C_6H_4.NH.C_6H_5; red ndls (from benz), mp 157°C; sol in acet, chl & 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 as 1445.1 kcal/mol and Q_V as minus 7.23 kcal/mol. Refs: 1) Beil 12, 751, (362) & [407]; 2) P. Juillard, Bull(Fr) 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'-Dinitrophenylamine,

O_2N.C_6H_4.NH.C_6H_4.NO_2; brick-red crstls, mp 220–221.5°C; 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 propellants stabilized with DPH. 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-Dinitrophenylamine,

(O_2N)_2C_6H_4.NH.C_6H_5; red-orn crstls, mp 106–108°C; sol in alc & acet; can be prep by heating 2-chloro-1,3-dinitrobenzene with aniline and Na acetate in alc. Refs: 1) Beil 12, 738, (365) & [413]; 2) K. van Aubers & E. Breze, Ber 59, 553 (1926).

3,3'-Dinitrophenylamine,

O_2N.C_6H_4.NH.C_6H_4.NO_2; om plts, mp 185.5°C; bp ~ expl mildly; insol in w and in ligroin; sol in cold pyridine & NBenz; sl sol in chl, alc, eth & glc ACOH; can be prepd by interaction of 3-bromo-1-nitrobenzene and 3-nitrococanilide. Refs: 1) Beil 12, not found; 2) H. Alpert & W. Linnel, JCS 1936, p91; 3) H. Hodgson & D. Dodgson, JCS 1948, pp 1004-06.
D 1432


4,4'-Dinitro-diphenylbenzene,
O₂N₃C₆H₄·NH·C₆H₄·NO₂; yel nlds with bluish reflex, mp 216–216.5⁰; 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 proplasts stabilized by DPhA. It gives on nitration explosive 2,4,4'-Trinitro(diphenylamine (Ref 4)

Refs: 1) Beil 12, 716, (351) & [387] 2) T.L. Davis & A.A. Ashdown, JACS 46, 1051 (1924)

Dinitro-N-nitroso-diphenylamine, C₁₂H₈N₄O₆; mw 288.22, N 19.44%. Four isomers are described in Beil:
2,2'-Dinitro-N-nitroso-diphenylamine (called Bis-(2-nitroso-aminonaphthalene), O₂N₂C₆H₄·NO₂C₆H₄·NO₂; not obtb in pure state; dec in alc to 2,2'-Dinitro-DPhA; obtb with the 2,4'-Dinitro-N-nitroso deriv by nitrating 2-nitro-N-nitroso-diphenylamine in glacial ac with nitric acid below 10⁰ (Ref 1)

2,4'-Dinitro-N-nitroso-diphenylamine,
O₂N₂C₆H₄·NO₂C₆H₄·NO₂; dk rose colored. phtls, mp 160–62⁰ (dec); sol in alc, v sl sol in glacial ac, alc & MeOH; obtb with other products on nitratibg Diphenylnitrrosamine with 2 moles of nitric acid (d 1.43) (Ref 2)

4,4'-Dinitro-N-nitroso-diphenyle, O₂N₂C₆H₄·NO₂C₆H₄·NO₂; dk orn-yel prisms, mp 150⁰ (dec); v sl sol in alc, tolueene & glacial ac; obtb with tetratitro deriv on treating 4-nitrodiphenylnitrosamine with 6 moles of nitric acid (d 1.43) (Ref 3)

2,4'-Dinitro-N-nitroso-diphenyle, (O₂N₂C₆H₄·NO₂C₆H₄·NO₂; fit yel prisms (from chlf), mp 149–51⁰; sol in concd sulfuric acid giving an orn-yel color; prepd by treating for several days 2,4-Dinitro-diphenylamine with isosamyl nitrite in glacial ac at 80⁰ (Ref 4)

Other props & methods of prep diphenyl-

Dinitro-N-nitroso-diphenylamine are found in the Refts


8) W.A. Schroeder, JACS 73, 1122 (1951) & CA 45, 6004 (1951) (H bonding & relative adhsption affinities on silicic acid of nitratibg derivs of DPhA) 9) W.A. Schroeder et al., Anal.Chem 23, 1740 (1951) & CA 46, 5434 (1952) (UV & visible adabsorption spectra in alc)

4,4'-Dinitro-diphenylisitramine; Bis (4-nitrophenyl) -nitramine; or N,N',4,4'-Trinitrodiphenyl-

amine, O₂N₂C₆H₄·NO₂C₆H₄·NO₂; mw 304.22, N 18.42%; cryssts, dec on crystall from ethyl acet forming 4,4'-Dinitro-diphenylamine; was prepd by action of NO on 4,4'-Dinitro-
diphenyl nitrogen oxide, (O₂NC₆H₄·NO₂ NO, in cold chlf (Refts 1 & 2)

Refs: 1) Beil 12, [396] 2) H. Wieland & K. Roth, Ber 53B, 227 (1920) & CA 14, 2638 (1920)

Trinitrodiphenylamines, C₁₂H₈N₄O₆; mw 304.22, N 18.42%. Following isomers are described in the literature:
2,4,4'-Trinitrodiphenylamine,
(O₂N₂C₆H₄·NO₂C₆H₄·NO₂; yel prisms, mp 186–186.2⁰; 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 175⁰ (Refts 1 & 2)

Refs: 1) Beil 12, 752 & [408] 2) T.L. Davis & A.A. Ashdown, IEC 17, 675 (1925)

2,4,3'-Trinitrodiphenylamine,
(O₂N₂C₆H₄·NO₂C₆H₄·NO₂; yel nlds (from glacial ac) or gm-yel cryssts (from acrat or pyridine), mp 193–95⁰; readily sol in alc, glacial ac & acetic hydrhde; mod sol in benz, chlf, alc, eth & CS₂; sl sol in petr eth; was prepd by reaction of 4-chloro or 4-bromo-

1,3-dinitrobenzene & 3-nitroaniline, and by other methods (Refts 1 & 2)


2,4,4'-Trinitrodiphenylamine,
(O₂N₂C₆H₄·NO₂C₆H₄·NO₂; yel nlds, mp 186–186.7⁰ (dec); sol in benz, acar anhyd, alc, chlf & eth. It is one of the products
isolated by Davis & Ashdown (Ref 2) and Becke & Hunold (Ref 3) from aged NC proplnts stabilized by DPHa. It can be prep from re-
action of 4-chloro-1,3-dinitrobenzene & 4-
nitroaniline in alc at 150°C
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'-Trinitrophenylamine,
(O2N)2C6H4-NH.C6H4.NO2; crys (from glaq-
acet ac), mp 220-222°; was prep'd when 2,6-
nitroaniline & o-nitroiodobenzene in tech
nitrobenz in the presence of Cu powd &
K2CO3 were heated at 205-10° for 10 mins.
Yield 44% (Ref 2)
Refs: 1) Beil, not found 2) B. Hgedus, Herv
33, 770 (1950) & CA 44, 8955 (1950)
2,4,6-Trinitrophenylamine or Picrylamine,
(O2N)3C6H4.NH.C6H4; om-red ndls (from eth-
acet) or om colored crys (from benz, acet
or alc), mp 179-80°; was prep'd by warming
Picrylchloride & aniline in alc, and by other
methods (Reps 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-Trinitrophenylamine, C12H4(NO2)3,
yel ndls (from acetic acid), mp 135°; readily
sol in alc; glaq acet ac, eth acet & benz; sol in
CS2; insol in w; was obt by heating acetyl
phenylamine with nitric acid (c 1.05)
Refs: 1) Beil 12, 247 2) J.C.1l1l & Allen,
Ber 18, 1997 (1885)
3,3,3-Trinitro-N-nitrosophenylamine,
C12H4N5O7; mw 333.22, N 21.02%; yel ndls
(from acet + chl), mp 370° & softens at 179°
& melts at 184-85°(dec); sol in chl & glaq acet
ac; s1 sol in alc; y s1 sol in acet; gives with
alc KOH a blood-red soln; was prep'd by pro-
longed treatment of 3-nitro-N-nitrosophenyl-
amine with 3 moles of nitric acid (d 1.42) in
glq ac acid (Reps 1 & 2). No expl props are
reported
Refs: 1) Beil 12, [383] 2) J.Ryan & T.
Glover, ProcRoyIrishAcad 34B, 104 (1918) &
CA 12, 2542 (1918)
Tetranitrodiphenylenamines (TeNDPha),
C12H4N5O4, 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. Lawrence, TransRoy-
SocCanada, 12, III, 7-11 (1918) & 13, 1452
(1919):
Although nitration of DPHa with nitric
acid is difficult, it could be accomplished by
placing 1 mol of DPHa in a large beaker sur-
rounded by ice-water and adding slowly, with
stirring, 8 mols of conc 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 re-
sulting ppt proved to be a Tetranitrodiphenyl-
amine of unknown structure. Its mp was above
250° and it was s1 sol in eth, acet chl and
alc; insol in petr eth and in CCl4
The same investigators reported the prep 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 191-92° was obt. The product was
insol in petr eth, eth and carbon tetrachloride;
s1 sol in alc & chl 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'-Tetranitrodiphenylenamine,
(O2N)2C6H4-NH.C6H4(NO2)2; red-bm lifts (from
alc); yel ndls or prisms (from glaq AcOH); mp
199-201.5°; diff sol in cold alc, eth acet or
toluene; sol in acet (2.6% at 17°); solubilities
in other solvents were det by L. Desvernes;
they are listed in Ref 1, p [409]; can be prep
by nitration of dinitrophenylamines (Reps
1 & 2) or by nitration of DPHa by HNO4+ClC13-
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. Lawrence, 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 & re-
late adsorption affinities) 8) W.A. Schroeder

2,4,6,2'-Tetranitrodihenylene or Picryl-2'-nitroaniline, \( \text{O}_2\text{N}_3\text{C}_6\text{H}_2\text{NH}_2\text{C}_6\text{H}_4\text{NO}_2 \); golden-plastic needles, mp 210–213°; sol in hot abs alc.; diff sol in cold alc.; insol in eth.; can be prep by treating Picrylchloride with 3-nitraniline. It is a mild expl; gives on nitration 2,4,6,2',3',4'-HexanitroDPhA

Refs: 1) Beil 12, 765 2) P. Juillard, Bull(Fr), [3], 33, 1188 (1905) 3) J.B. Tingle & W.A. Lawrence, TransRoyaSocCanada 12, III, 7–11 (1918) & CA 13, 1462 (1919)

2,4,6,3'-Tetranitrodihenylene or Picryl-3'-nitroaniline, \( \text{O}_2\text{N}_3\text{C}_6\text{H}_2\text{NH}_2\text{C}_6\text{H}_4\text{NO}_2 \); orangy crystals (from eth alc.), mp 210–213°; sol in hot abs alc.; diff sol in cold alc.; insol in eth.; can be prep by treating Picrylchloride with 3-nitraniline. It is a mild expl; gives on nitration 2,4,6,2',3',4'-HexanitroDPhA

Refs: 2) (?). Austen, Ber 7, 1248 (1874) 3) E. Wedekind, Ber 33, 431 (1900) 4) J.B. Tingle & W.A. Lawrence, TransRoyaSocCanada 12, III, 7–11 (1918) & CA 13, 1462 (1919)

2,4,6,4'-Tetranitrodihenylene or Picryl-4'-nitroaniline, \( \text{O}_2\text{N}_3\text{C}_6\text{H}_2\text{NH}_2\text{C}_6\text{H}_4\text{NO}_2 \); golden-yellow flts or prisms; mp 222–223°; insol in cold alc.; sol sol in eth., toluene, dfl., AcOH & carbon tetrachloride; sol in acet. & eth. alc.; can be prep by nitration of Picrylaniline with mixed nitric-acetic acid or nitric-oxalic acid. It gives on nitration 2,4,6,2',3',4'-PentanitroDPhA

Refs: 1) Beil 12, 765 (369) & [422] 2) P. Juillard, Bull(Fr), [3], 33, 1188 (1905)

2,4,3'-X-Tetranitrodihenylene, \( \text{O}_2\text{N}_3\text{C}_6\text{H}_2\text{NH}_2\text{C}_6\text{H}_4\text{NO}_2 \); yel prisms (from alc + acet.), mp 190°; diff sol in alc.; sol in chfl; very sol in acet.; was obtd after leaving for one week an acetic acid soln of 2,4,3'-TrinitroDPhA with iso-amyl nitrite. This seems to be the same as the unidentified product of mp 191–192° obtd by Tingle & Lawrence (See above)

Refs: 1) Beil 12, (369) 2) H. Ryan & T. Glover, ProcRoyIrishAcad 34B, 104 (1918) & CA 12, 2542 (1918)

Pentanitrodihenylene, \( \text{C}_12\text{H}_6\text{N}_6\text{O}_{10} \); mp 394.22, N 21.32%. Following isomers are described in the literature:

2,4,6,2',4'-Pentanitrodihenylene or Picryl-2',4'-dinitroaniline, \( \text{O}_2\text{N}_3\text{C}_6\text{H}_2\text{NH}_2\text{C}_6\text{H}_4\text{NO}_2 \); yel prisms (from toluene), mp 196–197°; explodes on heating above its mp; diff sol in cold alc., toluene or AcOH; can be prep by reaction of Picrylaniline with nitric acid (d 1.49) at RT for 24 hrs

Foms salts, some of them expl


2,4,6,3',4'-Pentanitrodihenylene or Picryl-3',4'-dinitroaniline, \( \text{O}_2\text{N}_3\text{C}_6\text{H}_2\text{NH}_2\text{C}_6\text{H}_4\text{NO}_2 \); bm-yel crs (from AcOH); mp 232°; explodes on heating above mp; solubilities not given; was prep 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 Duijn & B. van Lennep, Rec 38, 363 (1919)

Hexanitrodihenylene, \( \text{C}_12\text{H}_6\text{N}_6\text{O}_{12} \); mw 439.22, N 22.33%. OB to CO₃ minus 52.8%.

Following isomers are described in the literature:

2,4,6,2',4',6'-Hexanitrodihenylene (HNDPhA); Dipicrylamine, Hexite, Hexil or Hexyl [Hexa, Hexamin, Hexanitrodihenylene, Hexyl or SWA (Schellscholle Neuer Art) (New kind of Guncotton in Ger)]; (Hexanitrodihenylene in Fr); (Eanitrobenzenila oder Exil in Ita); (Hexanitrodifenilamina o Exilna in Span); (Gheksanitrodifenilamina in Rus), \( \text{O}_2\text{N}_3\text{C}_6\text{H}_2\text{NH}_2\text{C}_6\text{H}_4\text{NO}_2 \); 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 chl 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 prep'd 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 methylidiphenylamine. Mertens (Ref 4) was the first to prep it by direct nitration of DPHA. He used fuming nitric acid mixed with coned sulfuric. Julliard (Ref 7) also nitrated DPHA to hexanitro, but found that it was more convenient to nitrate 2,4-DNDDPA.

The large scale prep 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:

\[
2C_8H_6N + 2Cl\cdot C_8H_5(NO_2)_2 + CaCO_3 \rightarrow H_2O + CaCl_2 + CO_2 + H_2O + 2C_8H_6NH\cdot Ca\cdot C_8H_6(NO_2)_2
\]

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-DNDDPA 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 nitrification is followed by observing the color change from red of the dinitrocompd to yellow of the tetra-nitrocompd. 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), Tertiary and aniline in warm acetone (or benzene) furnish methylaniline and Tri-nitrodiphenylamine (TNDPhA). Then the latter is nitrated to HNDPhA.

Stettbacher (Ref 22, p292) described the following one-stage laboratory method of nitrification 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 crystals 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 86g. 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 prep 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 1000 kg of 99% nitric acid placed in a V2A stainless steel nitratior of 2 cu meters capacity (fitted with an agitator rotating at 60 RPM, a cooling jacket and cooking coils) 300 kg 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 nitratior 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 crystals were transferred to a "tray-drier" and then to screens. The yield was 420 kg per 300 kg of DPhA.

HNDPhA was also 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 40 kg "skip bomb" known as "Kurt Apparatus" (See Ref 44, p 14,17, 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 WWII, mostly TNT. A mixture of HNDPhA with Al powder was known as Schiesswolle 18 or TSMVI-101 (Ref 33)

Japanese prepdp HNDPhA during WWII by treating dinitrochlorobenzene with aniline in the presence of Na carbonate to obtain 2,4-Dinitrodiphenylamine, followed by one-stage nitratior 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, pp 8-2 & 8-3)

Various methods of HNDPhA prep, 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 prep and uses is described in Refs 14 & 26

HNDPhA is a pseudo-acid which was investigated by Aleksandrov (Ref 8) and Hantzsche & Opolski (Ref 8a)

Acqg to Davis (Ref 26, p 184) 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. Urbanski (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 Rakstad (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:
Briscance by Sand Test – 0.5g crushed 49.5g sand, vs 48.0g for TNT (103% TNT) (Ref 22a);
Hercules gave 105%
Briscance by Compression of Copper Block – 114% TNT (Ref 28)
Briscance by Kast Formula – 111% TNT (Vol 2 of Encycl, p 279)
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
charge of 90/10-MF/KClO4 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 Hexomite, contg
TNT 60, HNDPhA 24 & AI 16% had the fol-
lowing properties: mp 81°, briscance (by
Sand Test) 116% TNT, detonation velocity
5900m/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 the dissistance 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 decompp and all samples detonated
after tests without difficulty when initiated
by detonators contg 2g of MF, or its equivalent
Refs for 2,4,6,2',4',6'-Hexanitrodiphenylamine:
Austen, Ber 7, 1279(1874) 3) R. Gehr,8
Ber 7, 1399(1874) 4) K.H. Mertens, Ber 11,
843(1878) 5) A. Hausemann, 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, 205–6(1913) & CA 7,
3228(1913) 12) M. Giua, SS 10, 111–12
(1915) & CA 9, 3362(1915) 13) Marshall I
(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 1478818 (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) Cifft &  
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., PBRep 1820 (1945)  
30) C.H. Brooks et al., PBRep 38154 (1945)  
(Mainf and uses of Hexyl in Germany)  
31) G.C. Tibbits et al., PBRep 50394 (1946)  
(Mainf and 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);  
BritJ Dermatol 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.  
Méard & M. Thomas, MP 31, 173 (1949) &  
CA 46, 11684 (1952) (Heat of combst &  
meth 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) EtatFrancais, 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, Pólvoros (1952),  
104–85 (Hexilo or Hexamina) 41) Belgrano  
(1952), 140 (Exil or Esanitrodifenilamina)  
42) A. Stettbacher, Explosivstoffe No 5/6,  
63–4 (1953) (Lab prepn of HNDPhA) 42a) A.  
Langhans, Explosivst 1/2, 7 (1954) (Thermal  
decomn of HNDPhA) 43) L. Méard,  
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) Fe-  
donroff et al., PATR 2510 (1958), p88 (Hexa)  
45) Gia, Tratt.6 (1) (1959), 325–26 (Esan-  
itrodifenilamina) 46) CondChemDiet  
(1961), 573–L 47) Sax (1963), p875  
48) Ubański 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, usefull 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 dem in expl mixes.  
Thomas (Ref 1) used the solubility method  
deg the qualitative & quantitative analyses  
of binary mixes of HNDPhA & TNT and of  
other mixes 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 ded. Other  
tests used are mp, colors obtd in acetone  
soln with NaOH & NH3 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. Kerres &  
& 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 dem in N,N-dimethyl-  
formamide) 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) Electroanalytical props)  
6) R.D. Tiwari & J.P. Sharma, Talanta 10,  
933–34 (1963) (English) & CA 59, 12185 (1963)
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 & Hopenstrick (Ref 12)

Ammonium Salt, known as Aurantia,
NH₄C₁₂H₄N₄O₁₂, mw 456.25; N 24.56%; red-brown crystals; mp explodes on heating; heat of combustion 3128 cal/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, p37) (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

Borium Salt, Ba(C₁₂H₄N₄O₁₂)₂; red crystals, mp – explodes on heating; sol in w & alc;

was prepd by action of Ba(OH)₂ on hexanitrodiphenylamine

Cobalt Ammonia Salt, [Co(NH₄)₆][C₁₂H₄N₄O₁₂]₃ + 2.5H₂O; scarlet-red amorphous powd, mp – dec on heating to 95°C (Ref 1, p 1423)

Lead Salt, Pb(C₁₂H₄N₄O₁₂)₂ + 8 PbO, dk-red powd, mp – defgr at 250°C; insol in hot w

Lead Salt, Basic, Pb(OH)₂C₁₂H₄N₄O₁₂; mw 662.43, N 14.80%; was prepd in mixed or double crystals 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, KC₁₂H₄N₄O₁₂, 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, AgC₁₂H₄N₄O₁₂; mw 546.09, N 17.94%; violet crystals (from acet) or red-yel crystals; mp – darkens ca 190°C, decomposes below 200°C leaving a brown mass; explodes weakly when dropped on a surface of molten metal in a bath preheated to 310°C. 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 amnt 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 crystals 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:
9) Clift & Fedoroff 2(1943), pp A7 & H4
20) F. Taylor Jr, USP 3418372 (1968) & CA 70, 69785k (1969) (K salt of HNDPhA)

**2,4,6,2',3', 4'-Hexonitrodimethylamine**,

\[(\text{O}_2\text{N})_3\text{C}_6\text{H}_2\text{NH} \cdot \text{C}_6\text{H}_2\text{(NO}_2)_3\] ; mw 439.22, N 22.33%, OB to CO₂ minus 52.8%; ye crystals, mp 273-74°C; bp - explodes at higher temps; insol in w, alc or eth; sl sol in hot AcOH.

It can be prep 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 comp described in Bel 12, 769 as 2,4,6,x,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°C/minute and 282° at 5°/min

**Impact Sensitivity by Kast Apparatus**: maxim

**mum 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', 3', 4', 6'-HNDPhA for use as an expl


**Diphenylamine-sulfone and Derivatives**

Diphenylamine-sulfone,

\[\text{C}_6\text{H}_4 \cdot \text{NH} \cdot \text{SO}_2 \cdot \text{C}_6\text{H}_4\]

mw 231.27, N 6.06%; ndls (from xylol), mp 257-59°; sol in alc; sl sol in hydrocarbon solvs; was prep by heating 10-methyl-phenothiazine-9,9-dioxide with hydroiodic acid (Ref 1)

2,4,6,8-Tetranitrodiphenylamine-sulfone or
2,4,6,8-Tetranitrophenothiazine-5,5-dioxide,

\[\text{C}_6\text{H}_4 \cdot \text{NH} \cdot \text{SO}_2 \cdot \text{C}_6\text{H}_2\text{(NO}_2)_2\]

mw 411.27, N 17.03%; crystals (from Nitrobenz), mp 344-47°; was prep by nitratiring the parent compd with HNO₃-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 subsrstitutes for Black Powder (Ref 3)


**Diphenylamine-sulfoxide and Derivatives**

Diphenylamine-sulfoxide (called Phenthiazin-9-oxyd or "Diphenylaminsulfoxyl" in Ger),

\[
\text{C}_6\text{H}_4\text{SO} \quad \begin{array}{c}
\text{NH} \\
\text{C}_6\text{H}_4
\end{array}
\]

mw 215.27, N 6.51%; ndis (from acet), mp 250° (dec); was prepd by warming phenothiazine,

\[
\text{C}_6\text{H}_4\text{SO} \quad \begin{array}{c}
\text{NH} \\
\text{C}_6\text{H}_4
\end{array}
\]

with aq alcoholic KOH & 30% hydrogen peroxide on a w bath (Ref 1) 2,4,5,7-Tetranitro diphenylamine-sulfoxide (called 2.4,5,7-Tetranitrophenothiazin-9-oxyd in Ger, and Tetranitro sulfoxyl diphenylamine (TNSD) in French),

\[
\text{O}_2\text{N}_2\text{C}_6\text{H}_2 \quad \begin{array}{c}
\text{NH} \\
\text{SO}
\end{array}
\]

mw 395.27, N 17.72%; yel crds (from Nitrobenz), mp 280° to 282°; heat of combustion at const vol 3339cal/g; heat of formn at const vol 294.5cal/g (Ref 6); was prepd by nitrating phenothiazine, 2,4- or 2,7-dinitrophenothiazine or 2,4,7-trinitrophenothiazine with fuming nitric acid & conc sulfuic 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

**Diphenylaminocarboxylic Acid or N-Phenylanthranilic Acid.** See Anilinobenzoic Acid in Vol 1, pA421-R

**Diphenylaminochloroarsine.** One of the Chemical Warfare Agents, describes 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**

D(or Bis){phenylamino-N-ethanol,}

\[
\text{CH}_2\text{CH}_2\text{OH}, \text{ or Diphenylamino-N-ethylol}
\]

mw 213.27, N 6.57%; liq, bp 183–86° at 5mm press, d 1.121 at 20°, nD 1.6210 at 20°; was prepd by heating DPHA with ethylene oxide in an autoclave to 220–40°, with a yield of 60–65% (Refs 2 & 3)

Dinitrodiphenylamino-N-ethanol,
\[ \text{O}_2\text{N.C}_6\text{H}_4.N.C_6\text{H}_4\text{NO}_2 \]
\[ \text{CH}_2\text{CH}_2\text{OH}; \text{mw} \ 303.27, \text{N} \ 13.86\%; \text{not found in Beil or in CA thru 1966} \]

Dinitrodiphenylamino-N-ethanol nitrate,
\[ \text{O}_2\text{N.C}_6\text{H}_4.N.C_6\text{H}_4\text{NO}_2 \]
\[ \text{CH}_2\text{CH}_2\text{ONO}_2 \ \text{mw} \ 348.27, \text{N} \ 16.09\%; \text{not found in the literature thru 1966} \]

Trinitrophenylamino-N-ethanol,
\[ \text{C}_4\text{H}_12\text{N}_4\text{O}_7 \], not found in Beil or in CA thru 1966

Tetranitrodiphenylamino-N-ethanol or Bis[Dinitrophenyl]aminooethanol;
\[ \text{(O}_2\text{N})_2\text{C}_6\text{H}_4.N.C_6\text{H}_4\text{(NO}_2)\text{)}_2 \]
\[ \text{CH}_2\text{CH}_2\text{OH}; \text{mw} \ 393.27, \text{N} \ 17.81\%; \text{lemon-yellow powd, mp} \ 222^\circ; \text{obtd as a by-product of the action of 2,4-dinitrochlorobenzene & ethanolamine; used to prep 2,4-dinitrophenylaminooethanol which was nitrat} \]
\[ \text{ed to Pentryl (2,4,6-Trinitrophenyl-nitraminoethyl Nitrate) (Ref 2)} \]

Refs: 1) Beil, not found 2) L.V. Clark, IEC 25, 1385 (1933) 3) Not found in later refs thru 1966

Hexanitrodiphenylamino-N-ethanol, Dipicrylaminooethylol or 2-[D(or Bis)-2,4,6-trinitrophenylaminooethyl]-ethanol, \[ \text{C}_4\text{H}_12\text{N}_4\text{O}_7 \], not found in Beil or in CA thru 1966

β (Hexanitrodiphenylamino) Ethyl Nitrate; 2-[D(or Bis)(2,4,6-trinitrophenyl) amino-ethanol] Nitrate or Dipicrylaminooethylol Nitrate,

\[ \text{NO}_2 \]
\[ \text{N} \quad \text{CH}_2\text{CH}_2\text{ONO}_2; \text{mw} \ 528.26, \text{N} \ 21.21\%; \text{crysts, mp} \ 184^\circ; \text{O bal to} \ \text{CO}_2 \text{ minus} 51.5\%, \text{d} \ 1.69, \text{d at} \ 3400 \text{lb/sq in} \ 1.27. \text{It is prep'd from Tetranitrodiphenylamino-ethanol, which is a by-product of the action between dinitrochlorobenzene & ethanolamine, by nitrat} \]
\[ \text{ing with mixed acid below} \ 30^\circ \]

Its expl props are found in Blatt (Ref 3), as dtd 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 1944(1944)

Diphenylamino-ethylol. See Diphenylamino-ethanol

Diphenyl-(amino-4-guanidovaleric Acid and Derivatives

Di(or Bis) phenyl-(amino-4-guanidovaleric Acid or Diphenylarginine,
\[ \text{(C}_6\text{H}_9)_2\text{N.C(NH).NH(CH}_2)_2\text{CH(NH}_2)_2\text{COOH, mw} \ 326.39, \text{N} \ 17.17\%; may be considered as the parent compd of its hexanitro deriv although not used to prep it} \]

Di(or Bis)-(2,4,6-Trinitrophenyl)-(amino-4-guanidovaleric Acid) or Dipicrylarginine,
\[ [(\text{O}_2\text{N})_2\text{C}_6\text{H}_4\text{NH}_2]_2\text{C}_6\text{H}_4\text{N}_2\text{O}_2; \text{mw} \ 596.39, \text{N} \ 23.49\%, \text{crystalline mass; diffe sol in alc, eth & water. It was prep'd in impure state by Hyana by shaking picryl chloride, CIC}_6\text{H}_4\text{(NO}_2)\text{)_2 (dissolved in toluene) with an} \]
\[ \text{equiv proportion of arginine, H}_2\text{N.C(NH).NH(CH}_2)_2\text{CH(NH}_2)_2\text{COOH.} \]
Its expl props were not reported (Refs 3 & 4) Reofs: 1) Beil 4, 420, (510) & [845](Arginine) 2) Beil, not found [Diphenyl-(amino-4-guanidovaleric acid) 3) Beil 12, 770 (Dipiryethylarginine) 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(aniilino)-propane in Vol 2 of Encycl p B132-R

Diphenylaminopropanol and Derivatives
Diphenylaminopropanol, C₆H₅-N-CH₂CH₂OH mw 227.29, N 6.16%; was prep'd in 1887 by Fauconnier (Ref 2) by interaction of aniline, C₆H₅-NH₂, and glycine monochlorohydrom, CI.C₆H₅-CHOHCH₂OH. We could not find any later info on this compd nor on its nitrate derivs in the literature thru 1966 Reofs: 1) Beil - not found 2) Fauconnier, CR 106, 695(1887)

2,3-Diphenyl-5-nitrosamino-tetrazolium Hydroxide, ON:N:C N
or
ON:N.C N

H₂N.C N

or

N-N(C₆H₅)(N(C₆H₅))OH

mw 255.27, N 27.44%; the chloride is obt'd by action of isomyl nitrite & HCl on alcoholic N,N'-diphenyl-C-aminoformalan, C₆H₅-N:C(NH)NC₆H₅ or C₆H₅-N:NC₆H₅. From the chloride & AgO in aq soin the free base is obt'd, yel, unstable & volatilizes (Refs 1 & 2)
The Chloride salt, C₁₃H₁₂N₅Cl, ndls (from alc); mp - dec 269°; sol in w & alc; insol in eth, benz, chl & petr eth Dichlorate salt, (C₁₃H₁₂N₅)₂Cr₂O₇, om-yel ndls (from w); mp - detonates at 189°

2,3-Diphenyl-5-nitrosamino-tetrazolium Hydroxide and Derivatives

2,3-Diphenyl-5-amino-tetrazolium Hydroxide, HN:N:C N

or

HN-N(C₆H₅)₂-N(C₆H₅)OH

or

N-N(C₆H₅)(N(C₆H₅))OH

mw 284.27, N 29.57%; om-yel cubes (from acet or pyridine), mp 177°; v sl sol in acet, eth, benz, chl & pyridine; was prep'd by reaction of Na nitrite on the above chloride salt in dill HCl at 20°. Heating the compd with 70% alc for 3-4 hrs gives an isometric deriv, C₁₃H₁₀N₅O, om-yel ndls (from alc), mp 154-55°

Its Chloroaurate salt, 2(C₁₃H₁₀N₅O)AuCl₃, om-yel crystals, mp - sinters at 103° & melts at 119-23°; v sl sol in water; insol in chl, eth & benz

Hydrochloride salt, C₁₃H₁₀N₅HCl, flakes, mp 168°; sol in alc & dill HCl; insol in benz, petr eth & acet and Acetyl Chloridate salt, 2(C₁₃H₁₀N₅O)Ac, yel ndls, (from alc), mp - dec 157-61° deflagrating mildly; insol in chl & eth; sl sol in hot alc; sol in dill nitric acid (Refs 1 & 2) Reofs: 1) Beil 26, [247] 2) E. Bamberger et al, Ann 446, 302(1926) & CA 20, 1224(1926)

Diphenylarginine. See Diphenyl-(amino-4-guanidovaleriacid) in this Vol

Di(phenylazo)-amine [called 1.5-Diphenylpentazien-(1.4) or Bisdiazobenzamid in Ger] C₆H₅.N:NH.N:N.C₆H₅; mw 225.24, N 31.09%;
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-pentamethylen e-1,4-diamine

Di(phenylazo)-dihydroxy-naphthalene. See Bis(phenylazo)-dihydroxy-naphthalene in Vol 2, p B152-R

Di(phenylazo)-glycine or Di(benzenediaz o)-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,

\[
\text{N} \equiv \text{N(C}_6\text{H}_5)\text{-C-N-C} \equiv \text{N(N(C}_6\text{H}_5)\text{-N)} \equiv \text{N} \\
\equiv \text{N} \quad \equiv \text{N} \quad \equiv \text{N} \\
\]

mw 318.30, N 44.01%, OB to CO\text{2} minus 165.9%; yel crystls, mp 228°; gives a blood-red soln in concd sulfuric acid; reduction gives a hydrazo deriv, crystls + 2H\text{2O}, mp 190°(dec); was prep'd by passing Cl\text{2} thru 1-phenyl-5-aminotetrazole in boiling water (Ref 2)

Refs: 1) Beil, not found 2) R. Stolle et al, JPraktChem 134, 282(1932) & CA 26, 5565(1932)

2,2'-Diphenyl-5,5'-azotetrazole or 5,5'-Azo-2,2'-diphenyltetrazole,


**Dichromate salt,** \(2(C_2\text{H}_4\text{N}_3\text{O})_2\text{H}_2\text{Cr}_2\text{O}_7\text{ or } (C_2\text{H}_4\text{N}_3\text{O})_2\text{Cr}_2\text{O}_7\text{, citron-yellow powd, mp } 170.5^\circ\text{C}(\text{dec}), \text{turns green in sunlight; decomp in hot water} \)

**Permanganate salt,** \(C_2\text{H}_4\text{N}_3\text{O}^+\text{HMnO}_4^- \text{ or } C_2\text{H}_4\text{N}_3\text{O}^+\text{MnO}_4^-\), violet crys, mp - explodes on heating to 144° or by friction; sl decomposes in hot water

Refs: 1) Beil 26, [249] 2) E. Bamberger et al, Ann 446, 305 (1926) & CA 20, 1224 (1926)

---

**2,3-Diphenylbenzopyrylum Perchlorate.** See under Benzopyrylum Compounds in Vol 2, p B79-R

**Di(p-phenylbenzoyl)-furoxan,**

\[ \text{p-C}_6\text{H}_5\text{C}_6\text{H}_4\text{CO}^-\text{OC}_6\text{H}_4\text{C}_6\text{H}_5^- \]

mw 464.44, N 6.28%; wh crys (from ether, then from benz), mp 185° with decomp to a bm liq; prep in 60% yield when nitric acid in water was added to a soln of p-phenylacetophenone in glacial 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 crys 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,**

\( \text{C}_6\text{H}_4\text{C}_6\text{H}_4\text{CO}_2\text{CO}_2\text{C}_6\text{H}_4\text{C}_6\text{H}_4; \text{mw } 394.40, \text{O } 16.23%; \text{crysts (from toluene), mp } 171^\circ(\text{with very violent decomp), can be prep by adding (while shaking) to an acetonic soln of p-phenylbenzoyl chloride a slight excess of alkaline 10% hydrogen peroxide} \)

Refs: 1) Beil, not found 2) H. Wieland & G. Rasuwajew, Ann 480, 164-65 (1930) & CA 24, 4013 (1930)

---

**Diaryl-bis(4,4'-diozoniumhydroxide).** See Diphenyl-bis(4,4'-diozoniumhydroxide) in Vol 2, p B125-L

**1,1'-Diphenyl-5,5-bistetrazole,**

\[ \text{H}_5\text{C}_6\text{H}_5\text{N}=-\text{C}=-\text{C}=-\text{N.C}_6\text{H}_5 \]

\[ \text{N}:-\text{N} \quad \text{N}:-\text{N} \]

mw 290.28, N 38.60%; ndis (from glacial acetic acid), mp 212°; sol in benz & glacial acetic acid; sl sol in hot alc; prep'd by action of NaN₃ in boiling alc on oxalic acid-bis(phenylimidechloride)

(Refs 1 & 2)

Refs: 1) Beil 26, [363] 2) R. Stollé, Ber 55, 1296 (1922)

---

**Diphenylbromosamine, (C₆H₅)₃AsBr; wh crys, 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**


---

**Diphenylcarbamide (or Diphenylcarbamyl) Complexes,** proposed by Lévy (See Ref) as suitable stabilizers and gelatinizers in smokeless propellants, were prep'd by interaction of diphenylcarbamylchloride (DPhCCl), (C₆H₅)₂N.CO.Cl, (described in Beil 12, 428) with organic bases such as allylamine, C₆H₅NH₂. 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 prep'd and tested by Lévy:

**Bis-(diphenylcarbamyl) -ethylenediamine,**

(C₆H₅)₂N.CO.NH.CH₂.CH₂.NH.CO.N(C₆H₅)₂; mw 450.52, N 12.44%; wh crys, mp 184°; sol in alc; was prep'd by refluxing for 6 hrs, with agitation, DPhCCl with ethylenediamine in alc contg Na carbonate

**Diphenylcarbamylallylamine,**

(C₆H₅)₂N.CO.NH(CH₂.CH:CH₂); mw 252.30,
N 11.10%; wh crysts, mp 82°, sol in ethanol, methanol & acet; was prep'd by refluxing for 6 hrs, with agitation, DPhHCl with allylamine in alc contg Na carbonate

**Diphenylcarbamyl diethanolamine,**
\[(C_6H_5)_2N\cdot CO\cdot N\cdot (CH_2\cdot CH_2\cdot OH)_2;\] mw 300.35, N 9.33%; wh crysts; mp 124.5–125°; sol in alc; was prep'd by refluxing for 6 hrs, with agitation, DPhHCl with diethanolamine in alc contg Na carbonate

**Diphenylcarbamyl monoethanolamine,**
\[(C_6H_5)_2N\cdot CO\cdot NH\cdot (CH_2\cdot CH_2\cdot OH);\] mw 256.29, N 10.93%; wh crysts, mp 117.5°; sol in ethanol, methanol & acet; was prep'd by refluxing for 6 hrs, with agitation, DPhHCl with monoethanolamine in alc contg Na carbonate

**Diphenylcarbamyl morpholine,**
\[(C_6H_5)_2N\cdot CO\cdot N\cdot \left(\begin{array}{c} CH_2 \\ CH_2 \end{array}\right) \] mw 282.33, N 9.92%; wh crysts, mp 108.5°; sol in ethanol, methanol & acet; was prep'd by refluxing for 3 hrs, with agitation, DPhHCl with morpholine in alc contg Na carbonate

**Diphenylcarbamyl-\(\beta\)-naphthylamine,**
\[(C_6H_5)_2N\cdot CO\cdot NH\cdot (C_6H_5\cdot CH_2);\] mw 338.39, N 8.28%; wh crysts with sl rose tinge; mp 185–185.5°; sl sol in ethanol & methanol; was prep'd by refluxing for 6 hrs, with agitation, DPhHCl & \(\alpha\)-naphthylamine in alc contg Na carbonate

**Diphenylcarbamyl-\(\beta\)-naphthylamine,**
\[(C_6H_5)_2N\cdot CO\cdot NH\cdot (C_6H_5\cdot CH_2);\] mw 338.39, N 8.28%; wh crysts, mp 182°; sl sol in cold alc; was prep'd by refluxing for 6 hrs, with agitation, DPhHCl with \(\beta\)-naphthylamine in alc contg Na carbonate

**Diphenylcarbamyl piperidine,**
\[(C_6H_5)_2N\cdot CO\cdot N\cdot CH_2\cdot CH_2\cdot CH_2\cdot CH_2\cdot CH_2;\] mw 280.36, N 9.99%; wh crysts, mp 123.5°; sol in ethanol, methanol & acet; was prep'd by refluxing for 3 hrs, with agitation, DPhHCl with piperidine (cyclopentamethyleneamine) in alc, contg Na carbonate

**Ref:** R. Lévy, MP 32, 309–12 (1950)

**Diphenylcarbamyl Azide.** Same as Diphenylcarbamyl Azide

**Diphenylcarbamyl Azide (Diphenylcarbamidsäureazid in Ger),** \[(C_6H_5)_2N\cdot CO\cdot N\cdot S;\] mw 238.24, N 23.52%; ndls (from alc); mp 85° (decomp); v sl sol in w; v sol in alc, eth, chl & benz. It can be prep'd by interaction of diphenylcarbamyl chloride, \[(C_6H_5)_2N\cdot CO\cdot Cl\] with Na azide in boiling ethanol

Explodes mildly when heated in a flame


**Diphenylcarbazide or 1,5-Diphenylcarboxyhydrizide,**
\[(C_6H_5)_2N\cdot CO\cdot H_2;\] mw 242.27, N 23.13%; wh crysts, decomposing in light; mp 173–75°; insol in w & eth; sol in hot alc; can be prep'd by interaction of phenylhydrizine, \[C_6H_5\cdot NH\cdot C_6H_5\] and urea, \[H_2\cdot N\cdot CO\cdot NH_2\]

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,** \[C_6H_5\cdot N\cdot CO\cdot NH\cdot NH\cdot C_6H_5;\] mw 240.26, N 23.32%; om-red ndls, mp ca 127° (decomp); insol in w; sol in alc, eth, benz & chl; can be prep'd by action of phosgene on an ethereal soln of 1,5-diphenylhydrizine

**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 (C3L 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 distilled 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 solution in a small beaker add 0.1ml of alcoholic sample solution and observe if any violet color is produced. If no color is produced the sample must be rejected.


2,3-Diphenylcarbodiazone (called C-Hydroxy-diphenylterrazolium 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 Diphenylcarboxylot or Anhydride of 2,3-Dihexyl-5-oxyterazolium-hydroxide in Ger) D.C——N

N.N(C6H5)2.N.C6H5+mm 238.24, N 23.52%; bitter tasting platelets (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 prepared by oxidizing 3,5-diphenylcarbodiazole with isoamyl nitrite in glacial ac + acet on a water bath, or with KMnO4 in acet (Refs 1 & 2)

It forms salts, some of which are exp: Chloroplatinate, 2C12H10N4O+H2PtCl6,

flesh-colored powd, mp 196°(dec)

Dibromide, C13H10N2Br2, yel crysrs (from alc + eth), mp — darkens at 60° & decomp; sol in alc; insol in eth & w

Perchlorate, C13H10N2O+HClO4, col crysrs, mp — explodes at 194°

Picrate, 2C12H10N2O+C6H5N3O7, golden-yel platelets (from alc), mp 169°(dec); sol in hot w (Refs 1 & 2)


Diphenylcarbodrazide. See Diphenylcarbazid, p D1446-R in this Vol

Diphenylcarbonate or Diphenylester of Carbonic Acid, (C6H5O)2CO; mw 214.21; wh ndls (from alc); sp gr 1.272 at 14°; mp 78-80°; bp 302-306°; insol in w; sol in acet, benz, eth, glacial 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 AlCl3 at 170-90° (Reps 1 & 2)

Used as a plasticizer and solvent for NC

(Ref 3)


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 hexa-nitro-derivative were given but preps 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 cmpds here:

Di(N-phenylcarboxamide)-acetylene,

C8H8.NH.CO.C:CO.NH.C6H5; mw 264.27, N 10.60%; pink-wh crysrs (from MeOH+H2O), 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_2NH.CC.CO.CC.CO.NH.C_6H_2(NO_2)_3, mw 534.27, N 20.97%; wh crts, 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 in-completely 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 CHE's, 2nd Rept (1951), 153–54 4) Not found in later refs thru 1966


Diphenylcyclopentane and Derivatives
1,2-Diphenylcyclopentane, (C_6H_5)CH.CH.CH.CH.CH(CH_2)_3.C_6H_5; mw 222.31. It exists as cis-, crts (from petr eth), mp 47° and trans-, plts (from MeOH) or ndls (from acetic acid; can be prepd from (S)cis- or (Z)trans 1,2-diphenylcyclopentane-(3) or -(4) by heating with Zn amalgam & aq HCl in toluene (Refs 1 & 4) x.x.x.x-Tetranitro-1,2-diphenylcyclopentane, C_17H_10(NO_2)_4; mw 402.31, N 13.93%; plts (from glacial 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, 560, (316) & l20251
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

Diphenyldianthronilate and Derivatives
Diphenyldianthronilate,
\[ \text{CO}.N\left(\text{C}_6\text{H}_5\right) \]
\[ \text{C}_6\text{H}_4 \]
\[ \text{N}\left(\text{C}_6\text{H}_5\text{CO}\right) \]
mw 390.42, N 7.17%; may be considered as the parent compd of its nitrated derivs, although not used to prepd them: 2,4,2',4'-Tetranitrodiphenyldianthronilate or N,N'-Di(2,4-dinitrophenyl)-dianthronilate,

 mw 570.42, N 14.73%; ndls (from acet + alc), mp = dec; readily sol in acet, chl, 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'-Hexanitrodiphenyldianthronilate or N,N'-Dipicryl-dianthronilate,
\[ \text{CO}.N\left(\text{C}_6\text{H}_5\text{NO}_2\right)_2 \]
\[ \text{C}_6\text{H}_4 \]
\[ \text{N}\left(\text{C}_6\text{H}_5\text{NO}_2\right)_2 \]

 mw 660.42, N 16.97%; citron-yel ndls (from phenol + acet ac), mp > 200°(dec); sol in hot phenol & boiling ethylene bromide; 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-diazooaminobenzene or 4-Diazo-oaminobiphenyl. See I, III-Bis(p-biphenyl)-triazine in Vol 2, p B136-L
Diphenyldiazomethane. See Diphenyldiazomethane in Vol 2, p B125-R

Diphenyldiazonium Perchlorates
Davis & Huntress (Ref) patented the following method of prep of diazonium perchlorates of general formula R.N₂ClO₄, in which R is an organic radical, such as diphenyl-, nitrodiphenyl-, etc

*Example:* Diazotize 2,2'-dinitrobenzidine, H₂N.C₆H₅(NO₂)₂-C₆H₅(NO₂)₂.NH₂, 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)

Diphenyldibutyleurea. See Centralite, Butyl, under CENTRALITES in Vol 2, p B140-L

Diphenyldicarboxylic Acid or Diphenic Acid.
See Diphenyldicarboxylic Acid in Vol 2, p B125-R

Diphenyldiethyleurea. See Centralite 1 in Vol 2, p B127-L. It has been used as a stabilizer in some US prolpts such as Units No 548 & 549 described in cond Proplnt Manual SPIA M/2 (1962)

9,10-Diphenyl-9,10-dihydropyrazine and Derivatives

9,10-Diphenyl-9,10-dihydropyrazine

\[ \text{C}_6\text{H}_4 \stackrel{N(C_6H_5)}{\longrightarrow} \text{C}_6\text{H}_4 \]

mw 334.40, N 8.36%; ndls (from benz + alc), mp 172-75°; sol in chlf; sl sol in cold benz; v sl sol in eth, alc & acer ac; was obtd & separated from other products when tetraphenylhydrazine was boiled with toluene for 1/2 hr in an atm of CO₂ (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-dihydropyrazine,

\[ \text{C}_6\text{H}_4 \stackrel{N(C_6H_4NO_2)}{\longrightarrow} \text{C}_6\text{H}_4 \]

mw 424.40, N 13.20%; reddish-brn crysts, mp - sinters at 154° & melts ca 185°; sol in chlf & in sulfuric acid giving a taint gm color changing to violet on addn of CrO₃ in alc KOH with faint red color deepening on heating; sl sol in benz & acer 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)


Tetranitro, C₂₄H₁₄N₈O₈, and Hexanitro, C₂₄H₁₉N₈O₁₂, derivs of 9,10-Diphenyl-9,10-dihydropyrazine were not found in the literature thru 1966

Diphenyldiimid. 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

Diphenyldimethyleurea. See Centralite 2 in Vol 2, p B137-L and ADL Rept, Pt 1 (1947), p 111, where it is stated that powert & briskness of Hexanitrodimethylurea are lower than those values of Picric Acid

Diphenyleineimine. Same as Carbazole described in Vol 2, pp C45 to C50
4,4'-Diphenyl-diphenyllyl-amine. See Bis-(p-biphenyl)-amine in Vol 2, p B136-R

Diphenyl-dipicryl-ethylene-dihydrazine.
See a,β-Bis(a-phenyl-β-picryl-hydrazine)-ethane in Vol 2, p B154-R

Diphenyl-diquinone-3,4,3',4'-bis-dioxide-
(4,4'). One of the names for compd listed
as 4,4'-Bis(4-diazao)-3,3'-dioxy-biphenyl in
Vol 2, p B139-L

Diphenyldisulfide and Derivatives
Diphenyldisulfide or Bis(phenyl)-disulfide,
C₆H₅-S₂-C₆H₅; mw 218.34; ndls (from alc),
mp 61-62°, bp 310°; insol in w; s1 sol in
eth, benz & CS₂; sol in alc & in hot concd
sulfuric acid with a red-violet color; can be
prepd by heating thiophenol in ammonia-
ical qg soln & passing thru of air, and by
other methods (Ref)
Ref: Beil 6, 323, (148), [294] & |1027|

Mononitrodiphenyldisulfide,
C₆H₅-S₂-C₆H₅-NO₂; 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|

Nitrodiphenyldisulfide,
O₂N-C₆H₅-S₂-C₆H₅-NO₂; mw 308.34, N 9.09%.
2,2'-Nitrodiphenyldisulfide, yel ndls (from
benz or glacial acet ac), mp 197-98° (Ref 1)
3,3'-Nitrodiphenyldisulfide, yel ndls (from
alc), mp 81-84° (Ref 2)
4,4'-Nitrodiphenyldisulfide, crysts, mp
181-83° (Ref 3)
2,4'-Nitrodiphenyldisulfide, crysts (from
eth + chl), mp 159.2-159.6° (Ref 4)
2,4-Dinitrodiphenyldisulfide,
(O₂N)₂C₆H₅-S₂-C₆H₅, yel ndls (from alc),
mp 86-87° (Ref 5)

Other props & methods of prepn are found
in Refs
(160), [312] & |1077|  4) Beil 6, |1077|
5) Beil 6, |1101|

Trinitrodiphenyldisulfide,
(O₂N)₃C₆H₅-S₂-C₆H₅-NO₂; mw 353.34, N 11.89%
2,4,2'-Trinitrodiphenyldisulfide, crysts, mp
175-76°; was prepd by reaction of 2,4-dinitro-
thiophenol & 2-nitrobenzenesulfenyl chloride
in eth (Refs 1 & 2)
2,4,4'-Trinitrodiphenyldisulfide, crysts, mp
178°; was prepd from 2,4-dinitrothiophenol &
4-nitrobenzensulfenyl chloride (Ref 3)
Refs: 1) Beil 6, [1101]  2) V.O. Lukashevich
& M.M. Sergeeva, Dokl Akad Nauk 67, 1041
(1949) & CA 44, 1921 (1950); ZhurObshchKhim
19, 1493, 1504(1949) & CA 44, 2452(1950)
3) A.J. Parker & N. Kharatsch, JACS 82, 3075
(1960) & CA 55, 3497 (1961)

Tetranitrodiphenyldisulfide,
(O₂N)₄C₆H₅-S₂-C₆H₅-NO₂; 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 re-
acting 4-chloro-1,3-dinitrobenez & Na₂S₂
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 chl & acet ac;
s1 sol in eth, benzene & alc; was prepd by
heating 3,5-dinitrobenzene sulfonyl chloride
with Pb₃ at 100-105° 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, Dokl Akad Nauk 67, 1493, 1503(1945)

Pentanitrodiphenyldisulfide,
(O₂N)₅C₆H₅-S₂-C₆H₅-NO₂, not found in the
literature thru 1966

Hexanitrodiphenyldisulfide; Di(2,4,6-Trinitro-
phenyl)-disulfide or Dipicryl-disulfide,
1,3-Diphenyl-4-disulfonmethylene-tetrazone
(1) or Benzenediazoxophenyl-hydrazonemethane-
-disulfonic acid,
C₆H₄N₂·N(C₆H₅)₃·N:CS₂H₂. It is known in the form of its Dipotassium Salt,
K₂C₁₅H₁₀N₂O₄S₂, mw 460.56, N 12.17%, prepd by von Pechmann in 1896. It consists of orange-red crystals, dehydrating at 180° when confined; sol in w and in concd sulfuric acid. It can be prepd by pouring a well-cooled soln of benzenediazoxonium chloride, C₆H₅N₂.Cl, in aqueous KOH into an aq soln of the tripotassium salt of sulfonamidemethylenedisulfonic acid, (HO₃S)₂C·NH·SO₃H.
Refs: 1) Beil 16, 747 2) H. von Pechmann, Ber 29, 2165 (1896)

1,4-Diphenyl-3,5-endoanilino-1,2,4-triazole.
See 1,4-Diphenyl-3,5-phenylimino-1,2,4-triazole in this Vol

Diphenylene and Derivatives

o-Diphenylene or Biphenylene (called Biphenyle, or Cyclobutadienbenzol in Ger),

mw 152.18; lt-yel crysts (from alc), mp 110-120°; bp 105° at 4mm press; was prepd by distilling 2,2'-dibromobiphenyl or dibenzo-
iodonium iodide with cuprous iodide (Ref 2), and also by the action of cupric chloride on
the Grignard reagent prep'd from 2,2'-dibromo-
diphenyl (Ref 3). It forms a Picrate salt,
C₁₂H₁₀·C₆H₅·NO₃·O₇, red ndls (from alc), mp 122° (Ref 1)
Refs: 1) Beil 5, (299), [530] & [1935]
2) W.C. Lothrop, JACS 63, 1187, 1190 (1941)
3) W.S. Rapson et al, JChemSoc 1943, 326

Dinitro-o-diphenylene, C₁₂H₆(NO₂)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 nitratn of diphenylene, and by
heating either 4,5'-dinitrodi-phenylene-2,2'-
iodonium iodide or 2,2'-di-iodo-4,5'-dinitro-
diphenyl with cuprous oxide at 350° (Ref 3)
X,X-Dinitro-o-diphenylene, ndls (from alc),
mp 204°; insol in eth, benz & CS₂; sol in alc, glacial ac & ac; 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,
C₁₅H₄(NO₂)₄; mw 332.18, N 16.87%; yel ndls (from alc), mp 223°; sol in alc & glacial ac;
insol in other org solvs; was obtd by nit-}
trating 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, C₁₅H₄(NO₂)₆, not
found in the literature thru 1966

Diph(phenylene)-azo-trinitromethane. See
p,p'-Bi(phenylene-azo-trinitromethane) in
Vol 2, p B126-L

Diphenylene oxide. See Dibenzo[8]-furan 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: min crystals, 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 benzyl chloride 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 comds, are described in Vol 2, pp B110-R & B111-L 1,1-(or unsym)Diphenylethene, (C_6H_5)_2CH.CH_5; col oil, sp gr 1.004 at 20°, ft p -21.5°, bp 272°; insol in w; sol in alc, eth, chlf & CCl_4. It can be prepd by the action of acetaldehyde 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)_2.CH_2NO_2; mw 272.25, N 10.29%; ndis (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 & a,a-diphenylethylene in the cold (Refs 3 & 4)

1,1-Diphenyl-2,4-dinitrophenyl-ethane, (p-O_2N.C_6H_4)CH.CH_5; 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_5(NO_2)_2]_2.CH.CH_5; 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_{9}N_5O_{10}, not found in Beil or in CA thru 1966

Hexanitrodiphenylethane, C_{14}H_{9}N_5O_{12}, not found in Beil or in CA thru 1966


Diphenylether, Phenylether, Diphenyleuxide or Bis(phenyl)-ether, C_6H_5.O.C_6H_4; mw 170.20, col crystals, 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


Following nitratd 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_12H_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], [821], [860]

Trinitrodiphenylethers, C_12H_7N_3O_7; mw 395.20, 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], 1970.

They are, judging by their N content, weak expls.

Tetranitrodiphenylethers, C_12H_7N_4O_8; 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$_2$N)$_2$C$_6$H$_2$.O.C$_6$H$_3$(NO$_2$)$_2$; yel ndls (from alc), mp 194–95°C (186°C, probably impure); flash 
point above 360°C; diff sol in alc or eth; easily 
sol in hot AcOH, ethyl acet, benz or chl. 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, Wilminton, 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] & 1863] 
2) A. Mailhe & M. Murat, CR 154, 715 (1912); 
BullFr [4], 11, 444 (1912) & JCS 102, I, 346 
(1912) 3) A.H. Blatt, OSRD 1944(1944)

2,4,6,2'-Tetranitrodiphenylether, 
(O$_2$N)$_3$C$_6$H$_2$.O.C$_6$H$_3$(NO$_2$)$_2$; ndls (from alc), 
mp 172–73°C. It can be prep by heating alco-
holic picrylchloride with K-nitrophenolate 
Refs: 1) Beil 6, 291 & [970] 2) (?) Will-
gerodt & (?) Hüetlin, Ber 17, 1766 (1884). 
No later refs found in the literature

2,4,6,3'-Tetranitrodiphenylether, 
(O$_2$N)$_3$C$_6$H$_2$.O.C$_6$H$_3$(NO$_2$)$_2$; pale-yel lifts 
(from ethyl acetate), mp 169.5–171°C. It 
can be prep 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$_2$N)$_3$C$_6$H$_2$.O.C$_6$H$_3$(NO$_2$)$_2$; lifts (from alc), 
mp 153°C. It can be prep by warming an al-
coholic 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$_8$N$_6$O$_{13}$, 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$_2$N)$_3$C$_6$H$_2$.O.C$_6$H$_3$(NO$_2$)$_2$; crys (from eth 
acet); can be prep by warming on a water 
bath for 1 hr 4,6-dinitro-3-hydroxylaminodi-
phenylether 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$_2$N)$_3$C$_6$H$_2$.O.C$_6$H$_3$(NO$_2$)$_2$; 
crys (from acet), mp 210–210.5°C (Ref 1) & 
208°C (Ref 3); decomp by w; its solubility in 
various solvents was detd by Desverges (Ref 
2). It can be prep by nitrating with mixed 
nitric-sulfuric acid of 2,4,6-Trinitrodiphenyle-
ther or of 2,4,6,2'- or of 2,4,6,4'-Tetranitro-
diphenylether (Refs 1 & 3). It is a much more 
sensitive to impact than TNT and less power-
ful (93% of TNT) 
Refs: 1) Beil 6, 291, [282] & [970] 2) L. 
Desverges, Moniteur Scientifique [5], 16, 
204 (1924) & ChemZtr 1926, II, 3043 3) A.H. 
Blatt, OSRD Rept 1944 (1944)

2,4,6,3',4'-Pentanitrodiphenylether or 3',4'- 
Dinitrophenylether-picrate, 
(O$_2$N)$_3$C$_6$H$_2$.O.C$_6$H$_3$(NO$_2$)$_2$; lifts (from eth 
acet), mp 200–204°C; insol in w; s1 soln in alc 
or eth; sol in benz, toluene & Nitrobenzene; 
can be prep by nitrating of 3,4-dinitrophenyle-
ther with mixed nitric-sulfuric acid at 110°C 
(Refs 1, 2 & 3). It is a powerful expl patented 
in Germany by Westfälisch-Anhaltische AG 
during WW! for use as an HE (Refs 3 & 4) 
Refs: 1) Beil 6, (141) & [282] 2) C. van 
Duijn & B. van Leenan, Rec 39, 155 (1920) 
3) Clift & Fedoroff 2(1943), p 5 4) A.H. 
Blatt, OSRD Rep 1944 (1944)

Hexanitrodiphenylethers, C$_{12}$H$_8$N$_6$O$_{19}$, 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',4'-Hexanitrodiphenylether or 
Dipicyler ether or Dipicyloyde 
(O$_2$N)$_3$C$_6$H$_2$.O.C$_6$H$_3$(NO$_2$)$_2$; col ndls or lifts; 
mp 252°C (decomp) (Ref 4); insol in w, s1 soln 
in alc and in eth; very sol in nitrobenzene; 
can be prep by two-step nitrating of diphenyle-
ther using nitric acid-oleum, first to tetra 
and then to hexa, or by one-step nitrating of 
nitroderivatives unsubstituted in 3 and 3' 
positions, using mixed nitric acid-oleum at 
120°C (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, 1970| 2) A. Mailhe & M. Murat, CR 154, 715 (1912) & JCS 102, 1, 346 (1912) 3) Cline & 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-diphenyléther in Beil), (O_2N)_3C_6H_4O.C_6H_3(NO_2)_2; crystals, mp 269°, 278° (corr); insol in w; can be prep'd by interaction of m-nitrophenol and 2,4-dinitrochlorobenzene, followed by nitration; can also be prep'd by nitration with mixed acid at 100° the hexa-nitroderivatives of diphenyl ether, such as 2,4,6,3'-Tetranitrodiphenyl ether (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 Duijn & B. van Lennep, Rec 39, 145 (1920) 3) Steckbacher (1933), 294 4) A.H. Blatt, OSRD Rept 2014 (1944)

**Diphenylethylen and Derivatives**

1,2-Diphenylethylene, Biphenyl or Stilbene, C_6H_5.CH.CH.C_6H_5, mw 180.24

Its *trans* form, H.C.C_6H_5

H_2C.C_6H_4.CH , known as Toluylene, consists of colorless (or sl yel) crystals, 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 prep'd by passing toluene over hot oxide. It is used in manuf of dyes (Refs 1 & 3)

Its *cis* form, H.C.C_6H_3

H.C.C_6H_5 known as Iso-stilbene, is a yel oil, fr p 1° and bp 145° at 13mm, d 1.0143 at 20°; nD 1.6234 at 13°; can be obtd by exposing the trans-form to UV light, or from tololene (diphenylacetylene) thru electrolytic reduction at the Ni cathode in alcoholic sulfuric acid, and by other methods (Refs 2 & 3)

Diphenylethylen forms many addn comps & nitro derivs, some of which are expl


3) CondChemDict (1961), 1076-R & 1077-L

(Stilbene)

Mononitrodiphenylethylen 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 & o-Nitro-stilbene, C_6H_5.CH(CH(NO_2)).C_6H_5 (Ref)

**Refr:** Beil 5, 636, (305), [540] & [1966–68]

**Dinitrodiphenylethylen or Dinitrostilbene,** C_14H_10.N_2.O_4, mw 270.24, N 10.37%. The following isomers are known:

2,4-Dinitrostilbene, C_6H_5.CH.CH.C_6H_5(NO_2)_2, exists in high- & low-melting forms (Ref 1)

2,5-Dinitrostilbene, bm ndls (from alc), mp 149.5° (Ref 2)

2,6-Dinitrostilbene, om-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), l-yl ndls (from chlF), mp 191–96° & 199°, bp — explodes on heating to 420° in a tube; sol in alc, eth & pet eth; sol in hot benz; was obtd, in addn to a low-melting form by treating a soln of o-nitrobenzyl chloride in 98% alc with a soln of potash in alc, and separating the isomers by crysrgg the product from epichlorohydrin in which the higher-melting isomer is sol in the cold solv (Ref 3)

2,3'-Dinitrostilbene, om-yel ndls (from alc), mp 150°, bp — sublimes at 160° under 0.001mm pressure (Ref 4)
2,4'-Dinitrostilbene, trans, yel plts (from alc), mp 143° or yel ndls (from glc acet ac), mp 140°; bp — sublimes at 130—40° under 0.001mm press; cis, yel-bm ndls (from alc or glc acet ac), mp 140° (Ref 4)

3,3'-Dinitrostilbene, trans, yel ndls (from glc acet ac), mp 240—42°, bp — sublimes at 175° under 0.001mm press (Ref 4)

3,4'-Dinitrostilbene, trans, yel ndls (from glc acet ac, Nitrobenz or pyridine), mp 220—22°, bp — sublimes at 170° under 0.001mm press; cis, yel ndls (from glc acet ac), mp 155° (Ref 5)

4,4'-Dinitrostilbene, trans, yel ndls (from glc acet ac or Nitrobenz), mp 292—95° & 303—04°; cis, yel crystals or tablets (from glc acet ac or acetone), mp 183—86° & 234—35° (Ref 6)

2,4'-Dinitrostilbene.

C₆H₄.C(NO₂)₂:CH.C₆H₄.NO₂, lt-yel ndls (from alc), mp 106° (Ref 7)

3,4'-Dinitrostilbene, cis, crystals (from alc), mp 112° (Ref 7)

4,a'-Dinitrostilbene, cis, yel ndls (from ethyl acet & petr eth), mp 155—157.5° (Ref 8)

α,a'-Dinitrostilbene.

C₆H₄.C(NO₂)₂:C(NO₂)₂.C₆H₄, 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,a'-Dinitrostilbene.

C₆H₅.CH.C(NO₂)₂.C₆H₄.NO₂, crystals (from benz & petr eth), mp 179—80° (Ref 7)

Other props & methods of prep are found in the Refs

Trinitrodiphenylethylen or Trinitrostilbene, C₁₄H₈N₄O₆; 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,

C₆H₅.CH.CH.C₆H₄(NO₂)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 CCl₄, glc 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)-ethylen,

O₂N.C₆H₄.CH.CH.C₆H₄(NO₂)₂, trans, gm-yel crystals (from glc 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 glc 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 glc acet ac; was prepd by heating 2,4-dinitrotoluene & 4-nitrobenzaldehyde in the presence of piperidine (Ref 4)

3,4',a'-Trinitrostilbene or 1-(4-Nitrophenyl)-2-nitro-2-(3-Nitrophenyl)-ethylen,

O₂N.C₆H₄.C.C(NO).C₆H₄.NO₂, brownish crystals (from toluene), mp 236—37°; was prepd by heating 3-nitro-1-nitromethylenzene & 4-nitrobenzaldehyde in the presence of alcoholic methylamine soln (Ref 5)

4,4',a'-Trinitrostilbene or 1-Nitro-1,2-di(4-nitrophenyl)-ethylen, gm-yel crystals (from ethylacetate), mp 210,5—211°; was obtd with other products by nitrating 4,a'-Dinitrostilbene (mp 155°) with nitric acid (d 1.49) at minus 20° to 15° (Ref 5)

Other props & methods of prep are given in the Refs

Tetranitrodiphenylethylen or Tetranitrostilbene, C₁₄H₈N₄O₆; 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,

\( \text{O}_2\text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{CH} \cdot \text{CH} \cdot \text{C}_6\text{H}_4(\text{NO}_3)_2 \) (Ref 1 & 7), was prepd from reaction of 2,4,6-Trinitrotoluene & 2-nitrobenzaldehyde in the presence of piperidine in hot benzene (Ref 1 & 7).

2,4,6,3'-Tetranitrostilbene, trans, yel pigts (from benz + alc), mp 159°; was prepd by reacting 2,4,6-TNT & 3-nitrobenzaldehyde in the presence of piperidine in boiling benz (Ref 1 & 7).

2,4,6,4'-Tetranitrostilbene, yel ndls, mp 196°; readily sol in boiling glc 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,

\( \text{O}_2\text{N} \cdot \text{C}_6\text{H}_4 \cdot \text{CH} \cdot \text{CH} \cdot \text{C}_6\text{H}_4(\text{NO}_3)_2 \) (Ref 3, 8 & 9), was prepd by action of alcoholic KOH on 2,4-dinitrobenzyl chloride; forms the lower-melting form on exposure to light: orangered crystals (from glc acet ac), mp 263°; stable in sunlight; sol in hot Nitrobenz & hot glc acet ac; insol in acet & chllf (Refs 3, 8 & 9).

2,6,2',6'-Tetranitrostilbene or 1,2-Bis-(2,6-dinitrophenyl)-ethylene, tr-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.


Pentanitrodiphenylethylene or 2,4,6,2',4'-Pentanitrostilbene or 1-(2,4,6-Trinitrophenyl)-2-(2,4-dinitrophenyl)-ethylene,

\( \text{(O}_2\text{N})_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CH} \cdot \text{CH} \cdot \text{C}_6\text{H}_4(\text{NO}_3)_2 \) (Ref 1 & 7), was prepd by heating 2,4,6-trinitrotrans stilbene with nitric acid (d 1.41) & concd sulfuric acid at 100°. Oxidation 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.


Hexanitrodiphenylethylene, 2,4,6,2',4',6'-Hexanitrostilbene (HNS) or 1,2-Bis-(2,4,6-Trinitrophenyl)-ethylene,

\( \text{(O}_2\text{N})_2 \cdot \text{C}_6\text{H}_4 \cdot \text{CH} \cdot \text{CH} \cdot \text{C}_6\text{H}_4(\text{NO}_3)_2 \) (Ref 5), 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'-Hexanitrobenzil.

HNS is a heat-resistant expl developed at NOL (Ref 5). Several industries, such as Northrop-Carolina Inc, Del Mar Engineering Laboratories, and Jet Research Inc, now manuf HNS under Navy Procurement Specification WS-5003C & D (Ref 7). Del Mar, an incipitating 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:

(O2N)2C6H4CH:CH:C6H4(NO2)2; mw 405.24
N 17.28%; gm-yel ndls (from benz + acetone), mp 198–99°; prepd by heating 2,4,6-trinitrotrans stilbene with nitric acid (d 1.41) & concd sulfuric acid at 100°. Oxidation 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).
**N,N'-Diphenyl-ethylenediamine and Its Nitroated Derivatives.** See Bis(anilino)ethane and Derivatives in Vol 2, pB131-L. Its Hexanitro compd is code-named Bitetrol, Ditetrol or Octyl and is described on pp B131-R & B132-L.

---

**N,N'-Diphenyl-ethylene Ether or Stilbene Oxide and Derivatives (called a,a'-Diphenyl-ethylenoxyd or Stilbenoxyd in Ger),**

\[
\text{C}_6\text{H}_5\text{CH}==\text{CH}\text{C}_6\text{H}_5
\]

mw 196.24, O 8.15%; exists as trans form, melt (from 70% alc), mp 69–70.3°; v sl volatile on a steam bath; sol in eth; was prepd by treating N,N-dimethylidiphenylhydroxyethyamine-iodomethyate 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)

**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

---

<table>
<thead>
<tr>
<th>Melting point, °C</th>
<th>Type I</th>
<th>Type II</th>
</tr>
</thead>
<tbody>
<tr>
<td>°F</td>
<td>601(dec)</td>
<td>607(dec)</td>
</tr>
<tr>
<td>Particle size, microns</td>
<td>1–5</td>
<td>100–300</td>
</tr>
<tr>
<td>Bulk density, g/cc</td>
<td>0.32–0.45</td>
<td>0.45–1.0</td>
</tr>
<tr>
<td>Differential thermal analysis, onset of exotherm</td>
<td>315°C(599°F)</td>
<td>325°C(617°F)</td>
</tr>
<tr>
<td>Electrostatic spark sensitivity, fires above, μA</td>
<td>0.001 @8kv</td>
<td>0.0001 @17kv</td>
</tr>
<tr>
<td>Friction sensitivity, 10/10 No fires</td>
<td>440 kg/cm</td>
<td>440 kg/cm</td>
</tr>
<tr>
<td>Impact sensitivity, 50% height, cm</td>
<td>44</td>
<td>61</td>
</tr>
<tr>
<td>Vacuum stability @ 260°C</td>
<td>1st 20mins, cc/g/hr</td>
<td>0.3</td>
</tr>
<tr>
<td>Addnl 2hrs,cc/g/hr</td>
<td>0.6</td>
<td>0.2</td>
</tr>
<tr>
<td>@280°</td>
<td>2.7</td>
<td>2.7</td>
</tr>
<tr>
<td>Velocity of deton, m/sec</td>
<td>7000</td>
<td>7000</td>
</tr>
<tr>
<td>density, g/cc</td>
<td>1.70</td>
<td>1.70</td>
</tr>
<tr>
<td>Autoignition point</td>
<td>325°C(618°F)</td>
<td>325°C(618°F)</td>
</tr>
<tr>
<td>Decomposition rate @260°</td>
<td>0.1%/hr</td>
<td>0.1%/hr</td>
</tr>
<tr>
<td>Heat of combustion, cal/g</td>
<td>3451</td>
<td>3451</td>
</tr>
<tr>
<td>Mean firing voltage for EBW, 1 μfd</td>
<td>12950 @</td>
<td>12950 @</td>
</tr>
<tr>
<td>capacitor</td>
<td>0.9g/cm^3</td>
<td>0.9g/cm^3</td>
</tr>
</tbody>
</table>
D 1458

Dinitro-α,α′-diphenyl-ethylene Ether or Dinitrostilbene Oxide,
\[ \text{O}_2\text{N.C}_6\text{H}_4\text{HC} = \text{CH.C}_6\text{H}_4\text{O} \]

mw 286.24, N 9.79%. Three isomers are known:
2,4′-Dinitrostilbene Oxide, high-melting form:
crystals (from aceton or ethyl acetate), mp 160–61°; readily sol in chl, benz, acetone, glacial acetic acid & ethyl acetate; sl sol in alc & eth; v sl sol in w; and low-melting form: leaflets (from MeOH) & crystals (from alc), mp 127–28° & 112° (uncorrected); same solubility as h-melting form. Both forms prep'd 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: crystals (from MeOH or propyl alc), mp 116°. Both forms obt'd by heating 4-nitrobenzylchloride with 3-nitrobenzaldehyde in the presence of \( \text{K}_2\text{CO}_3 \) in MeOH (Refs 1 & 3)

4,4′-Dinitrostilbene Oxide, high-melting form:
tablets (from glacial acetic acid) or crystals (from ethyl acetate), mp 200–01°; sol in hot nitrobenzene, sl sol in glacial acid, alc, chl, acet & dioxane; and low-melting form: ndis (from glacial acetic acid), mp 162–63°. Both forms obt'd by heating 4-nitrobenzylchloride & 4-nitrobenzaldehyde with alcoholic KOH at RT, or by heating with \( \text{K}_2\text{CO}_3 \) in MeOH (Refs 1 & 3)

Refs: 1) Beil 17, [72, 73] 2) O. Chrzescinski, Dissertation, Strassburg (1911), 42, 43

NOTE: Higher nitrated derivs of Stilbene Oxide, such as Trinitro, \( \text{C}_4\text{H}_4\text{N}_3\text{O}_7 \), Tetranitro, \( \text{C}_4\text{H}_8\text{N}_4\text{O}_8 \), Pentanitro, \( \text{C}_4\text{H}_7\text{N}_5\text{O}_{11} \), and Hexanitro, \( \text{C}_4\text{H}_6\text{N}_6\text{O}_{13} \), were not found in the literature thru 1966

Diphenyl-ethyleneglycol Ether or Diphenoxystilbene. See Bis(phenyl)-ethyleneglycol Ether in Vol 2, p B153-R

1,1-Diphenyl-ethylene Peroxide, Polymeric,
\[ +\text{O}_2\text{H}_2\text{C.C}(\text{C}_6\text{H}_5)_2\text{O} \]

mp (212.24)x, O (15.08%); solid, mp – detonates on heating; can be prep'd by exposing 1,1-diphenylethylene, \( \text{H}_2\text{C.C(Ph)}_2\text{O} \), 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) T. Kobolsky & Mersobian (1954), 22 & 178

Diphenyl-ethylol and Derivatives
1,2-Diphenyl-1-ethylol; Diphenylethanol or Diphenyl-hydroxyethane,
\( \text{C}_6\text{H}_5\text{CH(OH)} \). \( \text{CH}_2\text{C}_6\text{H}_5 \); mw 198.25. Exists as inactive form: crystals (from eth & petr eth), dil alc or glacial acetic acid, mp 62–68°; its levorotatory form: crystals from eth & petr eth, mp 67°; and as dl-form: crystals, mp 66–69° (Ref). Other props & methods of prepn are given in Beil
Refs: Beil 6, 683, (329), [637, 638] & [3390]

2-Nitro-1,2-diphenyl-1-ethanol,
\( \text{C}_6\text{H}_5\text{CH(OH).CH(NO)}_2 \). \( \text{C}_6\text{H}_5 \); mw 243.25, N 5.76%; crystals (from benzene + petr eth), mp 97–98°; was first prep'd by Dornow & Gellrich (Ref 2) by reaction of phenyl-nitromethane (\( \text{PhCH}_2\text{NO}_2 \)) in \( \text{NaOH} \) with benzaldehyde treated with \( \text{NaHSO}_3 \) in \( \text{H}_2\text{O} \). See also Refs 3–6
Refs: 1) Beil, not found 2) A. Dornow &
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)

3,4'-Dinitrodiphenyl-1-ethylol or 1-(3-nitrophenyl)-2-(4-nitrophenyl)-ethanol,
O₃N·C₆H₄·CH(OH)·CH₂-C₆H₄·NO₂; mw 288.25, N 9.72%; crystals (from alc), mp 147–48°; sol in alc, glacial ac, ethyl acetate & acetone; less sol in eth & benz;
was prepd by heating 3-nitrobenzaldehyde & 4-nitrophenol in acetic acid with piperidine present for 1 hour at 110° & 2 hrs at 150°;
adda of water peps 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)

2-Nitro-1,2-diphenylethanol Nitrate,
C₆H₅·CH(ONO₂)·CH(ONO₂)·C₆H₅; mw 288.25, N 9.72%; crystals (from petr eth), mp 165–66° (dec); was obtd by action of dinitrogen peroxide (N₂O₅) in methylene chloride on trans-stilbene in tetraethylenmonium nitrate at minus 20° for 15 mins (Ref 2)
Refs: 1) Beil, not found 2) T.E. Stevens, J Org Chem 24, 1137 (1959) & JACS 81, 3593 (1959); CA 54, 5566 & 22706 (1960)

Trinitrodiphenyl-ethylol, C₁₂H₁₁N₅O₇, not found in the literature thru 1966

Tetranitrodiphenyl-ethylol, C₁₄H₁₀N₄O₉, not found in the literature thru 1966

2,4,2',4',5'-Pentanitrodiphenyl-ethylol or
(2) 2-(2,4-Dinitrophenyl)-1-(2,4,5-Trinitrophenyl)-2-ethanol,
(O₂N)₂C₆H₅·CH(OH)·CH₂-C₆H₃(NO₂)₃; mw 423.25, N 16.55%, OB to CO₂ 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 (Ref 2, 3 & 4)
Refs: 1) Beil 6, [6393] 2) H.A. Aaronson & W.H. Rinkenbach, JACS 52, 5042 (1930)

2,4,6,2',4',6'-Hexanitrodiphenyl-ethylol or Dipicryletibanol, C₁₄H₈N₆O₈, not found in the literature thru 1966

N,N'-Diphenylformamidine and Derivatives
N,N'-Diphenylformamidine or Methenyl-diphenylformamidine, C₆H₅·N·CH·NH·C₆H₅; mw 196.24, N 14.28%; ndls (from alc or benz), mp 139–43°; bp – sl decmp at higher temp; very sol in chl, 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); Ann Chirn [3], 54, 197 (1858) & Jahresber Fortschr der Chemie 1858, 354 3) W. Bradley & I. Wright, J Chem Soc 1956, 644 & CA 50, 13806 (1956) (Metal derivs)

Azido, C₁₃H₁₁N₃, and Diazido, C₁₃H₁₀N₅, derivs of N,N'-Diphenylformamidine were not found in the literature thru 1966

Mononitrodiphenylformamidine, C₁₄H₉N₂O₂; mw 241.24, N 17.43%. The following derivs are found in the literature:
**D 1460**

**N-Phenyl-N'-(3-Nitrophenyl)-formamidine, C₆H₅-N-ChNH.C₆H₄NO₂ or C₆H₅-NH.CH:NC₆H₄.NO₂; yel prisms (from abs alc), mp 145°; was prepd from reaction of 3-nitroisofomaranilide-O-methylether, (O₅N.C₆H₅-N:CH.O.CH₃), & aniline (Refs 1, 2 & 3)**

**N-Phenyl-N'-(4-Nitrophenyl)-formamidine, yel crystals (from toluene & then from alc), mp 181-85°; was obtld when p-nitroaniline & ethyl-N-phenylformamidate, [HC(:N.C₆H₄)OOC₂H₅] 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)

**Dinitrodiphenylformamidine, C₁₃H₁₂N₄O₄; mw 286.24, N 19.58%. Three isomers are known:**

**N,N'-Bis(2-nitrophenyl)-formamidine, O₂N.C₆H₅.N=N.CH.NH.C₆H₄NO₂, ndls, mp 160-61° [Beil, 124-25°; Ref 1]; was prepd by interaction of P pentachloride in a chlf medium upon o-nitroanilamide & 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-nitroisofomaranilide & 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 & decmp on heating (Ref 6)


**NOTE:** Higher nitrated derivs of Diphenylformamidine, such as Trinitro, C₁₃H₉N₃O₆, Tetranitro, C₁₃H₉N₄O₈, Pentanitro, C₁₃H₇N₇O₁₀ and Hexanitro, C₁₃H₆N₈O₁₂, were not found in the literature thru 1966

**Diphenylfuroxan and Derivatives**

**Diphenylfuroxan, Bis(phenyl)-furoxan or 3,4-Diphenylfuran Oxide, C₆H₅—C—C₆H₅ N.O.N.O mw 238.24, N 11.76%; ndls or leaflets (from alc or glc acet ac), mp 112-15°; bp — decmp 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)


**Dinitro-3,4-diphenylfuroxan or Bis(nitrophenyl)-furoxan Oxide,**
O2N.C6H4.C==C==C6H5NO2;

N.O:N.O

mw 328.24, N 17.07%. Two isomers are known:

**Bis-(3-nitrophenyl)-furaxan,** ndls (from glut acet ac or acet) & yel ndls (from xylene), mp 183–85°; readily sol in hot xylene, warm glut acet ac & acetone; insol in w, alc, eth, pett eth, benz & chlf; was prepd by oxida-
tion of 3-nitro-a-benzaldehyde with isomaylnitrite 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)-furaxan,** ndls (from alc or glut acet ac), mp 197–98°; readily sol in
warm glut acet ac; almost insol in w, alc, eth, benz & pett 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:** Tetrinitro, C14H4N6O10, and
**Hexanitro, C14H4N6O14** derivs of Diphenyl-
furaxan were not found in the literature thru
1966

**Diphenyl-glycerylether and Derivatives**

*a,a'-Diphenylglycerylether or 1,3-Diphenoxy-
2-propanol,C6H5—O.CH2.CH(OH).CH2.O—C6H5,
mw 244.28, O 19.65%; pearly pltls (from alc),
mp 80–82°, bp 224.5° at 17.5mm Hg, sublines
on careful heating w/o decomp, sp gr 1.749
at 24/4°; insol in w; sol in eth, chlf, benz &
hot alc; can be prepd by heating (C) epi-
chlordihydrin with Na phenolate & phenol in
dioxane at 100–05° and by other methods
(Ref 1)

1,3-Bis-O-(2,4-dinitrophenyl) glycererin or
1,3-Bis(2,4-dinitrophenoxy)-2-propanol,
(O2N)2C6H4.O.CH2.CH(OH).CH2.O.C6H5(NO2)2;
mw 424.28, N 13.21%; crys (from acet), mp
173.5–174°(Pt block). It was obtb with other
products by heating 4-chloro-1,3-dinitroben-
zene wth glycererin & NaOH (Refs 2 & 5)

No expl props are reported

1,3-Di-O-picrylglycererin; 1,3-Dipicryhydroxy-
2-propanol; or 1,3-Bis-(2,4,6-trinitrophenyoxy)
2-propanol;

(O2N)3C6H4.O.CH2.CH(OH).CH2.O.C6H5(NO2)3;
mw 514.28, N 16.34%; crys, mp 173–75°;

a color reaction occurs by treating the compd
in alc or acet soln wth alc KOH or alc NH3.

It was prepd by heating 1,3-bis-(2,4-dinitro-
phenoxy)-2-propanol wthaq nitric acid &
conc sulfuric acid (Refs 3 & 6)

No expl props are reported

1,3-Dipicryglycerylether-β-Nitrate or 2,4,6-
2',4',6'-Hexanitrodiphenyleglycerylether Nitrate,
(O2N)2C6H4.O.CH2.CH(ONO2).CH2.O—
C6H5(NO2)3; mw 559.28, N 17.53%, OB to CO2
minus 50%; dk-yel solid, mp 158–62°(dec).

It can be prepd by nitrating the parent compd in
sulfuric acid wth the usual mixed acid (Refs
4 & 5)

Its Impact Sensitivity with 2kg wt app is
115cm vs 40cm for Picric Acid. **Power by**
Trazul Test — 11% of TNT (Refs 7 & 8)

Refs: 1) Beil 6, 149, (86), [152] & [15821

2) Beil 6, (126) & [1853]

3) Beil 6, [1971]

4) Beil 6, (141)

5) Dynamite Nobel, BritP
24352(1914) & CA 10, 1597(1916); GerP
294813(1916)

6) L. Desvergne, AnnChim-
25, 811, 817(1931)

7) A.H. Blatt & F.C.
Whitmore, ORSD Rep 1085(1942), p47
8) A.H. Blatt, ORSD 2014(1944)

**Diphenylglycolic Ether.** See Bis(phenyl)-
edylene-glycol Ether in Vol 2, p B153-R

**Diphenylglycoluril and Derivatives**

7,8-Diphenylglycoluril, Diphenylacetylenedi-
ourein or Diphenylacetyleneurea.
N,N'-Diphenylhydrazine and Derivatives
N,N'-Diphenylhydrazine or Hydrazobenzene, C₆H₅-NH-NH-C₆H₅; mw 184.23, N 15.21%; rhomb bipyramidal tablets (from alc + eth), mp 126-27°C, sp gr 1.15 at 16°C; sol in alc; sl sol in benz, water & cold acet; was prep'd by hydrogenizing of nitrobenzene in the presence of Ni in aq NaOH under pressure at 110-120°C (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 mixes (Ref 7)

Dinitro-N,N'-diphenylhydrazine or Dinitrohydrazobenzene, C₁₂H₁₀N₄O₄; 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, C₁₂H₆N₈O₈; mw 364.23, N 23.08%. Two isomers are found in Beil:

2,4,2',4'-Tetranitrohydrazobenzene, (O₂N)₂C₆H₃.NH.NH.C₆H₃(NO₂)₂, yel crystals (from acet + alc), mp 250°C; sol in glc acet ac, concd sulfuric acid with yel color & in NaOH with blue color; insol in benz; was prep'd 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, (O₂N)₃C₆H₂.NH.NH.C₆H₄(NO₂), yel crystals (from a little eth acet + alc), mp 210-14°C; readily sol in glc acet ac, eth acet, alc, acet, alkali & concd sulfuric acid; can be prep'd by reaction of 4-nitrophenylyhydrazine & Picrylchloride in the presence of Ca carbonate in hot alc (Ref 3 & 6)

No expl props of the tetranitro derivs are reported

N,N'-Bis(mono- or di-)nitrophenylguanidine and Melanoline or N,N'-Bis(phenyl)-guanidine. See Bis(phenyl)-guanidine in Vol 2, p B154-L. It was proposed by E. Wyder [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 propels are given in conf Proplnt Manual SPIA/M2 (1962) (Unit Nos 618, 619, 620, 647, 64 & 533)

Nitrated cmpds: N,N'-Bis(mono- or di-)nitrophenylguanidine and N,N'-Bis(2,4,6-trinitrophenyl)-guanidine or Hexanitrophenylguanidine are described in Vol 2, p B154
glac acet ac, alkali & ethyl acet; less sol in alc, eth & benz; was prep'd 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, (O₂N)₃C₆H₂-NH.NH.C₆H₂(NO₂)₃; mw 544.23, N 24.67%; lt-yel n dl's (from gluc 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 prep'd by reaction of 1 mol hydrazine hydrate & 2 mols Picrylchloride in hot alc; or from reaction of equimol mixt of Picrylhydrazine & Picrylchloride at 120°

This compd forms a Potassium salt, K₃C₁₂H₈N₈O₁₂, green crysts having a metallic luster (from acet + alc), mp approx 188°, explodes on heating rapidly; sl sol in alc with a red color; v sl sol in alc; insol in w. It also forms a Dipotassium salt, K₅C₁₂H₄N₆O₁₂, gm platits, sol in acet & w with a blue to blue-violent 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: F1 is 74% PA

2,3-Diphenyl-5-hydroxytetrazolium Hydroxide [called 2,3-Diphenyl-5-oxo-tetrazoliumhydroxyl or 2-Phenyl-tetrazolon-(5)-hydroxyphenylat- (3) in Ger]

OC ——— N
HN.N(C₆H₅).N(C₆H₅.OH) or
HO.C ——— N
N.N(C₆H₅).N(C₆H₅.OH)
(See its Anhydride, Diphenylcarbodiabzone, in this Vol)

1,5-Diphenyl-1',5-iminosbis (1,2,3,4-tetrazole) [called [1-Phenyl-tetrazolyl-(5)]-5-phenyl-tetrazolyl-(1)]-amin in Ger]
C₆H₅.C — N.NH.C — N.C₆H₅
N.N:N
N.N:N
mw 305,30, N 41.29%; ndls (from alc), mp 192°(dec), deflgl 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 gluc acet ac, toluene & xylene; stable in alkaline & mineral acid solns. It was prep'd by heating bis(α-azidobenzylidene)-hydrazine with Na azide in alc & CO₂ stream. Its Silver salt is stable in light, sol in ammonium hydroxide, and deflgl on heating in a flame (Refs 1 & 2)
Refs: 1) Beil 26, (344) 2) R. Stolle & A. Netz, Ber 55B, 1301, 1303(1922) & CA 16, 3899(1922)

Diphenyl-iminoura. 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, SC ——— N
HN.N(C₆H₅).N(C₆H₅.OH) or
HS.C ——— N
N.N(C₆H₅).N(C₆H₅.OH)
(See its Anhydride, Diphenylthiocarbodiabzone, in this Vol)
Diphenyl-methane and Derivatives

Diphenyl-methane, Methylene-diphenyl, Dilane or Benzylenzene, C₆H₅-CH₂-C₆H₅; mw 168.23; col crys., 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, (C₆H₅)₂CHN₃, mw 209.24, N 20.08%; liq, bp 135° at 4mm pres., nD₃ 1.5883 at 22°. It was obt'd with other products when diphenylmethanol in chl reacted with hydrazoic acid-sulfuric acid with subsequent addn of ice & warming (Ref 8)

No expl props are reported (Refs 7, 9, 11 & 12)

a,a-Diazidodiphenylmethane or Benzenophene Dioxide, C₆H₅.C(N₃)₂.C₆H₅; mw 250.26, N 35.58%; crys (from MeOH), mp 42°, vapor press 0.04mm at 80° & 0.10mm at 90°, 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 alkalis; reacts explosively with sulfuric acid even in the cold; sensitive to shock. It was prep'd & isolated in 88% yield from reaction of dichlorodiphenylmethane & AgN₃ in ether soln (Refs 3, 4, 5 & 6)

See also Benzophenonedioxide in Vol 2 of Encycl, p B77-R


Diphenylmethane Hydroperoxide or Benzohydryl Hydroperoxide, (C₆H₅)₂CH.OOH; mw 202.23, O 15.98%; almost odorless nds or columns (from benz & eth acet), mp 51°, bp - decomp at 135° (gas evolution), delgr above 170°; even in absence of air & light it decomp in 6 weeks. It was obt'd 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 Na₂SO₄ sole, and with 10% sulfuric acid, it is cleaved to phenol & benz (Ref 2)

β-diketones as oxidation initiators)


12) Ruhrchemie Aktr-Ges, GerP 1041960 (1958) & CA 55, 11461 (1961) (Prepn by oxidizing Ph₂CH₂ with Mg phthalocyanine at 105° in the presence of O for 10 hrs)

13) D.H. Sharp & B.S. Wildi, USP 2883427 (1959) & CA 53, 17062 (1959) (Prepn from Ph₂CH₂ & O at 140–80° for <3 hrs)


Mononitrodiphenylmethane, C₁₃H₁₁NO₂; mw 213.23, N 6.57%. The following derivs are found in Beil: a-Nitrodiphenylmethane, (C₅H₄)₂CHNO₂, colorless oil, fr p – below –15°, sp gr 1.1727 at 20° (Ref 1)

a-acid-Nitrodiphenylmethane, (C₅H₄)₂C₆H₄NO₂, prisms (from eth), mp 90° (dec), unstable even at RT (Ref 2)

2-Nitrodiphenylmethane, C₆H₅.CH₂-C₆H₄NO₂, lt-yel liq, bp 183–84° at 10 mm 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 11 mm press, sp gr 1.278 at 0° (Ref 5)


Dinitrodiphenylmethane, C₁₃H₁₀NO₂₄, mw 258.23, N 10.85%. Several isomers are known: a,a-Dinitrodiphenylmethane, (C₅H₄)₂C(NO₂)₂, pltls (from dil alc), mp 78–78.5°; readily sol in chl, eth, benz & hot alc (Ref 1)

2,2’-Dinitrodiphenylmethane,

O₃N.C₆H₄.CH₂-C₆H₄NO₂, pltls or ndls (from w), mp 83.5°; sl sol in alc & eth; insol

in petr eth (Ref 2)

3,3’-Dinitrodiphenylmethane, pltls (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 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, C₁₀H₇.CH₂.C₆H₄(NO₂)₂, nor 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


Trinitrodiphenylmethane, C₁₃H₉NO₃, mw 303.23, N 13.86%. Three isomers are decribed in the literature:

x,x,x-Trinitrodiphenylmethane, C₁₃H₉(NO₂)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,

(O₂N)₂C₆H₄.CH₂.C₆H₄(NO₂), crysts (from alc), mp 111–13°; was obtd when 2,2’-dinitrodiphenylmethane in concd sulfurrific 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 107–109°; 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.


(Prep 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 2,4,4'-Tetranitro-diphenylmethane,

\((\text{O}_2\text{N})_3\text{C}_6\text{H}_3\cdot\text{CH}_2\cdot\text{C}_6\text{H}_2(\text{NO}_2)_3\), mw 348.23, N 16.09%; crystals (from glc acet acid or benz), mp 173–81°. It was prep'd by nitrating diphenylmethane with nitric acid (d 1.53) & conc'd sulfuric acid (Ref 1) and by decarboxylation of ethyl bis(2,4-dinitrophenyl)-acetic acid (Ref 2). No expl props are reported.


2,4,6,2',4'-Pentanitro-diphenylmethane,

\((\text{O}_2\text{N})_3\text{C}_6\text{H}_2\cdot\text{CH}_2\cdot\text{C}_6\text{H}_2(\text{NO}_2)_3\), mw 393.23, N 17.81%; it-yel tablets (from glc acet acid), mp 200°. It was prep'd 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.


Hexanitrodiphenylmethane,

\((\text{O}_2\text{N})_3\text{C}_6\text{H}_2\cdot\text{CH}_2\cdot\text{C}_6\text{H}_2(\text{NO}_2)_3\), not found in the literature thru 1966

Diphenylmethanol and Derivatives

Diphenylmethanol, Diphenylmethylol, Benzhydrol or Diphenylcarbinol, \((\text{C}_6\text{H}_5)_2\text{CHOH}\), mw 184.23; ndls, mp 68–69°, bp 298.5°; sl sol in w; v sol in alc, eth, chl & CCl4; insol in ligroin; can be prep'd by reduction of benzophenone \((\text{C}_6\text{H}_5)_2\text{CO}\) with Mg or Zn dust and by other methods (Refs 1 & 2)


Azido, \(\text{C}_1\beta\text{H}_1\text{N}_3\text{O}_3\), and Diazido, \(\text{C}_1\beta\text{H}_1\text{N}_2\text{O}\), derivs were not found in the literature thru 1966

4-Nitrosodiphenylmethanol (called 4-Nitroso-phenylbenzyl Alcohol in CA), \(\text{ON.C}_6\text{H}_4\cdot\text{CH(OH).C}_6\text{H}_5\); mw 213.23, N 6.57%; yel ndls, mp 114–17°; was obt'd with 4,4'-dibenzoyl azoxybenzene when \(\beta\)-(p-nitroso-phenylmethylthio) 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)


Diphenylmethylnitrite, \(\text{C}_6\text{H}_5\cdot\text{CH(ONO).C}_6\text{H}_5\); mw 213.23, N 6.57%; yel liq, bp – decmp on distillation even at 65° and 0.1 mm press. It was prep'd 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)


Diphenylmethyl Nitrate, \(\text{C}_6\text{H}_5\cdot\text{CH(ONO)2.C}_6\text{H}_5\); 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 decmp with evolution of bm fumes; on heating to 140–50° it smoothly decomd to benzophenone; its soln in methyl fumes was hydrolyzed to diphenylmethanol by shaking with saturated bicate soln for several hrs. It is too unstable to be isolated by ordinary methods, but can be obt'd in soln., by heating under reflux diphenylmethanol chloride & an excess of powdered Ag nitrate in dry ether. The etheral soln was evaporated in a vacuum (See Refs)

Mononitrodiphenylmethanol or Mononitrobenzhydrol, C₆H₅C₂H₃(OH).C₆H₄.NO₂, mw 229.23, N 6.11%. Three isomers are known:

2-Nitrodiphenylmethanol, plts (from petr etich), mp 59-60° & 63-64° (Ref 6), bp 170-80° at 0.74 mm press (Refs 2, 3, 7 & 8)

3-Nitrodiphenylmethanol, crystals, mp 68-71°, bp 208-12° at 6 mm press (Refs 1, 2 & 6)

4-Nitrodiphenylmethanol, yel crystals (from benz or benzol), mp 73-78° (Refs 1, 4, 5 & 6)

Methods of prepn & other props are found in Refs


Dinitrodiphenylmethanol or Dinitrobenzhydrol, O₂N.C₆H₅.C₂H₃(OH).C₆H₄.NO₂; mw 274.23, N 10.22%. Three isomers are known:

2,2'-Dinitrodiphenylmethanol or Bis-(o-nitrophenyl)-methanol, crysts (from cyclo-hexane + ether), mp 126°; was obted when, to a stirred soln of 2,2'-dinitrobenzophenone in tetrahydrofuran at -35°, a soln of LiAl hydride in tetrahydrofuran was added drop-wise 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° & 10⁻⁷ mm press and crystallized (Ref 8) 3,3'-Dinitrodiphenylmethanol or 3,3'-Dinitrobenzhydrol, wh crysts (from 60% MeOH), mp 106.5-107.6°; was obted 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 obted by reducing 4,4'-dinitrobenzophenone 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'-Dinitrodiphenylmethylnitrate or 4,4'-Dinitrobenzhydrol Nitrate, O₂N.C₆H₅.C₂H₃(ONO₂).C₆H₄.NO₂; mw 319.23, N 13.16%; wh ndls (from alc or glc acet ac), mp 160.5-162°; was prepd by adding portion-wise 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 glc acet ac and an addtl 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.
Diphenyl-methylenediamine and Derivatives

Diphenyl-methylenediamine or N-Methyl-diphenylamine, C₆H₅.N(CH₃).C₆H₅; 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-methylenamine,
C₆H₅.N(CH₃).C₆H₅(NO₂)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,

C₆H₅.N(CH₃).C₆H₅(NO₂)₂ + C₆H₅.C₆H₅(NO₂)₂

It gives on nitration Hexanitrodiphenyl-methylenamine

2,4,6,2',4',6'-Hexanitrodiphenyl-methylenamine; Dipicryl-methylenamine or Bis(2,4,6-trinitrophenyl)methylenamine, (O₂N)₂C₆H₅.N(CH₃).C₆H₅(NO₂)₂; mw 453.24, N 21.63%, OB to CO₂ minus 123.5%; crysts, mp 236°-37°; can be prepd by nitrating of 2,4-dinitrodiphenyl-methylenamine, or by condensing 2,4-dinitrochlorobenzene with mono-methylaniline and nitrating the product (Ref 3)

Its expl props listed in Blatt (Ref 4) are as follows:

Impact Sensitivity – 1/2 of Picric Acid
Power by Trauzl Test – 87% Picric Acid

Ref: 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)

Dinitrodiphenylmethylenediamine or Bis-(nitroanilino)-methane.

O₂N.C₆H₄.NH.C₆H₄.NH.C₆H₄.NO₂; 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, ong yel ndls (from alc), mp 213°; almost insol in alc; insol in w, ether, chl & benz. It was prepd by addy a 40% formaldehyde soln to a hot concd soln of 3-nitroaniline in alc. It forms a Picroloic acid, C₁₃H₂₃NO₄ + 2C₆H₅N₃O₇, 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)

Ref: 1) Beil 12, 184, (168) & [110]
2) Beil 12, 185 & [110]
3) Beil 12, 690, 702, 717
4) CondChemDict (1961), 739-L

Tetranitrodiphenylmethylenediamine,
C₁₅H₁₈N₆O₈, not found in the literature thru 1966

Hexanitrodiphenylmethylenediamine,
C₁₅H₁₈N₆O₁₂, not found in the literature thru 1966
Diphenylmethylenedihydroxyphenylpropane and Derivatives
Diphenylmethylenedihydroxyphenylpropane, C_{9}H_{12}O_{2}, mw 316.38; may be considered as the parent compd of its Dipicryl deriv, although not used to prep;
1,3-Dipicryl-2-(3,4-methylenedithyloxyphenyl)propane,

\[
[(O_2N)_3C_6H_2CH_2]_2 CH O O CH_2
\]

mw 586.38, N 14.33%; yel crys (from glacial acet acid), mp 137-40°C (dec). It was prep'd from reaction of 1 mol piperonal & 2 mols TNT in the presence of piperazine or piperidine in pyridine; in addition to a little 2,4,6-trinitro-3',4'-methylenedithyloxystilbene (Refs 1 & 2)

Diphenylnitrosoamine. 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_{6}H_{4}C \_CH_{3}

HO.C_{6}H_{4}C \_CH_{3}, mw 228.28; O 14.02%. This compd and its Dinitro, C_{15}H_{14}N_{2}O_{6}, and Tetranitro, C_{15}H_{14}N_{4}O_{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


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_{6}H(NO_{2})_{3} C(CH_{3})_{2}

HO.C_{6}H(NO_{2})_{3} mw 498.28, N 16.87%; mp 145°C; ignition temp 363°C for 5 sec & 337°C for 10 sec; Trauzl Pb block test 88 vs 100 for PA; insensitive to flame. It was prep'd by nitrating the tetranitro deriv with 73-75/24.5-nitric acid/sulfuric acid (250% excess) at 20-50°C and 1.5 hrs at 85°C (Ref 2)

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_{8}H_{5}.NH.CO.CO.NH.C_{8}H_{5} mw 240.25, N 11.66%; pltls (from benz or Nitrobenz), mp 247-54°C; insol in cold alc, hot w & eth; sl sol in hot alc; sol in benz (Ref 1)
N,N'-Dinitrodiphenyloxamide; N,N'-Bis(nitrophenoxy)-oxamide or Dinitrooxanilide, 
O\textsubscript{2}N.C\textsubscript{6}H\textsubscript{4}.NH.CO.NH.C\textsubscript{6}H\textsubscript{4}.NO\textsubscript{2} \text{; mw 330.25, N 16.97%}. Three isomers are known: 
N,N'-Bis (2-nitrophenoxy)-oxamide, yel plates (from aniline), mp 331\textdegree \text{ (dec)}; insol in w, alc, 
eth, CS\textsubscript{2}, benz & acet; sl sol in glacial acet ac; 
sl in hot aniline (Ref 2) 
N,N'-Bis (3-nitrophenoxy)-oxamide, almost colorless ndls (from aniline), mp 309–10\textdegree \text{ (dec)}; 
&amp; orn-colored rhombic crysts - mp 307\textdegree \text{ (dec)}; 
sl sol in alc, eth & benz; sol in aniline (Ref 3) 
N,N'-Bis (4-nitrophenoxy)-oxamide, yel ndls (from aniline), mp 358–59\textdegree \text{ (dec)}; 
sl sol in hot alc, benz & CHCl\textsubscript{3}; sl sol in hot glacial acet ac 
Methods of prepn & other props are found in 
Beil 

N,N'-Bis-(2,4-dinitrophenyl)-oxamide or 
2,4,2',4'-Tetranitrooxanilide, 
CO.NH.C\textsubscript{6}H\textsubscript{4}.N\textsubscript{2}O\textsubscript{4} \text{2} 
| 
CO.NH.C\textsubscript{6}H\textsubscript{4}.N\textsubscript{2}O\textsubscript{4} \text{2}; mw 420.25, N 20.00%; 
yel ndls (from Nitrobenz), mp 306\textdegree \text{ (dec)}; sl 
sol in alc, benz & glacial acet ac. It was prepd by 
reacting 2,4-dinitroaniline & oxalylchloride 
in boiling benz (Refs 5 & 8) 
Pentanitrodiphenyloxamide, C\textsubscript{14}H\textsubscript{12}N\textsubscript{4}O\textsubscript{12}, not 
found in the literature thru 1966 

2,4,6,2',4',6'-Hexanitrodiphenyloxamide; 
2,4,6,2',4',6'-Hexanitrooxanilide or N,N'- 
Dipencycloxamide, CO.NH.C\textsubscript{6}H\textsubscript{4}.N\textsubscript{2}O\textsubscript{4} \text{3} 
| 
CO.NH.C\textsubscript{6}H\textsubscript{4}.N\textsubscript{2}O\textsubscript{4} \text{3}; mw 510.25, N 21.96%; 
crystals, mp 295–300\textdegree \text{ (dec)}; sl 
sol in hot alc & benz; very low expla temp. 
It was prepd by nitration oxanilide. Its power 
&amp; brisance are comparable with those 
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’Explo- 
sifs(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, 
"Teoriya Vzyryvyvatkh Veshchestv," SbStaitei 
1963, 53 & CA 59, 12585(1963) (Impact tester 
is described for deg 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 % expls and 
rebound of wt as a % of its fall are reported) 

Di(phenyloxy)-ethylene or Ethyleneglycol- 
diphenyl-Ether. This compd & its Dinitro, 
C\textsubscript{14}H\textsubscript{12}N\textsubscript{2}O\textsubscript{2}, Tetranitro, C\textsubscript{14}H\textsubscript{10}N\textsubscript{4}O\textsubscript{10}, and 
Hexanitro, which should read: 
CH\textsubscript{2}.O.C\textsubscript{6}H\textsubscript{12}(NO\textsubscript{2}) \text{3} 
CH\textsubscript{2}.O.C\textsubscript{6}H\textsubscript{12}(NO\textsubscript{2}) \text{3}, derivs are described in 
Vol 2 of Encycl, p B153-R to B154-L under 
Bis(phenyl)-ethylene glycol Ether and Derivatives 
Their addnl refs are as follows: 
Refs: 1) Beil 6, [5681] (Parent) 
2) Beil 6, [802, 8231 (Dinitro derivs) 
3) Beil 6, [564] 
(Tetranitro deriv) 
4) (?) Lippmann, CR 68, 
1269(1869) (Was first to prepare the parent 
compd by heating K phenolate with 1,2-dibromo- 
ethane on a water bath) 
5) (?) Weddige, 
JPakrChem 21, 127(1880) & 27, 201(1883) 
[Prepn of o-, m-, & p-dinitrocompounds, 
corresponding to formula : 
(O\textsubscript{2}N)C\textsubscript{6}H\textsubscript{4}.O.CH\textsubscript{2}-CH\textsubscript{2}.O.C\textsubscript{6}H\textsubscript{4}(NO\textsubscript{2}) by 
interaction of ethylenebromide on two molecules 
of alkaline salts of corresponding mononitro- 
phenols] 

Dill[(phenyl-phenylamino)-ethyl] amino-ethane. 
See 1,2-Bis[(phenyl-phenylamino)-ethyl]- 
amino-ethane in Vol 2, p B154-R 

Diphenylphénylendiéamine and Derivatives 
N,N'-Diphenylphénylendiéamine, 
C\textsubscript{6}H\textsubscript{4}.NH.C\textsubscript{6}H\textsubscript{4}.NH.C\textsubscript{6}H\textsubscript{4} \text{; mw 260.32, N 10.76%}. 
The N,N'-Diphenyl-o- (Ref 1); N,N'-Diphenyl- 
m- (Ref 2); and N,N'-Diphenyl-p-phenylene- 
diámine (Ref 3) derivs are known 
Refs: 1) Beil 13, [13] 
2) Beil 13, 42 & [26] 
3) Beil 13, 80, (23) & [41]
Azido, $C_{18}H_{16}N_{3}$, and Diazido, $C_{18}H_{14}N_{8}$, derivs are not found in Beil.

Dinitro-N,N'-diphenylphenylenediamine,
$C_{14}H_{15}N_{4}O_{4}$, 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 crystals (from glacial acetic ac), mp 170–71°C, almost insol in alc & eth (Refs 1 & 4)
N-Phenyl-N'(2,6-dinitrophenyl)-o-phenylenediamine, resembles a lacquer (Refs 2 & 7)
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°C (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 Nitrobenzene), mp 233°C (Refs 4 & 6)

Other props & methods of prep r are found in Refs
Refs: 1) Beil 13, 18 2) Beil 13, [13]
5) Jackson & Cohoe, AmChem
26, 4(1901) 6) B.L. Manjunath, JIndianChem-Soc 4, 277(1917) & CA 21, 3199(1927)
7) F. Kehrmann & E. Effont, Helv 4, 520(1921) & CA 15, 3449(1921)

Trinitro-N,N'-diphenylphenylenediamine,
$C_{18}H_{12}N_4O_6$; 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 prills (from alc), mp = decmp on heating to 120°C or by heating with alc NaOH at 100°C. 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-Trimethyl-1,3-dianilinobenzene,
$C_{24}H_{19}N_2H(NO_2)_3.NH.C_6H_5$, red ndls (from glacial acetic ac), mp 217°C. It was prep'd 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-(methyl nitramino-benzene) in benz (Refs 2 & 4)
Refs: 1) Beil 13, 18 2) Beil 13, 17
3) F. Kehrmann & (7). Kramer, Ber 33, 3074, 3075(1900) 4) C.F. van Duin, Rec 38, 93(1919)

Tetranitrodiphenylphenylenediamine,
$C_{18}H_{12}N_4O_8$; mw 440.32, N 19.09%
N-Phenyl-N'-picryl-4-nitro-1,2-phenylenediamine or 5,2',4',6'-Tetranitro-2-anilinodiphenylamine,
$C_6H_5.NH.C_6H_2(NO_2)_3.NH.C_6H_4(NO_2)_2$, bm-yel crystals (from glacial acetic ac), mp = decmp on heating above 174°C, sl sol in alc; in sol in w; obd 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_5.NH.C_6H_4.NH.C_6H_2(NO_2)_2, crystals (from MeOH or acetic ac), mp 288°C. It was prep'd by warming a mixt of p-phenylenediamine in acetic ac or NaHCO_3 & 2,4-dinitrofluorobenzene, cooling & acidifying with AcOH (Ref 4)

N,N'-Bis (2,5-dinitrophenyl)-1,4-phenylenediamine, bm-red crystals (from Nitrobenz), mp 288–289.5°C. It was obd when a mixt of 2,5-dinitro-4-aminodiphenylamine & bromo-p-dinitrobenzene, intimately mixed by grinding was heated for 2 hrs at 155–60°C, and extracting the product by refluxing with 95% alc (Ref 3)

The tetranitro derivs are probably expl

Pentanitro-N,N'-diphenylphenylenediamine,
$C_{14}H_{11}N_4O_{10}$, not found in the literature thru 1966

Hexanitro-N,N'-diphenylphenylenediamine;
N,N'-Dipicycl-1,4-phenylenediamine; or
N,N'-Bis (2,4,6-trinitrophenyl)-p-phenylenediamine,
(O_2N)_2C_6H_5.NH.C_6H_4.NH.C_6H_2(NO_2)_2
N,N'-Dipicryl-5-nitro-1,3-phenylenediamine or 5-Nitro-1,3-bis(2,4-dinitrooxinilino)-benzene, (O₂N)₂C₆H₄NH·C₆H₄(NO₂)₂NH·C₆H₄(NO₂)₂; mw 575.33, N 21.91%; orn-ored crystals (from alc), mp – darkens on heating to 240 °C and melts with decomp at 260 °C. Prepd by heating 5-nitro-1,3-phenylenediamine with picryl chloride & Na acetate (Refs 1 & 2)

N,N'-Bis(2,4-dinitrophenyl) -2,4,6-trinitro-1,3-phenylenediamines; or 2,4,6-Trinitro-1,5-bis(2,4-dinitrophenylamino)-benzene, (O₂N)₂C₆H₄NH·C₆H₄(NO₂)₂NH·C₆H₄(NO₂)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


Di(phenyl)-phenylhydrazone. See Triphenylhydrazone in future volume

1,4-Diphenyl-3,5-phenylimino-1,2,4-triazole; 1,4-Diphenyl-3,5-endoonilino-4,5-dihydro-1,2,4-triazole; 1,4-Diphenyl-3,5-endoonilino-1,2,4-triazoline or Nitron [called Anhydro(1,4-diphenyl-3-phenylimino-4-dihydro-1,2,4-triazoliumhydroxid) or Anhydro (1,4-diphenyl-3-anilino-1,2,4-triazolium hydroxid) in Ger],

HC – N·C₆H₅

H₅C₆N⁺ – N⁻ – C·N·C₆H₅ or

HC – N·C₆H₅

H₅C₆N⁺ – N⁻ – C·N·C₆H₅ or

HC – N·C₆H₅

H₅C₆N⁺ – N⁻ – C·N·C₆H₅; mw 312.36, N 17.94%; lemon-yel fine ndls (from chl + petr eth) or yel pils (from alc), mp 189 °C (dec); sol in chl, acet & eth acet; sl sol in eth & alc; can be prepd by heating trifenilaminoguanidine & formic acid, diluting with water, filtering, and ppgk the product with NH₄OH; forms numerous salts, some of which decom on heating (Ref 1)

Nitron is used as a reagent for detecting the nitrile ion (NO₂) in very dil solns (Ref 2)


Diphenylphthalate (DPhPht), C₆H₄(COOCC₆H₄)₂; mw 318.31; colorless prisms, sp gr 1.28 at 20 °C; mp 73 °C, bp 405 °C at 759 mm; flash p 435 °F; insol in w; sl sol in alc or eth; sol in acet. It can be prepd from phthalic chloride & phenol or from phthalic acid monophenyl ester, phenol & phosphorpentoide in benz (Refs 1, 2 & 3)

Méard & Thomas (Ref 4) determined its heat of combustion at Cₙ as 2245.9 kcal/mol and heat of formation as 116.9 kcal/mol

Technical DPhPht has been used as a plasticizer for NC in the manuf of propixs. 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 0.5 g sample to the nearest mg into a dry 250 ml Erlenm flask with a ground joint and add 20 ml of benzine. Dissolve by warming on an electric hot plate, remove from the plate and add to the warm soln 125 ml of 0.5N alcoholic KOH soln. Reflux at 60 °C for 2 hrs, using a water condenser. Stopper the flask, cool to RT, add 75 ml 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 phpt 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
\( N = \) nomality 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 appara-tus 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

**SETTING POINT APPARATUS**

which the center hole serves for 'standardized' thermometer T; one of the side holes serves for 'correction' thermometer T1, while the other for the stirring rod R (Nichrome or Al wire ca 1½mm diam with a ring (r))

**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 T1 in the middle of the air space between the surface of liq-uid 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 T1. Correct the maximum temp for emergent stem by adding the value calculated from the formula:
0.000159N(t–t₁),
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
$t₁$ = temp of the exposed Hg column
detd by means of thermometer $T₁$
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. Méard & M. Thomas,
(1961), p 408-R

Di(phenylpicrylhydrazino)-ethane. See $a$,β-
Bis($a$-phenyl-$β$-picryl-hydrazino)-ethane in
Vol 2, p B154-R

Diphenylpiperazine and Derivatives

$N,N'\text{-Diphenylpiperazine}$,

\[
\text{C}_6\text{H}_5\text{N} \rightleftharpoons \text{CH}_2\text{CH}_2 \rightleftharpoons \text{N.C}_6\text{H}_5
\]

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 ethylene-
bromide in the presence of anhydrous Na car-
bonate. Schouten (Ref 3) prep'd 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
prepared with abs 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
Bitearyl or Ditetaryl (US) and Octyl (GB Britain).
Properties and prep of this compd are de-
scribed in Vol 2 of Encycl, p B131-R

The reaction observed by Schouten pro-
ceeded as follows:

\[
\text{C}_6\text{H}_5\text{N} \rightleftharpoons \text{CH}_2\text{CH}_2 \rightleftharpoons \text{N.C}_6\text{H}_5\text{HNO}_3
\]

\[
(\text{O}_2\text{N})_2\text{C}_6\text{H}_2\text{N(NO}_2\text{).CH}_2\text{CH}_2\text{N(NO}_2\text{).C}_6\text{H}_2(\text{NO}_2)\text{H}
\]

(Ditetaryl), 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

Bischoff, Ber 22, 1778(1889) 3) A.E.
Schouten, Rec 56, 870(1937) & CA 31, 7046
(1937)

$N,N'\text{-Bis(4-nitrosophenyl)-piperazine,}$

ON.C\text{C}_6\text{H}_4\text{N}:\text{C}_6\text{H}_5\text{N}:\text{C}_6\text{H}_4\text{N}:\text{NO}_2$; mw 296.32,
N 18.91%, gm-yel ppt or dk ndls (from acet-alc),
mp – decomp completely at 180°. It was prepd
by reaction of Na nitrite & $N,N'\text{-diphenyl-
piperazine in dil HCl}$ (Refs 1 & 2)

Refs: 1) Beil 23, 8 2) Morley, Ber 12,
1795(1879) 3) M.W. Swaney & S.B. Lip-
pincoitt, 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 vulcaniza-
tion activator) 5) R. Fabre & G. Bertrand,
RevGenCaoutchouPlastiques 42(3), 405
(1965) (Fr) & CA 63, 10149(1965) (Use same
as Ref 4)

$N,N'\text{-Dinitrodiphenylpiperazine,}$

\[
\text{O}_2\text{N.C}_6\text{H}_4\text{N} \rightleftharpoons \text{CH}_2\text{CH}_2 \rightleftharpoons \text{N.C}_6\text{H}_4\text{N}:\text{NO}_2
\]

mw 328.32, N 17.07%. Four isomers are known:
$N,N'\text{-Bis(2-nitrophenyl)-piperazine,}$, nm pldts,
mp 193–94°(Ref 2)
$N,N'\text{-Bis(3-nitrophenyl)-piperazine,}$, brn-yel
ndls (from acet), mp 220°(Ref 1) & om crys,
mp 206°(Ref 3)
$N,N'\text{-Bis(4-nitrophenyl) -piperazine,}$ crys
(from Nitrobenz), mp 261°(dec) (Ref 5) &
248°(dec) (Ref 1)
**N-(2,4-Dinitrophenyl)-N'-phenylpiperazine,**

\[
\begin{align*}
(O_2N)_2C_6H_3N & \ \overset{\text{CH}_2\text{CH}_2}{\longrightarrow} \ \overset{N\text{C}_6H_5}{\text{CH}_2\text{CH}_2} \\
\end{align*}
\]

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, J Chem Soc 1930, 149 & CA 24, 1863(1930)

**N,N'-Tetranitrodiphenylpiperazine,**

\[
\begin{align*}
(O_2N)_2C_6H_3N & \ \overset{\text{CH}_2\text{CH}_2}{\longrightarrow} \ \overset{N\text{C}_6H_6(NO_2)_2}{\text{CH}_2\text{CH}_2} \\
\end{align*}
\]

mw 418.32, N 20.09%. Two isomers are known: N,N'-Bis(2,4-dinitrophenyl)-piperazine, om-red ndls (from Nitrobenz), mp – dec with effervescence at 240°; sl sol in glc ace, pyridine, acet & chlf; can be prep'd by heating piperazine hydrate with 3 mols 4-chloro-1,3-dinitrobenzene (Refs 1, 2, 3 & 4)

N,N',N''(2,6-Dinitrophenyl)-piperazine, crysts, mp 269°. It was prep'd by refluxing for several hrs a mixt of halo-dinitrobenzene, piperazine, Na carbonate & anhyd alc (Ref 4)

Other props & methods of prepns are found in the Refs


**N,N'-Bis(2,4,6-trinitrophenyl)-piperazine or Dipicyryl piperazine,**

\[
\begin{align*}
(O_2N)_3C_6H_2N & \ \overset{\text{CH}_2\text{CH}_2}{\longrightarrow} \ \overset{N\text{C}_6H_6(NO_2)_3}{\text{CH}_2\text{CH}_2} \\
\end{align*}
\]

mw 508.32, N 22.05%; orn-yel crysts (from Nitrobenz), mp – dec at 287°. It was prep'd 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


**Diphenylpropene.** 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-quinamethane and Derivatives**

**Diphenyl-quinamethane or Fuchsone,**

\[(C_6H_5)_2C:C_6H_4:O, \ mw 258.30, bm-yel ndls, mp 168–69°, insol in w, sl sol in eth, sol in ace; can be prep'd by heating diphenyl(4-hydroxyphenyl)-methanol in glc acet ace & concentrating the soln at 90°(Refs 1 & 4)

**Mononitrodiphenylquinomethane,**

\[(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 pett eth. It was prep'd by heating 3-nitro-4-methoxytritylphenylchloromethane for 3 hrs at 180°(Refs 2 & 3)

**Dinitro, C_{19}H_{11}N_{2}O_{5}, Tetranitro, C_{19}H_{11}N_{3}O_{7}, and Hexanitro, C_{19}H_{11}N_{4}O_{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, Dissertation (Freiburg, Schweiz) (1919), 19 4) I.S.

Diphenylselenide and Derivatives

Diphenylselenide or Selenium diphenyl,
(C₆H₅)₂Se, mw 233.16; oil, sp gr 1.338 at 16/4°, bp 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)


Dinitrodiphenylselenide or Bis (Nitrophenyl) - selenide, O₃N.C₆H₄Se.C₆H₄NO₂; mw 323.16, N 8.67%. Three isomers are known:

2,2'-Dinitrodiphenylselenide, mp 143° (Refs 1 & 4)

4,4'-Dinitrodiphenylselenide, pale yel prisms (from ethyl acetate), mp 170-75° (Refs 2, 3, 5, 6, 7 & 8) and

2,4-Dinitrophenyl-phenylselenide, C₆H₄Se.C₆H₄(NO)₂, no description of compd is found in abstract (Ref 6)


Diphenylsulfide and Derivatives

Diphenylsulfide or Bis(phenyl)sulfide,
(C₆H₅)₂S, mw 186.26, colorless liq, sp gr 1.1,19 at 15/15°, fr p minus 25.9°, bp 296-97°; insol in w; sol in benz, eth & CS₂; sol in hot alc. It can be prepd by reaction of phenylchloride & K monoselenothiosulfate (K₂SeSO₃) in dil alc & in presence of Na carbonate (Refs 1 & 2), and from KSeCN & 2,4-(O₂N)₂C₆H₄Cl in alc (Ref 3)


Mononitrodiphenylsulfide, C₆H₅.S.C₆H₄NO₂; mw 231.27, N 6.06%. Three isomers are known:

2-Nitro-, yel-om prisms (from alc + eth), mp 79-82°

3-Nitro-, yel-om prisms (from petr eth), mp 55°

Other props & methods of prep are found in Beil

Ref: Beil 6, 337, 339, (154, 159), [305,311] & [1058, 1066, 1070]
Dinitrodiphenylsulfide,
\[ \text{O}_2\text{N.C}_8\text{H}_4\text{.S.C}_6\text{H}_4\text{.NO}_3; \text{ mw } 276.27, \text{ N } 10.14\% \]
Five isomers are known:
2,4'-Dinitrodiphenylsulfide, yel ndls (from ethacet + alc), mp 122-23°C, bp - sublimes with partial decomp (Ref 1)
2,4'-Dinitrodiphenylsulfide, crys (from glc acet ac), mp 163-64°C & 158-59°C (Ref 2)
3,4'-Dinitrodiphenylsulfide, yel crys (from alc), mp 125°C (Ref 3)
4,4'-Dinitrodiphenylsulfide, om-colored plds (from glc acet ac), mp 156-61°C (Ref 4)
2,4-Dinitrodiphenylsulfide,
\[ (\text{O}_2\text{N})_2\text{C}_8\text{H}_4\text{.S.C}_6\text{H}_4\text{H}, \text{lt-yel ndls (from acet or benz + alc), mp 119-21°C (Ref 5) \}
Other props & method of prep are found in Beil

Trinitrodiphenylsulfide, \[ \text{C}_12\text{H}_7\text{.N}_2\text{O}_5S; \text{ mw } 321.27, \text{ N } 13.08\% \]
Three isomers are known:
2,4,2'-Trinitrodiphenylsulfide,
\[ (\text{O}_2\text{N})_2\text{C}_8\text{H}_4\text{.S.C}_6\text{H}_4\text{.NO}_2; \text{ yel tablets (from alc), mp 131-33°C (Ref 1) } \]
2,4,4'-Trinitrodiphenylsulfide,
\[ (\text{O}_2\text{N})_2\text{C}_8\text{H}_4\text{.S.C}_6\text{H}_4\text{.NO}_2; \text{ yel tablets (from acet ac), mp 158-60°C (Ref 1) } \]
2,4,6-Trinitrodiphenylsulfide,
\[ (\text{O}_2\text{N})_2\text{C}_8\text{H}_4\text{.S.C}_6\text{H}_4\text{, crys, mp 120°C (dec) (Refs 2 & 3)} \]
Other props & method of prep are found in Refs
Refs: 1) Beil 6, 343 & [1095] 2) Beil 6, [1083] 3) A. Mangini & R. Passeireni, RAL (8) 7, 324, 327(1949)

2,4,2',4'-Tetranitrodiphenylsulfide or Bis(2,4-Dinitrophenyl)-sulfide,
\[ (\text{O}_2\text{N})_2\text{C}_8\text{H}_4\text{.S.C}_6\text{H}_4\text{.NO}_2; \text{ mw } 366.27, \text{ N } 15.29\% \text{, crys (from glc acet ac), mp 196-97°C, bp - explodes at 280°C, sol in nitrobenz & pyridine. It can be prep by reacting 4-chloro-1,3-dinitrobenzene with Na}_2\text{S or Na}_2\text{S}_2\text{O}_3 in aq alc, and by other methods (Refs 1, 2 & 3) \]

2,4,6,2',4'-Pentanitrodiphenylsulfide,
\[ (\text{O}_2\text{N})_3\text{C}_8\text{H}_4\text{.S.C}_6\text{H}_4\text{.NO}_2; \text{ mw } 411.27, \text{ N } 17.03\% \text{, large, thick yel crys (from glc acet ac), or small almost colorless pltls (from acet + w), mp 217°C, bp - explodes; sol in glc acet ec, benz & acet; almost insol in petr eth. It can be prepd by reacting an alcoholic soln of K}_2\text{S, 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-trinitrophonyl)-sulfide or Dipicryl-sulfide,
\[ (\text{O}_2\text{N})_2\text{C}_8\text{H}_4\text{.S.C}_6\text{H}_4\text{.NO}_2; \text{ mw } 456.27, \text{ N } 18.42\% \text{, OB to CO}_2 \& SO}_2 \text{ minus 56.1%; yel solid, mp 234°C (corr); resolidifies at } 213°C \text{ and remelts at } 234°C \text{; bp detonates at } 320°C; \text{ sp gr } 1.70 \text{ at } 30000kg/sq cm; \text{ insol in w; sl sol in CS}_2, \text{ petr eth, alc, eth or chlf; sol in acet or glc AcOH. It was first prep and patented in Germany in 1912 (Refs 1 & 2) \text{ and proposed for use in mixts with other exps for filling mines, torpedoes and bombs. Then it was patented in GB Britain (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 WWI in Ger drop bombs. Davis stated the fact that exp 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 prep 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

\[ 2C_6H_5(NO_2)_3Cl + Na_2S_2O_4 + H_2O \rightarrow \]
\[ 2NaCl + H_2SO_4 + [C_6H_4(NO_2)_3]_2S \]  
(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 Picryl chloride with 1 mol K sulfide in 1% alc soln

\[ 2C_6H_4(NO_2)_3Cl + K_2S \rightarrow 2KCl + [C_6H_4(NO_2)_3]S \]  
(Ref 1)
c) Gius (Ref 9) proposed to warm Picryl chloride with thiourea in 90–92% alcohol:

\[ 2C_6H_4(NO_2)_3Cl + CS(NH_2)_2 + C_2H_5OH \rightarrow \]
\[ 2HCl + [C_6H_4(NO_2)_3]S + HN: C(OC_2H_5)_2.HN_2 \]  
(See also Ref 14)

Following are properties of HNDPhS:

**Eutectic with 86.5% TNT** – mp 78°C

**Explosion Point** – 319°C if temp is raised at the rate of 20°C/min and 30°C at 5°C/min

**Heat of Combust** – 1425 cal/mol at Cu (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 2 kg fall for 0/6 shot is 36–39 cm vs 49–51 cm for Tetryl (Ref 7)

**Power by Trauzl Test** – 107% PA (Ref 8);

ADL Rept I gives 101% PA & 110% TNT

**Thermal Stability at 95°C** – no decompo in 30 eight hour days of heating (Ref 7)

**Toxicity**: The substance is non toxic, but its vapors cause intense respiration (Ref 15)

**Uses**: Besides its uses in mixts with TNT and other nitrohydrocarbons for filling bombs, shells, mines, torpedoes, 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, BricP 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) G. F. van Duin & B. C. R. van Lennep, Rec 39, 145 (1920)  
8) B. Flürschein, JSCI 40T, 97 (1921)  
9) M. Gius, GiumChinIndustrAppl 6, 166–67 (1924) & CA 18, 3273 (1924)
10) Stettbacher (1933), 394  
11) Pepin Leballeur (1935), 275 (Sulfure de picryl)
12) Thorpe 4 (1940), 484 (Use of HNDPhA for filling aerial bombs)  
13) Bebrie (1943), 83–84  
14) Clift & Fedoroff, 2 (1943), pH 5
15) Davis (1943), 187  
16) A. H. Blatt, OSRD Rept 1944 (1944)  
17) Pérez Ara (1945), 536 (Sulfuro de picrilo)  
18) Vivas, Feigenspan & Ladreda 2 (1946), 179–81 (Sulfuro-hexanitrodifenilico)  
19) ADL PureExploCompsds Pt 1 (1947), p 109  
20) ADL PureExploCompsds 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) Urbanskii 1 (1964), p 553  

Diphenylsulfocarbamide. See Diphenylthiocarbamide

Diphenylsulfocarbazono. See Diphenylthiocarbazono

Diphenylsulfocarbodiabazono. See Diphenylthiocarbodiabazono

Diphenylsulfone and Derivatives

Diphenylsulfone or Sulfobenzidine, (C_6H_5)_2SO_2, mw 218.26; ndls, sp gr 1.248 at 25/4°C, mp 128–29°C, bp 379°C; sol in hot w; sol in benz in hot alc. It can be prep'd from reaction of benzenesulfonyl chloride & benzene in the presence of FeCl_3 at 80–110°C, and by several other methods (Ref)

**Refs**: Beil 6, 300, (144), [290] & [992]
4,4′-Diozidodiphenylsulfone or Bis(4-Azido phenyl)-sulfone, \( \text{N}_2\text{C}_6\text{H}_4\text{SO}_2\text{C}_6\text{H}_4\text{N}_2 \); mw 300.30, N 28.05%, 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. Gunsit, JACS 70, 1268 (1948)

Mononitrodiphenylsulfone or Phenyl-(nitrophenyl)-sulfone, \( \text{O}_2\text{N}_2\text{C}_6\text{H}_4\text{SO}_2\text{C}_6\text{H}_4\text{H}_2 \); 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 & glc ac; acet; insol in w (Ref 1)
3-Nitrodiphenylsulfone, ndls (from alc), mp 81–85° (Ref 2)
4-Nitrodiphenylsulfone, plts (from alc or glc ac; acet), mp 140–43° (Ref 3)

Other props & methods of prep are found in Beil.

Dinitrodiphenylsulfone or Bis(nitrophenyl)-sulfone, \( \text{O}_2\text{N}_2\text{C}_6\text{H}_4\text{SO}_2\text{C}_6\text{H}_4\text{H}_2 \); mw 308.27 N 9.30%. Five isomers are known:
2,2′-Dinitrodiphenylsulfone, crysts (from glc ac; acet), mp 163° (Ref 1)
3,3′-Dinitrodiphenylsulfone, tablets, mp 198–203°; sol in hot glc ac; acet; si sol in alc & eth (Ref 2)
3,4′-Dinitrodiphenylsulfone, crysts (from glc ac; acet), mp 181° (Ref 3)
4,4′-Dinitrodiphenylsulfone, crysts (from glc ac; acet), mp 282–290° (dec); insol in w; v sl sol in alc; si sol in hot 80% ac (Ref 4)
2,4-Dinitrodiphenylsulfone, \( \text{O}_2\text{N}_2\text{C}_6\text{H}_4\text{SO}_2\text{C}_6\text{H}_4\); ndls (from alc or glc ac; acet), mp 161°; insol in eth; sl sol in alc; mod sol in hot glc ac & benz (Ref 5)

Other props & methods of prep are found in Beil.

Trinitrodiphenylsulfone or Nitrophenyl-(dinitrophenyl)-sulfone, \( \text{(O}_2\text{N}_2\text{C}_6\text{H}_4\text{SO}_2\text{C}_6\text{H}_4\text{N}_2 \); mw 355.27, N 11.89%. Two isomers are known:
2,4,3′-Trinitrodiphenylsulfone, crysts (from glc ac; acet), mp 196–97°; was prepd by heating 4-chloro-1,3-dinitrobenezene 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°; prep by heating 3-nitrophenyl-(4-nitrophenyl)-sulfone with a mixt of fuming nitric acid & sulfuric acid at 150° (Refs 2 & 4)

2,4,2′,4′-Tetranitrodiphenylsulfone or Bis(2,4-Dinitrophenyl)-sulfone, \( \text{(O}_2\text{N}_2\text{C}_6\text{H}_4\text{SO}_2\text{C}_6\text{H}_4\text{H}_2 \); mw 398.27, N 14.40%; crysts (from aq ac; acet), mp 174–75°; sol in ac; mod sol in glc ac; acet; insol in w; dec in alkalies. Can be prepd from bis(2,4-dinitrophenyl)-sulfide by heating for 6 hrs with an excess of \( \text{Cr}_2\text{O}_7 \) in glc ac or by heating with \( \text{K}_2\text{Cr}_2\text{O}_7 \) 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 \( \text{K}_2\text{Cr}_2\text{O}_7 \) in 57% sulfuric acid at its boiling temp (Refs)
2,4,2',4',6'-Pentanitrodiphenyl-sulfone (PNDSH0) or Thionol,
\[(O_2N_2)C_6H_4SO_2C_6H_4(NO_2)_3\]; mw 448.27,
N 17.21%, OB to CO_2 & SO_2 minus 45.9%.
It yel crystal, 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 prep 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 Trauzi Test - 107% PA (Ref 10).
Thermal Stability - no decomp took place
in thirty 8hr days at 95° (Ref 4); did not decomp
after being stored for a long time at
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 airbal bomb (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) Ciftr & Fedoroff
2(1943), p H5 9) Davis(1943), 187-88
10) A.H. Blatt, OSRD 1041(1944) 11) Pérez
Ara (1945), 537 12) Vivas, Feigenspan &
Ladreda 2(1946), 181 (Hexanitrodiphenyl-
sulfone) 13) E. von Herzs St & E. von Herzs
Jr, GerP 859868(1952) & CA 52, 14171(1958).
14) K. Oko, BiulWojewodzkiej AkadTechnicznej
im J.Dabrowskiego 7, No 38, 6 (1957) & CA
53, 4181(1959) 15) J.E. Hughes & D.N.
Thatcher, USP 2952708(1960) & CA 55, 3062
(1961) (Dipicyrsul fate expls) 16) DuPont,
(Monoclinic Dipicyrsulfone vs orthorhombic
form) 17) DuPont, GerP 1078920(1961) &
CA 56, 624(1962) (Dipicyrsulfone, prep &
use in elec fuzes) 18) Urbanski I(1964),
554.

Diphenylsulfourea. See Diphenylthiourea in
this Vol.

Diphenyltetraacetylene or 1,8-Diphenyl-
1,3,5,7-octatetrayne.
C_6H_5-C-6C-6C-6C-6C-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 decomd explosively
with much soot. It shows no color
reaction with sulfuric acid; is more sol than
tolol & (C_6H_5-C)_2 in polar solvs such as
MeOH, alc & acetone. It was prepd by oxi-
dative coupling of cuprous acetylde,
C_6H_5-C-6C-6C-Cu, with CuCl_2 (Ref 5).
Refs: 1) Beil, not found 2) M. Nakagawa,
Kagaku no Ryūsiki (JJapanChem) 4, 564;
Diphényltetrazole and Derivatives

1,5-Diphenyl-1H(or a)-tetrazole or 1,5-Diphenyl-1,2,3,4-tetrazole,
(C₆H₅)₂C——N(C₆H₅)

N — N

mw 222.24, N 25.21%; ndls (from alc), mp 146°. It was prep'd by heating benzophenone chloride with Na azide in diisooamyl ether and in a CO₂ stream at 140–60° (Refs 1 & 2)
Refs: 1) Beil. 26, 362 & (112) 2) G. Schröder, Ber 42, 2341–43, 3359–60 (1909)

2,5-Diphenyl-1,2,3,4-tetrazole,
(C₆H₅)₂C——N

N — N(C₆H₅)₂N

ndls (from alc or petr eth), mp 101.5–102°; sol in common org solvs; insol in w. It was prep'd by heating benzaldehyde-phenylhydrazine 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. Merzbach, Ber 40, 2402–04 (1907) & CA 1, 2610 (1907)

Mononitrodiphenyltetrazole, C₁₅H₁₁N₃O₂;
mw 267.24, N 26.21%.
1-Pheny1-5-(4-nitrophenyl)-tetrazole,
O₂N·C₆H₄·C——N·C₆H₅

N — N

1-yel plts, mp 177–78°. It was prep'd by heating phenyl-(4-nitrophenyl)-dichloromethane with Ag azide in diisooamyl ether (Refs 1 & 4)
Refs: 1) Beil 26, 362 & (112) 2) G. Schröder, Ber 42, 2341–43, 3359–60 (1909)

1-yel prisms (from ethyl acetate + alc), mp 149°; insol in w & petr eth; sl sol in alc; readily sol in ethyl acet. It was prep'd by heating 1-(4-nitrophenyl)-4-benzoyl-1-tetrazene with aq NaOH (Refs 2 & 5)
2-Pheny1-5-(4-nitrophenyl)-tetrazole,
O₂N·C₆H₄·C——N

N — N(C₆H₅)₂N

crysts (from acet), mp 199–200°; sol in clhf, acet, glc acet ac & benz; sl sol in alc, eth & petr eth. It was prep'd by carefully heating
N-phenyl-N'-guanyl-C-(4-nitrophenyl)-tomazan with concd nitric acid at 70°C (Refs 1 & 3) 
Refs: 1) Beil 26, 365 2) Beil 26, (112) 
3) E. Wedekind, Ber 31, 475, 478 (1898) 
4) G. Schmoeber, Ber 42, 3360 (1909) 5) O. 
Dimroth & G. de Mannolin, Ber 43, 2914 
(1910) & CA 5, 494(1911)

Dinitrodiphenyltetrazoles, $C_{13}H_{3}N_{3}O_{4}$; 
$1,5$-Bis(nitrophenyl)-1,2,3,4-Tetrazole, 
mw 312.24, N 26.92%. 
O$_2$N$\text{C}_6\text{H}_4\text{C} \equiv \text{N}$N$\text{C}_6\text{H}_4\text{NO}_2
N=NN

The 1,5-Bis (o-), mp 209°, (m-), mp 244°, and 
(p-), mp 262° derivs were prepd by Braun & 
Rudolph (Ref 2) by nitratd the parent: compd 
with a 1/1 mixt of fuming nitric acid + sul-
furic acid on a water bath. Horwitz & Gra-
kaukasus (Ref 4) prep the 1,5-Bis(p-dinitro) 
deriv by coupling diazotd p-nitroaniline 
with 1-acetyl-2-(4'-bromobenzoyl) hydrazine, 
separating the tetrzole following cyclization 
1-(m-Nitrophenyl)-5-(p-nitrophenyl)-tetrazole, 
crysts, mp 170° (Ref 2) 
5-(2,4-Dinitrophenyl) -1-(phenyl) -tetrazole, 
(O$_3$N)$_2$C$_6$H$_5\text{C} \equiv \text{N}$C$_6$H$_5
N=NN

crysts, mp 164° (Refs 2 & 3) 
1-(2,4-Dinitrophenyl)-5-(phenyl) -tetrazole, 
C$_6$H$_5\text{C} \equiv \text{N}$C$_6$H$_4$(NO$_2$)$_2
N=NN

crysts, mp 183°; was prepd by stirring a 
suspension of C$_6$H$_5$Cich:C$_6$H$_5$NO$_2$2,4 in 
acetic acid with Na azide in alc for 3 hrs at RT 
(Refs 3 & 4) 
Refs: 1) Beil, not found 2) J.V. Braun & 
W. Rudolph, Ber 74B, 267, 271 (1914) & CA 
35, 2892(1914) 3) F.L. Scott et al, JACS 
75, 5310(1953) & CA 49, 169 (1955) 4) J.P. 
Horwitz & V.A. Graukaukas, JOrgChem 19, 
199-200(1954) & CA 49, 3714(1955) 5) G. 
Bianchetti et al, Gazz 94(3-4), 210(1964) 
& CA 61, 5633(1964)

NOTE: No higher nitro derivs of Diphenyl-
tetrazole were found in the literature thru 1966

N,N'-Di(2-phenyl-5-tetrazolyl)-urea (called 
N,N'-Bis(2-phenyl-tetrazolyl-5)-hamsoff 
or Bis(2-phenyl-tetrazolyliden-5)-hamsstoff 
in Ger)

C$_6$H$_5$N=NH-CN.CO.N=C=NH-N-C$_6$H$_5$

or

C$_6$H$_5$N=C=NH.CO.NH-CN=NN-C$_6$H$_5$

mw 348.32, N 40.19%, yel ndls (from glc 
acet ac), mp 216°(dec); insol in w; v sl sol 
in eth; sl sol in hot alc; sol in hot glc 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. Stolle & 
O. Orth, Ber 58, 2100, 2103(1923) & CA 20, 
763(1926)

p-Diphenyltetrazonium-2,1-naphthol-sulfonate, 

(HO)$_2$C$_6$H$_5$SO$_3$N--N=C$_6$H$_4$-C$_6$H$_4$-N=O$_3$S.C$_6$H$_4$(OH) 
mw 566.65, N 9.89%, brownish-yel powd, mp ~ 
explodes; diff sol in w & neutral solvls; sol 
in alkalies. It was obtd when benzidine, 
H$_3$N.C$_6$H$_4$-C$_6$H$_4$.NH$_2$, was diazotized and 
then treated with 2,1-naphthol-sulfonic acid, 
2,1-C$_6$H$_4$(OH)SO$_3$H, 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 Diphenyl-
 thiourea

$N, N'$-Diphenylthiocarbazone or Diphenyl-
sulfo carbazone,
C$_6$H$_5$N-N.CS.NH.NH-C$_6$H$_5$; mw 256.32, 
or 
C$_6$H$_5$N-N.C(SH)N.H-NH-C$_6$H$_5$ N 21.86% 
dk-violet ndls (from chlf + pett), 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 gm soln. It can be prepd by the action of light on a soln of 1,5-diphenyl-thiocarbonyldiazide in aq alcoholic NaOH, and by other methods (Refs 1, 2 & 3).

It forms numerous salts of which the Lead salt, Pb(C_13H_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-thioacetanethione,

\[ \text{SC} \cdot \text{N} \\]

\[ \text{N}(\text{C}_6\text{H}_5)_2 \cdot \text{N} \cdot \text{C}_6\text{H}_5; \text{pale yel crysts, exploading at } 180.5° \text{ depending on rate of heating} \] (Ref 2)


NOTE: Compare with Diphenylcarbzone in this Vol

2,3-Diphenyl-5-thiocarbodiazone (called "Diphenyltetrazolium-thiobetaine" in Ger). It is the Anhydride of 2,3-Diphenyl-5-thio-
tetrazolium Hydroxide (qv):

\[ \text{SC} \cdot \text{N} \\]

\[ \text{N}(\text{C}_6\text{H}_5)_2 \cdot \text{N} \cdot \text{C}_6\text{H}_5; \text{mw 254.31}, \text{N 22.03%; red nldls (from alc); mp } \text{exploading at } 180.5° \text{ depending on rate of heating w/o melting); sol in clhf, concd sulfuric acid, warm w & hot alc; diff sol in eth & benz; insol in alkalies. It was first prepd in 1882 by Fischer & Beshtrorn (Refs 1 & 2) by heating on a water N,N'-diphenyl-C-mercaptoformazan (listed in Beil 16, 26), C_6H_5N:NC.SH.NH.C_6H_5 or C_6H_6N:NC(SH):NH.C_6H_5, with manganodioxycarbonate, Mn(OH)_3, in the presence of alcoholic KOH. Another method is to heat N,N'-diphenyl-C-nitroformazan, C_6H_5N:NC(NO_2):NH.C_6H_5, in an ethereal

soln with isopentynitrile introduced dropwise


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 Hydroxide in Ger)

\[ \text{SC} \cdot \text{N} \\]

\[ \text{HN}(\text{C}_6\text{H}_5)_2 \cdot \text{N}(\text{C}_6\text{H}_5)_2 \cdot \text{OH} \] or

\[ \text{HSC} \cdot \text{N} \\]

\[ \text{N}(\text{C}_6\text{H}_5)_2 \cdot \text{N}(\text{C}_6\text{H}_5)_2 \cdot \text{OH} \]

Exists as its Anhydrite. See Diphenyl-
thiocarbodiazone

Diphenylthiourea and Derivatives

N,N'-Diphenylthiourea, N,N'-Diphenylsulfourea or Thiocarbanilide, 
(C_6H_5N-H)_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 gm liq. It can be prepd by heating N,N-diphenyl-N'-acetyl-thiourea with alcoholic NaOH (Ref 2)


Azido, C_11H_11N_3S, and Diazido, C_11H_10N_8S, derivs were not found in the literature thru 1966
Mononitrodiphenylthiourea or Nitrothiocarbonanilide, O₃N₃C₆H₄.NH.CS.NH.C₆H₄; mw 273.31, N 15.37%. Three isomers are known:

N-Phenyl-N'-(2-nitrophenyl) -thiourea or 2-Nitrothiocarbonanilide, yel crs (from glc 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, O₃N₃C₆H₄.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-Nitrothiocarbonanilide, ndls or yel scales, mp 155° & 160°; sol in warm alc; sl sol in benz, chf, carbon disulfide & cold alc. It can be prepd by heating 3-nitroaniline & phenyl mustard oil, C₆H₅.N:CS (Refs 2, 3 & 4)

N-Phenyl-N'-(4-nitrophenyl)-thiourea or 4-Nitrothiocarbonanilide, crs, mp 100°. No addy info found in abstract (Ref 5)


Dini trodiphenylthiourea or Dinitrothiocarbonanilide, O₃N₃C₆H₄.NH.CS.NH.C₆H₄.N(NO₂)₂; mw 318.31, N 17.60%. Two isomers are known:

N,N' -Bis(3-nitrophenyl)-thiourea or 3,3'- Dinitrothiocarbonanilide, yel ndls, mp 160°-61° & 168°. It can be prepd from reaction of 3-nitroaniline & carbon disulfide in the presence of sulfuric chloride, and by other methods (Refs 1, 3, 4, 5, 6 & 8)

N,N'-Bis(4-nitrophenyl)-thiourea or 4,4'- Dinitrothiocarbonanilide, orn-bm crs, mp 195-96°. It was prepd by heating 2 mols 4-Nitroaniline with 1 mol thiosphogene in water (Refs 2, 7 & 9)


N-Phenyl-S-picyrly-isothioura, C₆H₅.N.CS.C₆H₅(NO₂)₂ or C₆H₅.N:CNH₂S.C₆H₅(NO₂)₂; mw 363.31, N 19.28%; obtd with other products by uniting of Picryl chloride & N-phenylthiourea in cold acetone or better in glc acet ac. It was isolated as the Picrate, mp 82°, explodes at high temp (Refs 1 & 2)


N,N', Bis(3,5-dinitrophenyl)-thiourea or 3,5,3',5'-Tetranitrothiocarbonanilide, (O₃N₃C₆H₄)₂NH.CS.NH.C₆H₄(NO₂)₂; mw 408.31, N 20.58%, yel prisms (from petr eth + glc acet ac), mp 160°. It was prepd by treating 3,5-dinitroaniline with thiosphogene in water for ½ hr (Refs 1 & 2)


Pentanitro, C₁₃H₁₃N₅O₁₅S, and Hexanitro, C₁₃H₉N₅Oₑ₂S, derivs of Diphenylthiourea were not found in the literature thru 1966

Diphényltolylpropane and Derivatives
1,1-Diphenyl-2-p-tolylpropane or p-Tolyl-dibenzylmethane, CH₃.C₆H₄.CH(CH₃.C₆H₄); may be considered as the parent compd (Ref 1) of its hexanitro deriv, although not used to
prep it:
1,3-Diphenyl-2-p-tolylpropene, 2,4,6,2',4',6'-Hexanitrodiphenyltolypropene or 1,3 Bis-(2,4,6-trinitrophenyl)-2-p-tolylpropene, (O_2N)_3C_6H_5.CH_2.CH(C_6H_4.CH_3).CH_2.C_6H_5(NO_2)_3; mw 556.40, N 15.11%, cinnor-yel ndls (from glc ac et ac), turn in the light, mp 183–86°. It was prep by reaction of p-tolualdehyde, 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, [524] 3) I.A. Pastak, BullFr 39, 77 (1926) & CA 20, 3000 (1926)

NOTE: No lower nitrated derivs of Diphenyl-tolypropene were found in the literature than 1966

1,3-Diphenyltriazene is described in this Vol, p D1156-R, as Diatoamino-bentene

It was described and code named PAD-2 in ADL, SynthHE's, 4th Rept (1956), p 107

Diphenyltriazene and Derivatives

Diphenyltriazene, C_6H_5.H_2.N_3; mw 221.25, N 18.99%. Seven isomers are found in Beil: 1,5-Diphenyl-1,2,3-triazole, C_6H_5.C-N(C_6H_5)-N

HC-N-N; tablets (from alc), mp 113–14° (Ref, p 68)

2,4-Diphenyl-1,2,3-triazole, HC-N-N.C_6H_5

C_6H_5.C==N; plts or ndls (from alc), mp 56–57° (Ref, p 34)

4,5-Diphenyl-1,2,3-triazole, C_6H_5.C=N-N

C_6H_5.C==N; N-dls (from dil alc), mp 139°. It forms a Silver salt, AgC_6H_5.H_2.O_3, light-sensitive powd (Ref, p 79)

1,3-Diphenyl-1,2,4-triazole,

HC-N(C_6H_5)-N

N---C.C_6H_5; ndls (from dil alc), mp 96–97°, di stills 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 Picate, tablets (from alc), mp 148° (Ref, p 68)

1,5-Diphenyl-1,2,4-triazole,

C_6H_5.C-N(C_6H_5)-N

N---CH; 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,

HC-N-N


3,5-Diphenyl-1,2,4-triazole,

C_6H_5.C-N-N

C_6H_5.C=N-N; or

HN==C.C_6H_5

N---C.C_6H_5;

prisms or plts + water (from dil alc or benz), mp 190–92° (becoming anhyd), bp ~ dec at 280°. It forms a Silver add compound, AgC_6H_5.H_2.O_3, ndls (from dil HNO_3), mp ~ softens at 140° & dec ca 170°; and a Silver salt, AgC_6H_5.H_2.O_3, cts, mp ~ dec 246° (Ref, pp 81 & 21)

Other props & methods of prep are found in Ref

Refs: Beil 26, 68, 69, 79, 81, (21) & (34)

5-Azido-1,4-diphenyl-1,2,3-triazole,

N_3.C-N(C_6H_5)-N

C_6H_5.C==N; 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 w ith foaming; in inert solvs its soln is red and evolves N gas at temps as low as 30°. It was synthesized from 1,4-diphenyl-5-amino-tetrazole in HCl by diazotizing it with Na nitrite in water at 0° (Ref 2)
Mononitrophenyl-phenyl-triazole, $C_6H_5NO_2$, mw 266.25, N 21.04%. Several isomers are described in the literature:

1-(4-nitrophenyl)-4-phenyl-1,2,3-triazole,

$$\text{HC} = \text{N}(\text{C}_6\text{H}_4\text{NO}_2) - \text{p} - \text{N}$$

$C_6H_5\text{C} - \text{N}: \text{yel plts (from DMF + alc), mp 252-53°; prepd from 4-nitrophenylazide & phenylacetylene in toluene by refluxing for 24 hrs (Ref 9)}$

1-(4-nitrophenyl)-5-phenyl-1,2,3-triazole,

$$\text{HC} = \text{N}(\text{C}_6\text{H}_4\text{NO}_2) - \text{p} - \text{N}$$

$C_6H_5\text{C} - \text{N}: \text{It-yel plts (from benz + petr eth), mp 163-164.5° (Ref 9) & 159° (Ref 5); was prep by refluxing 4-nitrophenylazide & benzylethylacetate, cooling & decarboxylating the soln with acetonitrile (Ref 9)}$

1-(4-nitrophenyl)-3-phenyl-1,2,4-triazole,

$$\text{HC} = \text{N}(\text{C}_6\text{H}_4\text{NO}_2) - \text{p} - \text{N}$$

$N - \text{C}_6\text{H}_5\text{C}: \text{crysts (from benz + petr eth), mp 202-03°; prepd by treating N-(4-nitrophenyl) -C, N'-diphenylformazan in DMF with 2N NaOH & aq CH}_3\text{O}, heating the soln, and treating again with 2N NaOH, heating, diluting with water, and pptg repeatedly with DMF (Ref 7)}$

3-(4-nitrophenyl)-5-phenyl-1,2,4-triazole,

$$\text{HC} = \text{N}(\text{C}_6\text{H}_4\text{NO}_2) - \text{p} - \text{N}$$

$N - \text{C}_6\text{H}_5\text{C}: \text{wh ndls (from methanol or acet & then petr eth), mp 240°. It was obtd when phenyl cyanide & p-nitrobenzhydrazide benzenesulfonate were heated at 200° for 3 hrs, cooled & the crystalline melt extracted with cold NaOH soln & the extract acidified with concd HCl (Ref 2) and by other methods (Refs 3, 4, 6 & 8)}$

Trinitro-2,4-diphenyl-2,1,3-triazole,

$$\text{HC} = \text{N}(\text{C}_6\text{H}_4\text{NO}_2) - \text{p} - \text{N}$$

$O_2\text{N.C}_6\text{H}_4\text{C} = \text{N}: \text{mw 356.25, N 23.59%}, \text{crysts (from acet ac), mp 238-39°. It was obtd on long standing of the mother liquor resulting from refluxing 2,4-diphenyl-2,1,3-triazole & nitric acid & pptg the trinitro deriv by pouring into wwater (Ref 2). No expl props are reported (Ref 2)}$

Tetranitro-2,4-diphenyl-2,1,3-triazole or 2,4-Bis(dinitrophenyl)-2,1,3-triazole,
\[
\text{HC} = \text{N} - \text{N}[\text{C}_6\text{H}_3(\text{NO}_2)_2] \\
\left[(\text{O}_2\text{N})_2\text{H}_2\text{C}_4\right] \text{C} = \text{N}
\]

mw 401.25, C 24.44%, straw colored crystals (from acetic acid), mp 178-79°C. It can be prepared 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, C₁₄H₁₃N₄O₁₀, and Hexa, C₁₅H₁₉N₄O₁₂, nitro derivs of Diphenyltetrazole were not found in the literature thru 1966.

N,N'-Diphenyltrimethylene diamine. See 1,3-Bis(anillino)-propane in Vol 2, p B132-R

asym-Diphenylurea or N,N-Diphenylurea. See Acandite 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 Dipicylurea given on p B156-L there was not included the information on explosive properties given in A.H. Blatt, OSRD Rep. 2014 (1944) and ADL Pure-Expl Compds, Pt 1 (1947), because these, originally conf repts, were not reclassified 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; decompy by moisture

Stability, Thermal – good

Diphenylethane and Derivatives

Diphenylethane or N,N-Diphenyl-ethyl-carbamate, (C₆H₅)₂N·CO₂C₂H₅; 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 comb as 7697 cal/g at C₉ and heat of formation 287 cal/g at C₉. Tavemier (Ref 4) gave heat of formation at C₉ as 245 cal/g and 267 at C₉.

Diphenylethane was prep 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 WW II as a stabilizer of NGU (Nitrogen guanidine) contg propellants, known as Gadulpulver (Ref 5, p Ger 37-L & Ger 81).


NOTE: Azido, C₁₅H₁₄N₄O₂, and Diazido, C₁₅H₁₈N₄O₂, derivs of Diphenylethane were not found in Beil.

Mononitrodiphenylethane, C₁₅H₁₄N₂O₂, mw 286.28, N 9.79%. Only one isomer is found in Beil.

4-Nitrodiphenylethane,

O₂N·C₆H₅·N(C₆H₄)·CO₂·C₂H₅; prisms (from alc), mp 68°; readily sol in eth, alc, chll, benz & acet; sol in petr eth. It was prep by treating diphenelethane with cold nitric acid (d 1.42) or with 3 mols fuming nitric acid in chll (Refs 1 & 2).


Dinitrodiphenylethane, C₁₅H₁₈N₃O₆, mw 331.28, N 12.69%. Two isomers are found in Beil:

2,4'-Dinitrodiphenylethane,

(O₂N·C₆H₅)₂N·CO₂·C₆H₅, 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 N₂O₄ in CCl₄ (Refs 1 & 2)

4,4'-Dinitrodiphenylurethane, yellow ndls (from alc), mp 153–34⁰C; readily sol in benz, acet, eth & chl; sl sol in alc; almost insol in petr eth (Refs 1 & 2). Its prep is given above


Trinitrodiphenylurethane, C₁₅H₁₂N₄O₅; mw 376.28, N 14.89%. Not found in the literature thru 1966

2,4,2',4'-Tetranitrodiphenylurethane or Bis(2,4-dinitrophenyl)-ethyl-carbonate, [(O₂N)₂C₆H₄]₂N.CO₂C₂H₅; mw 421.28; N 16.63%, yellow prisms (from chl & alc), mp 184–85⁰C; sol in chl, benz & alc; sl sol in petr eth. It was prep by nitrating diphenylurethane or 4-Nitrodiphenylurethane with mixed nitric-sulfuric acids (Refs 1 & 2). No expl props reported


**NOTE:** Pentanitro, C₁₅H₁₀N₈O₁₂, and Hexanitro, C₁₅H₁₂N₄O₄, derivs of Diphenylurethane were not found in the literature thru 1966

**Diphenylamine; Xylenamine 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

**Diphenylacetic Acid Peroxide or Dipthaloyl Peroxide.** See Bis(phthalic acid)-peroxide in Vol 2, p B156-L

**Dipicolinic Acid and Derivatives**

Dipicolinic Acid or Pyridine-2,6-dicarboxylic Acid, HOOC-C- N = C-COOH

\[
\text{HC} = \text{CH} = \text{CH} \quad ; \text{mw} 167.12
\]

N 8.38%, prisms + water or hair-shaped ndls + 1½ water, mp 226–37⁰C(dec); sl sol in w, alc, eth & glacial acet ac. It was prep 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 Dipicolinoyl diazide, C₅H₅N[CO(N₃)₂]₉; mw 217.15**

N 45.16%, prisms (from ether), mp 110–11⁰C (dec, followed by expls); very irritating to mucous membranes. It was prep by action of Na nitrite & cold dil HCl on dipicolinic acid dihydrazide, NC₅H₅(CO.NH.NH₂)₂ (Refs 2 & 3)


**Di(β-picolino)-diazido-copper Complex, [(N₃)₂Cu(C₆H₄N)₂]; mw 270.29, N 41.46%;**

solid; mp – exploded violently when dropped on a metallic block preheated to 207–15⁰C; it burned in flame with crepitation and exploded by impact of 1 kg weight dropped from 80cm height. It was prep by Citrulis & Straumanis (Ref 2) by addg NaN₃ to a dipicolinic acid soln of the Cu⁺⁺ salt (Ref 2)

Refs: 1) Beil, not found 2) A. Citrulis & M. Straumanis, JPakChem 162, 311(1943) & CA 38, 1969(1944)

**Dipicrylonil.** Same as 2,4,6,2',4',6'-Hexanitrodiphenylamine described under Diphenylamine and Derivatives in this volume
Dipicrylamino-anthraquinone. See Bis-(trinitroanilino)-anthraquinone under Bis-(anilino)-anthraquinone in Vol 2, p B131-L

Dipicrylaminooethyl Nitrate. See under Diphenylnaminoethyl Nitrate in this Vol

Dipicrylarginine. See under Diphényl(aminomethyl-4-guanidino-valeric Acid) and Derivatives in this Vol

Dipicrylcaboxamide-acetylene. See under Di(N-phenylcarboxamide)-acetylene and Derivatives

Dipicryldianthranilide. See under Diphényldianthranilide and Derivatives in this Vol

Dipicryldisulfide. See under Diphényldisulfide 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 Diphénylethanol

Dipicrylether. See under Diphénylethers

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 US Bitetrayl or Ditetryl and in Gr Brit 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 Diphénylglycerylether and Derivatives

Dipicrylguanidine. See under Bis(phenyl)-guanidine in Vol 2, p B154-L

Dipicrylmethane. See under Diphénylmethane and Derivatives

Dipicrylmethylamine. See under Diphénylmethylamine and Derivatives

Dipicrylmethylenedihydroxyphenylpropane. See under Diphénylmethylenedihydroxyphenylpropane and Derivatives

Dipicryloxaamide. See under Diphényloxaamide and Derivatives

Dipicrylphenylenediamine and its S-Nitro Derivative. See under Diphénylphenylenediamine and Derivatives

Dipicrylpiperazine. See under Diphénylpiperazine and Derivatives

Dipicrylpropylenediamine (Code named Methylene Ditetrayl). See under Bis(anilino)-propane in Vol 2, p B133-L

Dipicrylselenide. See under Diphénylselenide and Derivatives

Dipicrylsulfide. See under Diphénylsulfide and Derivatives

Dipicrylsulfone. See under Diphénylsulfone and Derivatives
Di(picrylthiol)-ethylbenzene. See 2,4-Bis-(2',4',6'-trinitrophenylethio)-1-ethylbenzene in Vol 2, p B155-R

Di(picrylthiol)-xylene. See Bis(2,4,6-trinitrophenylethio)-dimethylbenzene in Vol 2, p B155-L

Dipicrylitolylpropane. See under Diphenylitolylpropane and Derivatives

_sym-Dipicyrlyurea or Hexanitrocyanuric acid.
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_2C_5.C_5H_9NH; mw 168.28, N 16.65%. The 2,2', 2,3', 3,2', 3,3', 3,4', & 4,4'-derivs are found in the literature (Ref 1)

_Dipiperidino-diazido-copper, [(N_3)_2Cu(C_5H_11N)_2], mw 317.88, N 33.25; solid, mp 109°, decom 160–65°. It was prepd by addg NaN_3 to a dipiperidine soln of the Cu^++ salt (Ref 4)

_Dipiperidino-tetrazido-copper, (C_5H_12N)_2Cu(N_3)_4, mw 403.94, N 48.54%; solid; mp 125°, bp – expl at 200–205° when thrown on the pre-heated metallic block; burns in flame with a crackling noise; very insensitive to impact (100cm with 1 kg wt). It can be prep'd 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'-Dinitrosodipiperidine, crystals (from alc), mp 159° (Ref 3, p37)
1,1'-Dinitroso-2,2'-dipiperidine, crystals (from eth + a little alc), mp 88° (Ref 3, pp 38 & 32)
1,1'-Dinitroso-3,3'-dipiperidine, crystals, mp 102° (Ref 3, p 32)
1,1'-Dinitroso-3,4'-dipiperidine, crystals, mp 149° (Ref 3, p 32)
1,1'-Dinitroso-4,4'-dipiperidine, yel crystals (from alc), mp 141–43° & 150° (Ref 3, pp 38 & 33)
Ref's: 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. Cinulis & M. Straumanis, JPraktChem 162, 312(1943); Ber 76B, 825(1943) & CA 38, 1969(Diazido-Cu) & 1971 (Tetrazido-Cu) (1944)

NOTE: No nitro derivs found in the literature thru 1966

Diposal or Salysal. See Disalicylic Acid in this Vol

Diplumbic Dinitrosoresorcinol. See Basic Lead Salt of 4,6-Dinitro-1,3-dihydroxybenzene or Lead 4,6-Dinitrosoresorcinolate, p D1275-R

Dipole Moment (Momente 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

<table>
<thead>
<tr>
<th>Inorganic Compds</th>
<th>State or Solvent</th>
<th>Expt'l Dipole Moment Debyes</th>
<th>Ref</th>
<th>Theoretical Dipole Moment Debyes</th>
<th>Ref</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen Azide *</td>
<td>gas</td>
<td>0.84</td>
<td>5, 8</td>
<td>1.08**</td>
<td>11</td>
</tr>
<tr>
<td>Hydrogen Fulminate</td>
<td>gas</td>
<td>3.06</td>
<td>9</td>
<td>2.58**</td>
<td>11</td>
</tr>
</tbody>
</table>

Nitroaliphatic Compd

<p>| | | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitromethane</td>
<td>gas</td>
<td>3.54</td>
<td>10</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>benzene</td>
<td>3.13</td>
<td>10</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Nitroaromatic Compds

<p>| | | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrobenzene</td>
<td>gas</td>
<td>4.2</td>
<td>10</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>benzene</td>
<td>4.0</td>
<td>10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>o-Dinitrobenzene</td>
<td>—</td>
<td>6.0</td>
<td>10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>m-Dinitrobenzene</td>
<td>—</td>
<td>3.88</td>
<td>10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>p-Dinitrobenzene</td>
<td>—</td>
<td>0.00</td>
<td>10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,3-Dinitrotoluene</td>
<td>benzene</td>
<td>5.81</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,4-Dinitrotoluene</td>
<td>benzene</td>
<td>4.33</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,5-Dinitrotoluene</td>
<td>benzene</td>
<td>0.58</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,4,6-Trinitrotoluene</td>
<td>benzene</td>
<td>1.16</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2,4,6-Trinitroanisole</td>
<td>benzene</td>
<td>2.00</td>
<td>7</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Nitramine Compds

<p>| | | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Dimethylnitramine</td>
<td>dioxane</td>
<td>4.61</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>RDX (Cyclotrimethylene Trinitramine)</td>
<td>dioxane</td>
<td>5.78</td>
<td>7</td>
<td>5.48***</td>
<td>6</td>
</tr>
</tbody>
</table>

Nitrate Compds

<p>| | | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>PETN (Pentaerythritol Tetranitrate)</td>
<td>dioxane</td>
<td>2.48</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NG (Nitroglycerin or Glycerol Trinitrate)</td>
<td>benzene</td>
<td>3.38</td>
<td>7</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* 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


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 Hydroazobenzene-Azobenzene.
See Azobenzene-Dipotassium Hydroazobenzene in Vol 1, p A647-L

Dipotassium Nitroacetate (DKNA). See under Acetic Acid in Vol 1, p A27-R

Dipropylamine or Bis(hydroxypropyl)-amine.
See Di(propylol) -amine

Di(iso-propanol)-amine. See Di(iso-propyl)-amine

Di(propylol)-ethylenediamine. See Di(propylol) -ethylenediamine

Dipropargylbutyral-nitroform Product,
CH₃.CH₂.CH₂.CH.O.CH₂.C.CH

\[ \text{C(NO}_2\text{)}_3 \]

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)

Dipropionylperoxide or Propionic Peroxide,
CH₃.CH₂.CO–O–O–CO.CH₂.CH₃; mw 146.14, O 43.79%; fp p remained liq at minus 20 °
bp 15°C; blows up on rapid heating above 80 °C; sol in w (with slow hydrolysis to propionic acid); sol in org solvents. It can be prepd by the action of Ba peroxide on an ethereal soln of propionic aldehyde at ca 0 °C. Another method is electrochemical oxidation on a platinum anode of a soln of propionic acid or its salts.

Di-n-propyladipate, H₃C₄O₂C(CH₂)₄.CO₂C₃H₇; mw 250.30, colorless liq, sp gr 0.979 at 20/4°C; fp p minus 20.3 °C, bp 143–145 °C at 10 mm Hg; insol in w; sol in alc & 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 propiants Unit Nos 386, 390, 391 & 550 described in conf Manual (Ref 2)
Dipropylamine and Derivatives

Di-n-propylamine, C₆H₁₃.NH.C₆H₇, 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 prep'd 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 comps, some of which are unstable when heated:

Azide, C₆H₁₅.N.HN₃, leaflets (thru sublimation), mp 101°

Hydrogen iodide, C₆H₁₃N.HI, ndls (from alc), mp 230°-35°(dec)

Perchlorate, C₆H₁₃N.HClO₄, crysts, explodes at 308°

Tetraiodoethane, C₆H₁₃N₂I₂.C.C.I₂, ndls or platls (from chl), sinters at 116°

Di(n-propylamino)-diazido-copper,

\[ \text{[N₃Cu(C₆H₁₃NH₂)]} \], 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 prep'd by dissolving Cu(N₃)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. Citulis & M. Straumanis, JAnorgChem 251, 346 (1943) & CA 37, 6574(1943)

Azido, C₆H₁₄N₃, and Diazido, C₆H₁₃N₇, derivs of Dipropylamine, were not found in the literature thru 1966.

N-Nitrodipropylamine or Dipropyl nitramine, H₅C₂.CH₂.N(NO₂).CH₂.C₂H₅; mw 146.19, N 19.17%; oil, fr p 1.0-1.6°, bp 103°-04° at 10mm press, nD₅ 1.4559 at 20°; stable in 70% sulfuric acid at 25°. It was prep'd by nitrating di-n-propylamine with 99% nitric acid in the presence of acetyl chloride & acetic anhydride in glacial ac (Refs 1 & 2)

Refs: 1) Beil 4, 1264 2) W.R. Kingdom & G.F. Wright, JACS 72, 1031 (1950)

Dinitro, C₆H₁₃N₂O₄, and Trinitro, C₆H₁₂N₄O₆, derivs of Di-n-propylamine were not found in the literature thru 1966

2,2',2'-Tetranitro-dipropylamine or Bis(2,2-dinitropropyl)-amine,

\[ \text{CH₂.C(NO₂)₂.CH₃} \]

\[ \text{CH₂.C(NO₂)₂.CH₃} \]

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)


Pentanitrodipropylamine, C₆H₁₄N₆O₁₀; mw 326.19, N 25.77%. Two isomers are known:

N,2,2,2',2'-Pentanitrodiphenylamine or Bis(2,2-dinitropropyl)-nitramine,

\[ \text{CH₂.C(NO₂)₂.CH₃} \]

\[ \text{CH₂.C(NO₂)₂.CH₃} \]

crysists (from MeOH), mp 187°-89°. It was prep'd by dropwise addn of 100% nitric acid to a soln of the above tetranitro deriv in ethyl acetate (Ref 3 & 6). The IR absorption bands were dept (Ref 6)

2',2',3,3,3-Pentanitrodipropylamine,

\[ \text{CH₂.C(NO₂)₂.CH₃} \]

\[ \text{CH₂.C(NO₂)₂.CH₃} \]

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, \((O_2N)_2CCH_2CH_2NH_2\), and 2,2-dinitropropanol in water, and cooling the mixt in an ice bath (Refs 2, 4, 5 & 6). These cmpds are used as exp1 ingredients, or as O donors in propellant fuels (See Refs)


**N,2',3,3,3-Hexanitrodipropylamine or N-(3,3,3-Trinitropropyl)-N-(2,2-Dinitropropyl)nitromine**

\[ O_2N\cdotN\cdotCH_2\cdotC(NO_2)\cdotCH_2\cdotC(NO_2)\cdot \]

mw 371.19, N 26.42%; crystallizes (from CCl₄), mp 59-109.5°(dec). It was prepared by nitration of the Pentaamidodinitro deriv by using abs nitric acid & acetic anhydride at 5-10°, or by 100% nitric acid & conc sulfuric acid and a soln of 3,3,3-trinitropropyl amine dihydrochloride & 2,2-dinitropropanol in water with 4.877NaOH added dropwise (Refs). This cmpd is sensitive to impact & useful as an exp1 additive


**Dipropylaniline and Derivatives**

**N,N-Dipropylaniline, C₆H₅N(C₃H₇)₂**, mw 177.28, N 7.90%; yel oil; sp gr 0.9104 at 20.4°, bp 244.4°; insol in w; insol in alc & ether. It can be prepared 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, C₂H₃H₅N₄**, and **Diazido, C₁₂H₁₁N₇** derivs were not found in Beil

**4-(or p)-Nitroso-N,N-dipropylaniline**, ON\(C₆H₄\cdotN(C₃H₇)₂\), mw 206.28, N 13.58%; emerald-gm triclinic crystals (from petr eth), mp 42°, sol in alc, eth, chl & benz; decomp by heating in KOH soln to p-nitrosophenol & dipropylaniline. It can be prepared by treating an ice-cold HCl soln of dipropylaniline with Na nitrite. It forms a hydrochloride salt, \(C₁₂H₁₈N₂O\cdotHCl\), greenish microcryst powd, dec on heating rapidly at 160-65°

Ref: Beil 12, 685, (338) & [366]

**Mononitro-N,N-dipropylaniline**, \(O₂N\cdotC₆H₅\cdotN(C₃H₇)₂\), mw 222.28, N 12.60%: 2-Nitro-N,N-dipropylaniline, orm-yel, unpleasant acid smelling oil, fr p - below solid CO₂ temp, bp - dec on heating; sol in w; sol in common org solvs. It was prepared by reacting 2-chloro-1-nitrobenzene & dipropylaniline in the presence of Cu powd in alc at 100°(Refs 1 & 4). It forms cryst addn salts, some of which are unstable: 

Chloride, \(C₁₂H₁₈N₂O₂\cdotHCl\cdotAuCl₃\), yel prisms (from alc), dec on heating in alc

Cyanate, \(2C₁₂H₁₈N₂O₂\cdotHFe(CN)₄\cdot2H₂O\), bm crysts (from alc), dec on exposure to light Cyanate, \(2C₁₂H₁₈N₂O₂\cdotH₃Fe(CN)₄\cdot2H₂O\), yel crystals, unstable in light Iodide, \(C₁₂H₁₈N₂O₂\cdotHI\), ndls, decmp in light

Picrate, \(C₁₂H₁₈N₂O₃\cdotC₆H₅₃O₇\), golden shiny plts (from abs alc), mp 93-94°, degr on heating rapidly (Ref 1) 3-Nitro-N,N-dipropylaniline, not found in the literature thru 1966

4-Nitro-N,N-dipropylaniline, 1-t green crystals, mp 59°. It was prepared by heating 4-bromo-1-nitrobenzene with dipropylaniline in a tube at 183°(Refs 2 & 3)


**Dinitro-N,N-dipropylaniline**, \(O₂N\cdotC₆H₅\cdotN(C₃H₇)₂\), mw 267.28, N 15.72%. Only two isomers have been prep3: 2,4-Dinitro-N,N-dipropylaniline, bm-yel rhomb bipyramides (from acet), mp 40° or yel crystals, mp 41°; d 1.231 at 20°. It was prep3 by re-
acting 4-bromo- or 4-chloro-1,3-dinitrobenzene with dipropylamine (Refs 1, 2, 4 & 6)
2,6-Dinitro-N,N-dipropylaniline, cyrstals, mp 50–52°; was prepd by reaction of 2,6-dinitro-chlorobenzene & dipropylamine 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)

2,4,6-Trinitro-N,N-dipropylaniline,
(O₂N)₃C₆H₄N(C₆H₅)₃; mw 312.28, N 17.94%; om-red triclinic crystals (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 & dipropylaniline 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′-aza-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,
(C₃H₇) – C – NH – N – NH – C(C₆H₅)
     N — C — C — N
mw 220.28, N 38.16%; wh crystals, mp – sublimes; sol in acet & chlf; sol in acids; sl sol

in hot w & ammonia. It was prepd by sustained heating of cyanophenylhydrazine,
(C₇H₇NO₅)₃N – NH – C(C₆H₅)₃ & propionic anhydride,
(C₃H₇CO)₂O
No nitrated derivs are reported in the literature thru 1966
Refs: 1) Beil, not found 2) G. Dedichen, AvhndlNorskeVidenskaps-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′-Dipropylphenylene or Dipropylaminophenyl,
H₇C₃-HN.C₆H₄C₆-H₇; 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:
Dinitrodi(propylbenzidine, C₁₈H₂₂N₂O₄, not found in the literature thru 1966
3,5,3′,5′-Tetranitro-N,N′-dipropylbenzidine,
H₇C₃-HN.C₆H₃(NO₂)₂-C₆H₄-H₂(NO₂)₂-NH.C₆H₄-H₇; mw 448.39, N 18.74%; dk-red nlds (from methyl salicylate), mp 200°. It was prepd by heating propylaniline with tetrinitrophenol 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,
H₇C₃-N(NO₂)₂.C₆H₃(H₂)(NO₂)₂-C₆H₄-H₂(NO₂)₂-N(NO₂)₂-C₆H₄-H₇; mw 538.39, N 26.82%; It-yel pldts (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 (Patent compd) 2) Beil, 13, [109] 3) Beil, 13, [110] 4) G. van Rombouhgh, Rec 41, 42 (1922) & CA 16, 1238 (1922) 5) Neither the parent compd nor its nitrated derivs were found in later refs thru 1966

Dipropylbenzidine and Derivatives
N,N′-Dipropylbenzidine, N,N′-Dipropylphenylene or Dipropylaminophenyl,
(H₈C)₃-C₆H₃-H₇, C₆H₄-H₇, CH(CH₃)₂; mw 268.39, N 10.44%. It may be considered as the parent compd (Ref 1) of its nitrated
Dipropylbutyrate and Derivatives

Dipropylbutyrate or Propylbutyrate,
\[ \text{CH}_3\text{CH}_2\text{CH}_2\text{O.CO.CH}_2\text{CH}_2\text{CH}_3 \] mw 130.18
liq, fr p – minus 95.2°, bp 142.7° at 764 mm press; sp gr 0.8730 at 20°; nD 1.4001 at 20°. It was prep'd by reacting butyric acid with propyl alcohol in the presence of p-toluenesulfonic acid in hot benz.

Ref: 1) Beil, 2, 271, (120), [246] &[596]

Azido, \( \text{C}_3\text{H}_7\text{N}_3\text{O}_2\), and Diazido, \( \text{C}_3\text{H}_7\text{N}_2\text{O}_2\), derivs, not found in the literature thru 1966.

2-Nitropropyl-butyrte,
\[ \text{C}_3\text{H}_7\text{COO.CH}_2\text{CH(NO}_2\text{)CH}_3 \] mw 175.18, N 8.00%, liq, bp 226–28° (some decompn), sp gr 1.0860 at 20°, nD 1.4321 at 20°; was prep'd by heating 2-nitropropyl alc with butyric acid & concd sulfuric acid in benz (Refs 1 & 2)

Ref: 1) Beil 2, 271; 2) J.B. Tindall, IEC 33, 65 (1941) & CA 35, 2113 (1941)

Dinitro, \( \text{C}_3\text{H}_7\text{N}_2\text{O}_5\), Trinitro, \( \text{C}_3\text{H}_7\text{N}_3\text{O}_8\), and Tetranitro, \( \text{C}_3\text{H}_7\text{N}_4\text{O}_{10} \), derivs not found in the literature thru 1966.

2,2-Dinitropropyl-4,4,4-trinitro-butyrte, (DNPTNBu)
\[ \text{CH}_3\text{C(NO}_2\text{)CH}_2\text{O.CO.CH}_2\text{CH}_2\text{C(NO}_2\text{)}_9 \] mw 555.12, N 19.72%, OB to CO2 minus 29%; wh crs, sp gr 1.68, mp 95–96°; exists in three crystalline modifications, of which Forms I & II are unstable, while Form II is stable and its mp is 95°. It was prep’d 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:
\[ \text{CH}_3\text{C(NO}_2\text{)CH}_2\text{O.CO.CH}_2\text{CH}_2\text{C(NO}_2\text{)}_9 \rightarrow \text{DNPTNBu} \]
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:

\begin{itemize}
  \item \textbf{Explosion Temperature} – 300° in 5 secs
  \item \textbf{Rate of Detonation} – 7600m/sec at sp gr 1.67
  \item \textbf{Vacuum Stability Test} at 100° – 0.5cc/40 hrs
\end{itemize}

It can be stored dry and cast loaded. Upon solidification of molten material, Form II is always observed.


6) A. Solomonovici & S. Blumberg, Israel J. Chem 3(2) 63 (1965) (Engl) & CA 63, 6851 (1965)
7) M.E. Hill, USP 3223725 (1965) & CA 64, 6570 (1966); USP 3230427 (1966) & CA 64, 9598 (1966)

Hexanitropropylbutyrate, \( \text{C}_3\text{H}_8\text{N}_6\text{O}_{14} \), not found in the literature thru 1966.
Di(propylenediamino)-copper (II) Dioxide, [Cu(CH₃CH₂NH₂₂.CH₂NH₂₂)_2(N₃)₂; mw 295.84, N 47.35%; dk violet ndls; two modifications; mp 165-66°C; bp = burns in flame with crepitations; dissolves in w imparting a violet coloration. It can be prep'd 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, 6537 (1943)

Dipropylene glycol and Derivatives

Dipropylene glycol at 1,1'-Oxydi-2-propanol, (CH₃CHOH.CH₂)₂O, mw 134.17, O 35.77%; colorless liq, sp gr 1.025 at 20/20°C, fr p >-50°C, bp 231.8°F, viscosity 1.07 poise at 20°C; sol in w & in toluene. It can be prep'd by the condensation of propylene oxide & propylene glycol at 117-18°C (Refs 1 & 2). It is twice as toxic as propylene glycol. Industr prep is described in Ref 3.
It is a fairly good solvent for NC

Dipropylene glycol Dinitrate (DPGDN), CH₃.CH(OONO₂)₂.CH₂.O.CH₂.CH(OONO₂)₂.CH₃, mw 224.17, N 12.5%; colorless liq, fr p not reported, bp 100°C at 0.1mm press; sp gr 1.2310 at 21°C, nD 1.4460 at 15°C (Ref 3). It can be prep'd by nitration of dipropylene glycol. Macy & Saffitz (Ref 2) prep'd it in PicArsn Chem Lab by nitration of product supplied by Carbide & Carbon Corp using a nitrating mix composed of acetic anhydride, acetic acid, and 93% nitric acid at 3-5°C. DPGDN is volatile to the extent of 0.35mg/sq cm/hr at 65°C (Ref 2)
It is an exql with impact sensitivity of 9 inch damp 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°C. Its % acidity was 0.123 in 5 days at 60°C in Hydrolysis Test (Ref 2)

It is suitable for use as an ingredient of double-base propells. 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, C₆H₄O, mw 102.17, O 15.66%.
The following isomers are known:
*n-Dipropyl Ether, (C₃H₇₂.CH₂)₂O, colorless liq, fr p minus 122°C, bp 90.1°C; sl sol in w; sol in alc & ether (Ref 1)
Di-(iso-Propyl) Ether, [(CH₃)₂CH]₂O, colorless liq, fr p minus 60°C, bp 68.5-69°C; sl sol in w; sol in alc & ether (Ref 2)
n-Propyl-iso-propyl Ether, C₆H₄₂.CH₂.O.CH[(CH₃)₂], colorless liq, bp 82-83°C; sl sol in w (Ref 3)
Morgan & Pickard (Ref 4) described two exqls 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

Di(or Bis)[2-propyleniminoethyI] sulone
[called 1,1'-((Sulfonyldiethylen)bis[2-methyl-aziridine] in CA Formula Index 56-65 (1962-66), p115F],

[CH₃]
[CH
\[N.CH₂.CH₂\]
SO₂]

mw 237.35, N 11.81%; water-white to lt-amber liq, bp 145-48°C at 0.8mm press, sp gr 1.0846 at 25°C, nD 1.4816 at 25°C and viscosity of 70cp at 25°C. It was produced by reaction of divinyl sulone with propylenimine in a mole ratio of ~2.15 at 15-25°C. 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 Thiolec Chemical Corp, BritP 1003385(1955) & CA 63, 12963(1965); USP 3235544(1966) & CA 64, 14168(1966)

Dipropyl-fumarate and Derivatives

Di(propyl)-fumarate (called in CA Dipropyl Ester of Fumaric Acid),

CH₃COO.CH₂.CH₂.CH₃
CH₂.CO.CH₂.CH₂.CH₃, mw 200.23, O 31.96%, liq. bp 110° at 5mm press, sp gr 1.0129 at 20°, nD 1.440 at 20°. It was prep by reacting Ag fumarate with propyl bromide in boiling benzene (Ref 1)

Bis(2,2-dinitropropyl)-fumarate,

CH₃COO.CH₂.C(NO₂)₂.CH₃
CH₂.CO.CH₂.C(NO₂)₂.CH₃, mw 380.23, N 14.73%, transparent ndls (from chlf & hexane) mp 85.6-86.2°; sp gr 1.60, sol in CC1₄, CS₂ dichloro ethylene & nitrobenz. It was prep'd in 80% yield by heating 45° dinitropropyl alcohol with fumaryl chloride in chlf and AlCl₃ 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 comps (Refs 3 & 4)


Di(propyl)-amino and Derivatives

Di(iso-propylol)-amine; N,N-Di(2-propylol)-amine or Bis(β-hydroxypropyl)-amine,

HN
CH₂.CH(OH).CH₃

CH₂.CH(OH).CH₂, mw 133.19, N 10.52%, crys, mp 44.5-45.5°, bp 249-50° at 745mm press, sp gr 1.003 at 25°; sol in w & alc; sl sol in ehd. It can be prep'd by reaction of propylene oxide & ammonia in ag soln. It forms cryst salts of Hydrochloride, Hexachloroplatinate & Picrate (Ref 1). It gives on nitration the compd listed below


Di(2-nitroxy-isopropyl)-amine Nitrate; N,N-Di(β-nitroxy-isopropyl)-amine Nitrate or N,N-Bis(β-nitroxy-isopropyl)-amine Nitrate,

HN
CH₂.CH(NO₂).CH₃

CH₂.CH(NO₂).CH₂, mw 286.20, N 19.58%, OB to CO₂ minus 55.9%; crys (from w), mp 119.6°(Ref 1) & 144° (Ref 2). It can be prep'd by treating N,N-di(β-propylol)-amine with 98% nitric acid at below 15°(Ref 1). It was used by Blomquist & Fiedorek for prep of the compd listed below

Refs: Same as under next compd

Di(2-nitroxy-isopropyl)-nitramine; N,N-Di(β-nitroxy-isopropyl)-nitramine or N,N-Bis(β-nitroxy-isopropyl)-nitramine,

(O₂N)N
CH₂.CH(NO₂).CH₃

CH₂.CH(NO₂).CH₂, mw 268.19, N 20.89%, OB to CO₂ minus 59.7%; crys (from ether), mp 103-104°(Ref 2). It was prep'd 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

Dipropylketone or 4-Heptanone. See Butyroine in Vol 2, p B393-L
detonated by primary expls. It was recommended as a base chge in compd detonators


\[ \text{N, N'-Di(3-nitroxypropyl)-nitramine, called by Fishbein Dihydroxypropyl Trinitrate,} \]
\[ \text{O}_2\text{N} \bigg\langle \text{CH}_2\text{.CH}_2\text{.CH}(\text{ONO}_2) \bigg\rangle \text{CH}_2\text{.CH}_2\text{.CH}_2(\text{ONO}_2), \text{mw 268.19,} \]
\[ \text{N 20.89%, OB to CO}_2 \text{ minus 55.9%; crys (from water), mp 143°} \text{ explosive on heating at higher temp. It can be prepd by nitration of dipropylamine with mixed nitric-sulfuric acid at a temp between 0° & 10°} \]

It was patented in Europe for use as an explosive alone or in mixtures


Di(propylol)-ethylenediamine and Derivatives
\[ \text{N, N'-Di(propylol)-ethylenediamine, N, N'-Di-(propanol)-ethylenediamine or N, N'-Bis(β-hydroxypropyl)-ethylenediamine,} \]
\[ \text{HO.H}_3\text{C}_{H-3}.\text{HN.CH}_2.\text{CH}_2.\text{NH.C}_3\text{H}_7.\text{OH,} \text{mw 178.27, N 15.71%. This compd may be considered as the parent compd of the nitrated derivatives listed below, although they were not prepd from it:} \]
\[ \text{N, N'-Di(propylol)-ethylenedinitramine; N, N'-Di(propanol)-ethylenedinitramine or N, N'-Bis(β-hydroxypropyl)-ethylenedinitramine,} \]
\[ \text{HO.H}_7\text{C}_3.(\text{O}_2\text{N}.\text{N.CH}_2.\text{CH}_2.\text{N} \text{(NO}_2)_2).\text{C}_3\text{H}_7.\text{OH;} \text{mw 268.28, N 20.89%; sl colored glossy oil. It was obtd by stirring for several days a suspension of ethylenedinitramine and propylene-oxide in water contg some NaOH. The product did not crystallize} \]
\[ \text{Nitration of this product with 98% nitric acid at 5-10° gave two crops of crys. The} \]

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:
\[ \text{N, N'-Di(2-nitroxypropyl)-ethylenedinitramine,} \]
\[ \text{(O}_2\text{N).H}_7\text{C}_3.(\text{O}_2\text{N}.\text{N.CH}_2.\text{CH}_2.\text{N} \text{(NO}_2)_2).\text{C}_3\text{H}_7(\text{ONO}_2)_2,} \text{mw 358.28, N 23.46%; obtd in two crops: 1st crop (alc), mp 94-96°, 2nd crop (alc), mp 78-81°. The above oil was added, while stirring, to 98% nitric acid at 5-10° for 15 mins, and the reaction quenched in ice & water. The crude product was twice recrystallized.} \]

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
\[ \text{Dipropylpentaerythritol Ether,} \]
\[ \text{C}_3\text{H}_7.\text{O.CH}_2.\text{C.CH}_2.\text{O.C}_3\text{H}_7 \]
\[ \bigg\langle \text{(CH}_2.\text{OH)}_2 \bigg\rangle \]
\[ \text{mw 220.30, O 29.05%, liq, bp 115° at 1mm press, sp gr 0.993 at 20°, n_D 1.4461 at 20°. It was prepd by hydrogenating at RT & atmospheric press, using Pt oxide, a soln of diallyl pentaerythritol in 95% alc (Ref 3) \]

Dipropylpentaerythritol Dinitrate [called 2,2-Bis(propoxymethyl)-1,3-propanediol Dinitrate in CA],
\[ \text{C}_3\text{H}_7.\text{O.CH}_2.\text{C.CH}_2.\text{O.C}_3\text{H}_7 \]
\[ \bigg\langle \text{(CH}_2.\text{ONO}_2)_2 \bigg\rangle \]
\[ \text{mw 310.30, N 9.03%, liq sp gr 1.144, n_D 1.4470 at 20°. It was prepd by nitrating the parent compd with nitric acid (Ref 3). It has possible use as a gelatinizer of NC} \]

Refs: 1) Beil, not found (Parent)  2) Beil, not found (Dinitrate)  3) R. Evans & J.A. Gallagher, 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, C₆H₁₃−OO−C₃H₇, 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 smaller pressures than Diethyl Peroxide (Refs 3, 4 & 5)


Di(iso-propyl)-peroxide, CH₃CH−OO−CH(CH₃)₂, mw 118.17, O 27.08%; liquid, bp ~94° at 760mm & 55−58° at 233mm press w/o expln; not expld by impact; undergoes photo-initiated, auto-inhibited chain decmpn at 26° (Ref 6)

The only detailed method of prepn, by distilling a mixt of isopropyl methane sulfonate, 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 200° in an O stream


Dipropyl Succinate and Derivatives

Di(or Bis)(propyl)-succinate (called Dipropyl Ester of Succinic Acid in CA) (Called Bernstein's dipropylester, in Ger)

CH₂CO₂CH₂CH₂CH₃

CH₂CO₂CH₂CH₂CH₃, mw 202.24, O 31.64%, liq. fr p minus 5.9°, bp 101.5° at 3mm press, sp gr 1.0020 at 20°, nD 1.4250 at 20°. It can be prepd by azotropic distillation of succinic acid, propyl alcohol & benz (Ref 1)

Dinitrodipropyl Succinate,

CH₂CO₂CH₂CH₂(NO₂)₂CH₃, 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 comps is found in CA

2,2',2'-Tetranitrodipropyl Succinate,

CH₂CO₂CH₂CH₂(NO₂)₂CH₃, mw 382.24, N 14.68%, crs 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 insensitive compd (~30% Impact Sensitivity, 2.5kg wt >32cm), with excellent thermal stability (ignition Temp >400° & 0.10cc gas/g/48hrs at 100°)

This compd is a casetable expl


Dipropyl-sulfone and Derivatives

Dipropyl-sulfone (Called Propylsulfone in CA),
D 1501

H₃C.CH₂.CH₂.SO₂.CH₂.CH₂.CH₃, mw 150.24, O 21-30%, flakes; mp 29.5-30.5°, sp gr 1.0278 at 50°, nD 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, C₆H₄(NO₂)₂SO₂NH₂, and Tetranitro, C₆H₁₄NO₄S₄, derivs were not found in the literature thru 1966

3,3,3',3',3'-Hexanitropropyl-sulfone or Di(3,3,3-trinitropropyl)-sulfone,

(O₃N)₃.C.H₂.CH₂.SO₂.CH₂.CH₂.C(NO₂)₃; mw 420.24, N 20.00%; wh crystals, 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 1/2 hr (Refs 3 & 4). This compd is an expl and is detonated by a heavy hammer blow (Refs: 1) Beil 1, 359, [373] & [439] (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, N Ord 10139, P9 4) P.O. Tawney, USP 3146270 (1964) & CA 61, 13195 (1964)

Dipropylureas and Derivatives

N,N'(or sym)-n-Dipropylurea,

H₃C₂.CH₂.NH.CO.NH·CH₂.CH₂₃, 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,

(H₃C₂.CH₂)₂N.CO.NH₂, mw 144.21, N 19.43%, ndls (from petr eth), mp 76°; v sol in w. It can be prepd from reaction of dipropylamine & nitourea in water. It forms cryst salts, such as Nitrato, Oxalate & Picrate (Ref 2)

N,N'-Di-isopropylurea,

H₃C.CH=NH.CO.NH·CH₂.CH₃

CH₃

C(NO₂)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 isobutyl hydroxamylbenzoate (Ref 3)

N,N'(or unsym)-Di-iso-propylurea,

[(H₃C₂.CH₂)₂N.CO.NH₂, mw 144.21, N 19.43%; crysts (from eth), mp 105°. It can be prepd by treating diisopropylamine sulfate with KCNO. It forms salts of Nitrato, Picrate & Oxalate, 2C₇H₁₄N₂O₄ + C₆H₁₂O₄, ndls (from alc), dec at 111° (Ref 4)


Dinitro, C₇H₁₄N₄O₈, and Tetranitro, C₇H₁₂N₆O₈, derivs of Dipropylurea were not found in the literature thru 1966

3,3,3',3',3'-Hexanitro-N,N'-n-dipropylurea or 1,3-Bis(3,3,3-trinitropropyl)-urea,

\n\[
\text{OC} \quad \text{NH.CH₂.CH₂.C(NO₂)₃}
\]

3,3,3',3',3'-Hexanitro-N,N'-n-dipropylurea, or 1,3-Bis(3,3,3-trinitropropyl)-urea, & Dipropylamine and Derivatives

1,1,1',1',1'-Hexanitro-di-isopropylurea or Bis(1,1,1-trinitroiso-propyl) -urea,

H₃C₂.CH=NH.CO.NH·CH₂.CH₃

C(NO₂)₃

C(NO₂)₃

mw 414.21, N 27.05%, v sol in alc & eth. It was prepd by refluxing the hexanitrodipropylisocyanate with a soln of acetone & water until crysts prpd. Prepn of starting material is described (Ref 3). This compd is expl

1,1,1',1',1'-Hexanitro-di-isopropylurea or Bis (1,1,1-trinitroiso-propyl) -urea,

H₃C₂.CH=NH.CO.NH·CH₂.CH₃

C(NO₂)₃

C(NO₂)₃

mw 414.21, N 27.05%; wh crystals, mp 145° (decomp explosively). It was prepd by reaction of nitroform (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)

Ref: 1) Beil, not found 2) USRubberCo, Quart Progress Rept 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-propylo)-amine; N,N-Bis(propanol) -amine: N,N-Di(3-hydroxypropyl)-amine or Amino-dipropylol,
Dipyridine Diazido-Metal Complexes

Dipyridine Diazido (II) Diazide,
\[ \text{[Cd} \text{(C}_6\text{H}_5\text{N})_2\text{N}_2]_2 \text{;} \text{ mw 354.65, N 31.59%; solid; mp-on slow heating melts, evolving pyridine; then it assumes yellow color and detonates with a loud report; sol in hot water. It can be prepd by reaction of pyridine Cd chloride in w with a soln of NaN}_3 \text{ in pyridine (Ref 2, p213)} \]

Dipyridine Diazido or Dipyrido- Diazidocopper (II), \[ \text{[Cu} \text{(C}_6\text{H}_5\text{N})_2\text{N}_2]_2 \text{;} \text{ mw 305.78, N 36.62%; exists in two modifications: 1- dark green ndls melting at 138°, but starting to decomp above 100° lea} \]

very difficultly sol in cold w; decomp 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)

Dipyridiminediylammonium (II) Diazide,
\[ \text{[Ni} \text{(C}_6\text{H}_5\text{N})_2\text{N}_2]_2 \text{;} \text{ mw 300.93, N 37.23%; lt green crysts; mp - not reported. It can be prepd from dipyridine Ni chloride & a NaN}_3 \text{ soln (Ref 2, p217-18)} \]

Dipyridiminediylammonium (II) Diazide,
\[ \text{[Zn} \text{(C}_6\text{H}_5\text{N})_2\text{N}_2]_2 \text{;} \text{ mw 307.62, N 36.43%; wh crysts, mp - not reported. It can be prepd from Zn dipyridine chloride, pyridine & an aq NaN}_3 \text{ soln (Ref 2, p212)} \]

Dipyridylamine and Derivatives

Dipyridylamine, (NC}_6\text{H}_4\text{N})_2\text{NH;} \text{ mw 171.20, N 24.55%}. It exists as Di-a (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}_6\text{H}_4\text{N}(NO}_2\text{)}_2\text{NH;} \text{ mw 261.20, N 26.81%}. 3,3'-Dinitro-(di-a-pyridylamine), yell pltrs (from 80% acet acid), mp 179-80°. 5,5'-Dinitro-(di-a-pyridylamine), lt-yel ndls (from 80% acet acid), mp 219-20° or lt-bm ndls (from pyridine), mp 224°. X,X-Dinitro-(di-a-pyridylamine), yell-bm ndls (from toluene), mp 195-96°

Other props & methods of prepn are found in the Refs (Refs 2 & 5)

3,5,5'-Trinitro-(di-a-pyridylamine),
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

Diorescinol or Tetrahydroxybiphenyl. See Birescinol in Vol 2, p B126-R. Hexanitro-biorescinol 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 to friction and insufficient chemical stability
Ref: Daniel (1902), 206

Disalicylic Acid and Derivatives
Disalicylic Acid; Disposal or Salysal,HO.C_6H_4.CO_2.C_6H_4.CO_2.H; mw 258.22; colorless crst., 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-disalicylde.

\[
\text{C}_6\text{H}_4\text{O}.\text{CO.O}\text{C}_6\text{H}_4, \quad \text{with glacial acetic acid}
\]

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_4(NO_2).CO_2.C_6H_4.CO_2.H; mw 303.22, N 4.62%, yel ndls (from chl-ads alc), mp 196° (Ref 3). No other props given

NOTE: No Azido, C_14H_21N_5O_5, Dioxide,
C_14H_21N_5O_5, or higher nitro derivs were found in the literature thru 1966
Ref: 1) Beil 10, 84, (40), [54] & [147] 2) Beil 10, not found (Nitro derivs) 3) C.

Directed Explosives. One of the terms for Shaped Charges, described as Munroe-Neumann Effect in Vol 4, under DETONATION, p D442-R

Di(iso-quinolino)-diazido Copper (II) Complex or Di(iso-quinolino)-copper(II) Diazido,

[Cu(C_6H_4N_2)^{\text{NO_2}}]_2; mw 403.88, N 27.75%, yel-green crst.; mp - explodes at 197-200°; burns in flame; sol in ethylenediamine with violet color freeing some iso-quinolino; darkens in warm acid. It is insensitive to impact. It was prepd by dissolving Cu azide in an iso-quinolino amine soln in alc, by the addn of NaN_3 to an amine soln of the Cu^{2+} salt in MeOH, or by rearrangement of the cuprate amine (Ref 2)

Ref: 1) Beil, not found 2) A. Cinulis & 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

Diorescinol or Tetrahydroxybiphenyl. See Birescinol in Vol 2, p B126-R. Hexanitro-biorescinol 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 to friction and insufficient chemical stability
Ref: Daniel (1902), 206

Disalicylic Acid and Derivatives
Disalicylic Acid; Disposal or Salysal,
HO.C_6H_4.CO_2.C_6H_4.CO_2.H; mw 258.22; colorless crst., 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-disalicylde.

\[
\text{C}_6\text{H}_4\text{O}.\text{CO.O}\text{C}_6\text{H}_4, \quad \text{with glacial acetic acid}
\]

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_4(NO_2).CO_2.C_6H_4.CO_2.H; mw 303.22, N 4.62%, yel ndls (from chl-ads alc), mp 196° (Ref 3). No other props given

NOTE: No Azido, C_14H_21N_5O_5, Dioxide,
C_14H_21N_5O_5, or higher nitro derivs were found in the literature thru 1966
Ref: 1) Beil 10, 84, (40), [54] & [147] 2) Beil 10, not found (Nitro derivs) 3) C.

Directed Explosives. One of the terms for Shaped Charges, described as Munroe-Neumann Effect in Vol 4, under DETONATION, p D442-R

Diorescinol or Tetrahydroxybiphenyl. See Birescinol in Vol 2, p B126-R. Hexanitro-biorescinol 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 to friction and insufficient chemical stability
Ref: Daniel (1902), 206

Disalicylic Acid and Derivatives
Disalicylic Acid; Disposal or Salysal,
HO.C_6H_4.CO_2.C_6H_4.CO_2.H; mw 258.22; colorless crst., 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-disalicylde.

\[
\text{C}_6\text{H}_4\text{O}.\text{CO.O}\text{C}_6\text{H}_4, \quad \text{with glacial acetic acid}
\]

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_4(NO_2).CO_2.C_6H_4.CO_2.H; mw 303.22, N 4.62%, yel ndls (from chl-ads alc), mp 196° (Ref 3). No other props given

NOTE: No Azido, C_14H_21N_5O_5, Dioxide,
C_14H_21N_5O_5, or higher nitro derivs were found in the literature thru 1966
Ref: 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-Solz. German designation of Dimethyl-ammonium Nitrare, 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

Disorm. 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).


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.

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).


Disk Test using a lead disk is one of the output tests measuring brisance of a stab de-
Disilite (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-nitroaminotetrazole. 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

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 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 colloidal smokeless propmts, NC is "the dispersed phase"; solvent for NC - "the dispersion medium", which may also be called "the dispersing agent", and the propmt itself "the dispersed system"
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.


Dispino [fluorene-9,3′-s-tetrazone-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.

Ref's: 1) Marshall 2 (1918), 608–9 2) Davis (1943), 74–8


Disposal of Captured German Warfare Materials. The disposition of more than 12000 tons of munitions and toxic chemicals, including mustard gas, arsenicals & chloracetophenone, is described by Loucks & Elliot (Ref)


Disposal of Defective or Obsolete Projectiles in Russia. Accord 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 neatly 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°C (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, especially 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.

Recovery and 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 Reconnaisance 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 expls 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 (Kreuzaehr, in Ger). This device was used in Germany when a propnt charge of semi-fixed artillery ammunition was shorter than the cartridge case. The piece consisted of one or several tubular sticks of


Disposal Slide Rule. This is a handy, ready reference slide rule for Explosive Ordnance.
double-base propelant, which were inserted into the propelant 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 projectile), while the lower end held the bag against the primer. With this arrangement the propelant charge was not loose and, being held close to the primer flash hole, the propelant was readily ignited.

Ref: Fedoroff et al, PATR 2510 (1958), p 38-1

Distance Piece and Distance Wadding. When the capacity of a cartridge case in fixed ammo is greater than the volume of the propelant charge, 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 propelant back around the primer. In case of separate-loaded ammo the cardboard serves to maintain the proper distance between the mouth plug and the wad which covers the propelant charge.

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 293, 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
The 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°C) 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, p379.

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 prep 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) Hach's Dictionary (1944), pp 283 & 813 3) K.C. Hickman et al, IEC 38, 28-9 (Jan 1946) (Molecular distillation), and in succeeding years 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")


**Distler, Blecher & López Explosives** were patented safety expls consisting of nitroxylene as chief constituents. The nitrocompds had varying amounts of cumol, mesitylene & xylene nitro constituents mixed with AN, other nitrates or KClO₃ or metal oxides. For example, if 12 parts nitroated solvent naphtha are intimately mixed with 88 parts of AN, a very insensitive expl is produced, which is unaffected by either shock or concussion; 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

Distribution of Charge in a Bore Hole. This important factor is, accdg to Bertliner (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. Bertliner, 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-Di substituted Tetrazoles. The prepn of 1,5-di-substituted tetrazoles by the coupling of diazotized p-nitroaniline with 1-acetyl-2-para-substituted benzoyl hydrazines and cyclization of the tetrazines formed as intermediates has been investigated. From the ratio of the two tetrazoles isolated:

\[
\text{1,5-Di}(d\text{-nitrophenyl})\text{tetrazole,}
\]

\[
\text{p-NO}_2\text{C}_6\text{H}_4\text{N} - \text{C}_6\text{H}_4\text{N} - \text{NO}_2\text{p}
\]

\[
\text{1-(d'-nitrophenyl)-5-methyltetrazole,}
\]

\[
\text{p-NO}_2\text{C}_6\text{H}_4\text{N} - \text{C}_6\text{H}_8
\]

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)(1H) tetrazolium benaines 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)


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°C (with 3ecmpa)

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


Di(5-tetrazole)-hydrazine or 5,5'-Hydrazobis(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(tetrazoly1-5)-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

N1,N6-(Ditazolyl-5)-hexazadiene. See [N1,N6-Bis(a-tetrazolyl-5)]-hexazadiene
under 5-AMINO-a-TETRAZOLE in Vol 1, p A260-R

(Ditazolyl-5)-hydrazine; Di[(4-hydrotetra-
zolyl-5)]-azine; Azino-di[4-hydrotetrazole]-
5] or Hydrazo-di(tetrazolyl-5). See Bis(5-
tetrazolyl)-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

1,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 Diethyl Lead Salt is on the same page

Diteteryl or Diteteryl (US), Octyl (GbBrit).
See 1,2-Bis(2',4',6'-trinitro-nitroanilino)-
ethane in Vol 2, p B131-R

Addn Refs: A) Ye.Yu. Orlova, "Khimia i
Tekhnologiya Brizantnykh Vzryvchatykh
Veshchestv", GosizdatOborProm, Moskva
(1960), Engl translation, "Chemistry and
Technology of High Explosives", by Tech-
nical Documentation Liaison Office, MCLTD,
WP-AFB, Ohio, MCL-844/112, June 1961,
p 472 (compd called Diteteryl) B) R.
Simkins, "Diteteryl", ERDE (Explosives
Research & Development Establishment),
TechMemo No 21/M/64, Sept 1964 C) T.
Urbanski, "Chemistry and Technology of
Explosives", Translated from Polish, Per-
called Diteteryl)

Dithekite. A Sprungel 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 con-
tainer. 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 Repr 2014(1944)
2) ArmamentEngng(1954), 71 3) Cook(1958),
190-94

Dithiocarbamate of Ammonium,
H2N.C.S.S.NH4; mw 110.20, N 25.42%; It-yel
unstable crystals; 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
decom. Its lab prep 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); Org-
SynthCollective Vol 3(1955), 763-65

Dithiocarbamyl-hydrazine. See N,N1-
Bis-(thiocarbamyl)-hydrazine in Vol 2 of Encycl,
pp B158-R & B159-L

Dithiocarbonyl Azide. See Azidodicarbonyl-
carbonic Acid and Derivatives, Vol 1, p
A632-L
Dithiocarbonyldiazide Disulfide or Azidocarbodiisulfide, \((N_3)\text{CS}_2\text{S}_2\)\text{S}_2, \text{mw 236.33, N 35.56%}. \text{See Diazidodithioformyl Disulfide or Diazidodithiocarbonyl Disulfide in this Vol}

**Dithio-hydrazo-di carbonamide.** One of the names for \(N,N^{\prime}\)-Bis(thiocarbamyl)-hydrazine, described in Vol 2, p B158-R

1,2-Dithiol-ethyl-phosphorous Acid, \((\text{CH}_3\text{S})_2\text{PO.C}_2\text{H}_5\), \text{mw 128.24; liq with unpleasant odor; sp gr 1.2629 at 20/0\(^\circ\), n\(_D\) 1.5922 at 20\(^\circ\); bp 98–99\(^\circ\) at 5mm. It can be prepd by adding \(\text{C}_4\text{H}_8\text{OPCl}_2\) dropwise to ice-cooled \((\text{CH}_3\text{SH})_2\) with pyridine in ether. It ignites with flame on contact with concd nitric acid (Ref 2). It yields adducts with \(\text{CuCl} \& \text{Cul which rapidly decompn in air}

1,2-Dithiol-methyl-phosphorous Acid, \((\text{CH}_3\text{S})_2\text{PO.C}_2\text{H}_3\), \text{mw 134.20; liq with unpleasant odor; sp gr 1.3426 at 20\(^\circ\), n\(_D\) 1.6200 at 20\(^\circ\). It can be prepd by adding \(\text{CH}_2\text{OPCl}_2\) dropwise to ice-cooled \((\text{CH}_2\text{SH})_2\) with pyridine in ether. It ignites with flame on contact with concd nitric acid.

Heating of methyl derivative with \(\text{CH}_3\text{I}\) in a sealed tube produced a dark liquid which exploded with loss of the product


**Dithizonates or Dithiozonates.** These are salts of dithzone or diphenylthiocarbazone, \(\text{C}_6\text{H}_5\text{N}\text{.C}6\text{H}_5\text{.SH}\). Dithizonates of silver, lead, zinc & copper were prepd in two ways: 1) ppm by addn of the cation soln to an ammoniacal soln of dithzone and 2) treatment of a chl soln of dithione with an aq soln of the cation. All showed rapid, almost expl decompn at the following temps: \(\text{Silver 186\(^\circ\), Lead 215\(^\circ\), Zinc 225\(^\circ\) & Copper 150\(^\circ\). Above the expl decompn, slow addnl decompn proceeds with the final formation of \(\text{Ag, PbS, PbO, ZnO} \& \text{CuO, respectively}


**Di(toluidino)-propane.** See Bis(methylphenylamino)-propane in Vol 2, p B151-L

**Ditoly or Bis-tolyl.** See Bitolyl in Vol 2, p B163-L

**Ditolyl-amine or Dimethyl-diphenylamine and Derivatives**

**Ditolyl-amine, \((\text{CH}_3\text{C}_6\text{H}_4\text{)}_2\text{NH}, \text{mw 197.27, N 7.10%}. The following isomers exist:**

\(Di-o\text{-tolyl-amine (2,2')}, \text{liq, bp 312\(^\circ\) at 727,8mm (Ref 1)}\)

\(Di-m\text{-tolyl-amine (3,3'), liq, bp 320–324\(^\circ\) (Ref 2)}\)

\(Di-p\text{-tolyl-amine (4,4'), ndls, mp 79\(^\circ\), bp 330.5\(^\circ\) (Ref 3)}\)

\(Di-o, m\text{-tolyl-amine (2,3'), Lt-yel oil, fr p < -15\(^\circ\), bp 187\(^\circ\) at 22mm press (Ref 4)}\)

\(Di-o, p\text{-tolyl-amine (2,4'), Lt-yel liq, fr p - does not solidify on cooling, bp 183\(^\circ\) at 10mm press (Ref 5)}\)

\(Di-m, p\text{-tolyl-amine (3,4'), liq, bp > 300\(^\circ\) (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, \(\text{C}_4\text{H}_4\text{N}_3\text{N}_4\), and Diazido, \(\text{C}_4\text{H}_4\text{N}_3\text{N}_7\) derivs of Ditolyl-amine were not found in the literature thru 1966

**Dinitro-ditolylamine or Dinitro-dimethyl-diphenylamine, \(\text{C}_6\text{H}_4\text{H}_1\text{N}_3\text{O}_2\), \text{mw 287.27, N 16.43%}. The following isomers are found in Beil:**

\(4,6\text{-Dinitro-2,2'-dilolylamine, \text{CH}_3\text{C}_6\text{H}_4\text{N}_3\text{O}_2\text{.NH.C}_6\text{H}_4\text{.CH}_3, red crysts (from alc) mp 109\(^\circ\) (Ref 7, p 462)}\)

\(2,6\text{-Dinitro-3,3'-dilolylamine, brick-red crysts (from alc) mp 110\(^\circ\) (Ref 7, p 479)}\)

\(4,6\text{-Dinitro-3,3'-dilolylamine, om-colored} \)
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,
CH₃C₆H₄(NO₂)₂.NH(NO₂).C₆H₄.CH₃,
red ndls (from benz), mp 192-93° (dec) or
yel-brown prills (from alc), mp 191° (Ref 7,
p1002 & 536)
4',6'-Dinitro-2,3'-ditolylamine,
CH₃C₆H₄(NO₂)₂.NH.C₆H₄.CH₃, brn-red
crysts (from alc), mp 99° (Ref 7, p461)
2',6'-Dinitro-2',4'-ditolylamine, black-red
crysts, mp 124° (Ref 7, p443)
2,6-Dinitro-3,4'-ditolylamine,
CH₃C₆H₄.NH(NO₂)₂.C₆H₄.CH₃, 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,
CH₃C₆H₄(NO₂)₂.NH.C₆H₄.CH₃, dk-red
crysts, mp 127° (Ref 7, p443)

Other props & methods of prep are
given in the Refs

Tetranitro-ditolylamine, C₁₄H₁₁N₆O₆, not
found in the literature thru 1966

Hexanitro-di-tolylamine or Bis(2-methyl-
trinitro-phenyl)-amine,
(O₂N)₃C₆H₄H(CH₃)₂-NH-(CH₃)C₆H₄(NO₂)₃;
mw 467.27, N 20.98%, not found in the
literature thru 1966
2,4,6,2',4',6'-Hexanitro-m-ditolylamine or
Bis(2,4,6-trinitro-m-phenyl-phenyl)-amine,
(O₂N)₃C₆H₄H(CH₃)₂-NH-(CH₃)C₆H₄(NO₂)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 ANILIN-
OXYLENE AND DERIVATIVES (Refs 8 & 11)
3,2,6,2',3',6'-Hexanitro-di-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
4) Beil 12, [494]
5) Beil 12, [494]
6) Beil 12, (414)
7) Beil 12, 1002, (443) & [462, 479, 481,
494, 530]
8) Beil 12, [482] (Hexanitro-m-
ditolyl) 9) Beil 12, 1013 (Hexanitro-p-
ditolyl) 10) A. Lehne, Ber 13, 1543 (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(toly1) disulfides or Dimethyl-diphenyl-
disulfide and Derivatives

Di(toly1) disulfide, H₃C.C₆H₄-.S.S-.C₆H₄.CH₃, mw 246.37;
exists as three isomers:

Di-o-tolyl-disulfide, prills (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

Other props & methods of prep are found
in the Refs

Dinitro-di-tolyl disulfide or Dinitro-dimethyl-
diphenyl-disulfide, C₁₄H₁₂N₂O₄S₂; 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,
H₃C.C₆H₄(NO₂)₂-S.S-(NO₂).C₆H₄.H₃.CH₃
1-t-yl ndls (from glc ac et), 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 glc ac et; 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 glc ac et), mp 163°; sol in hot aq
Na₂S soln (Ref 4, p 1339)
6,6'-Dinitro-3,3'-ditolyl-disulfide, yel-gm
crysts (from glc ac et or benz), mp 152°
(Ref 4, p 1338)
2,2'-Dinitro-4,4'-ditolyldisulphide. 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'-ditolysulphide; 4,6,4',6'-Tetranitro-3,3'-dimethyl-diphenyl-disulphide; or Bis(4,6-Dinitro-m-tolyl)-Disulphide

C(CH3)3-CH [H-C-S-S-C] C(NO2)
C(NO2)=CH

mw 426.39, N 13.14%, yel pptts (from xylene) or lit-yel ndls (from Nitrobenz-talc 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 Na2S2O3 in alc (Refs 5 & 6). It was prepd later by Kiprianov & Zhuravlova (Ref 7) using somewhat the same procedure


Hexanitroditolysulphide, C1H2N6O12S2, not found in the literature thru 1966

Di(toly1)-ethylenediamine. See Bis(methylphenylamino)-ethane in Vol 2, p B150-R

N,N'-Di-tolylguanidine, Derivatives

N,N'-Di-tolylguanidine, mp 168-70° & 166.5-167.5° (Ref 3)

N-o-Tolyl-N'-p-tolylguanidine, ndls (from w), mp 120-21° (Ref 4)

Other props & methods of prep are found in Refs

N,N'-Di-(2(?)nitrotolyl)-guanidine or N,N'-Bis(2(?)-nitro-4-methyl-phenyl)-guanidine, 

[1H2C6H5(NO2)2]NH2-CN; mw 329.31, N 21.27%; om-red prisms (from alc), mp 197° (dec); sol in acds, sl sol in cold alkalds; 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 Ditolylguanidine were found in the literature thru 1966


Di(tolyl)-hydroazine, Hydrazotoluene or Dimethylhydroazobenzene and Derivatives

N,N'-Di-(or Bis(tolyl))-hydroazines or Hydrazotoluenes, C1H2C6H4-NH-C6H4-CH3, mw 212.28, N 13.20%; four isomers are known: N,N'-Di-o-tolyl-hydroazine, litfts, mp 165°, sol in eth & benz (Ref 1)

N,N'-Di-o-m-tolyl-hydroazine, colorless oil, fr p - not reported; v sl sol in alc (Ref 2)

N,N'-p-tolyl-hydroazine, crysts, mp 133-34°, bp - decomp (Ref 3)

N-m-Tolyl-N',p-tolyl-hydroazine, plotts (from petr eth), mp 74° (Ref 3)

Other props & methods of prep are found in Refs

Dinitro-N,N'-ditolyl-hydroazine or Dinitrodimethyl-hydrazobenzene, C1H14N2O4, mw 302.28, N 18.54%. Three isomers are known:

4,6-Dinitro-2,3'-dimethyl-hydrazobenzene, 1H2C6H5(NO2)2NH-NH-C6H4-CH3, om-red plotts (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, 
on-pol nl (from alc), mp 165-66°(dec); 
sol in chl, alc, acet & benz; sl sol in petr 
eth (Ref 4, p 153 & Ref 6)

4,6-Dinitro-3,4'-dimethyl-hydrazobenzene, 
on-pol nl (from alc), mp 159-60°(dec); 
sol in chl, 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 

derives not found in later refs thru 1966

4,4',6,6'-Tetranitro-di-(m-tolyl) -hydrazine;
1,2-Bis(4,6-dinitro-m-tolyl) -hydrazine (CA 
nomenclature); or 5,5'-Dimethyl-2,4,2',4'-
tetrinitrohydrobenzene (Authors' name), 
H₃C.C=CH-CH₃-CH=NH.NH-CH=CH₃, C₆H₅O₂₅ 
O₂N.C=CH-CH₃, C₆H₅O₂₅ 
mw 482.28, N 23.24%, pol-nil crystals (from 
acet et), mp 244°C; gives an intense purple 
color when dissolved in a soln of KOH (1%) 
in alc. It was obt by heating 5-methyl-
2,4-dinitrophenylhydrazine, 5-methyl-2,4-
dinitrofluorobenzene & CaCO₃ 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, C₁₄H₁₀N₆O₁₂, 
not found in the literature thru 1966

Di(tolyl)-phthalide and Derivatives

Di( or Bis)tolyl -phthalide,

C₆H₄
\[
\bigl(\bigl(C₆H₄\bigr)₂\bigr)\bigl(C₆H₄\bigr)\bigl(C₆H₄\bigl)₂\bigr)
\]
mw 314.36; exists as two isomers: 
3,3-Di-o-tolyl-phthalide, crystals (from glc 
acet et), mp 128-31°(Ref 1) 3,3-Di-p-tolyl-phthalide, crystals (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, 
C₆H₄
\[
\bigl(C₆H₄\bigl(NO₂\bigr)₂\bigr)(C₆H₄)₁₂\bigr)
\]
mw 404.36, N 6.93%, crystals (from alc), mp 
157-58°; was prepd by nitrating 3,3-Dip-t 
tolyl-phthalide with nitric acid at -10° 
(Refs 3 & 6)

Tetranitro, C₂₉H₁₂N₄O₁₀, and Hexanitro, 
C₂₉H₁₂N₆O₁₄, derives not found in the 
literature thru 1966

Octanitro-3,3-di-p-tolylphthalide, 
C₆H₄₁₀O₆(NO₂)₈, mw 674.36, N 15.87%; 
dils, 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, C₁₄H₁₈O₂; 
mw 254.31. Three isomers are known:

\[
\begin{align*}
2,2-Di-p-tolylpropionic Acid, & \quad \text{C₆H₄₁₈O₂} \\
& \quad \text{(C₆H₄)₂C(CH₃)₂COOH, cubic crystals} \\
& \quad \text{(from eth or alc) or monoclinic prisms, mp} \\
& \quad \text{151-52°; volatilizes w/o decomp; sol in} \\
& \quad \text{eth, benz, chl, carbon disulfide, glc acet} \\
& \quad \text{acid & hot alc (Ref 1)} \\
3,3-Di-p-tolylpropionic Acid, & \quad \text{(C₆H₄)₂C(CH₃)₂COOH, crystals (from} \\
& \quad \text{alc), mp 187-89° (Ref 2)} \\
3,3-Di-o-tolylpropionic Acid, & \quad \text{crystals (from} \\
& \quad \text{benz), mp 133-34°; mod sol in alc & benz} \\
& \quad \text{sl sol in petr eth; almost insol in w (Ref 2)} \\
x x-Dinitro-2,2-di-p-tolylpropionic Acid or 
2,2-Bis(x-nitro-4-methylphenyl)-propionic Acid, & \quad \text{(H₃C.C₆H₄(NO₂)₂C(CH₃)₂COOH, mw} \\
& \quad \text{344.31, N 8.14%, yel crystals (from glc acet} \
\end{align*}
\]
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-tolyldipropionic Acid,** [H₃C₆H₄(NO₂)₃]₂C(CH₃)₄COOH, mw 434.31, N 12.90%, hydrated yel crys (from alc) which decomp in the light, mp 223–25° (dec), mod sol in alc, eth, acet, chlf, carbon disulfide & glc 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


7) Not found in later refs thru 1966

**Hexanitri-l-ditolyl-propionic Acid, C₁₇H₁₈N₄O₄, not found in the literature thru 1966**

**Di(toly)-sulfide or Dimethyl-diphenylsulfide and Derivatives**

**Di(toly)-sulfide, (H₃C₆H₄)₂S, mw 214.32.**

Several isomers are known:

**Di-o-toly-sulfide, tablets (from alc), mp 64°, bp 285° (Ref 1)**

**Di-m-toly-sulfide, yel oil, bp 175° at 15mm & 290° at 760mm (Ref 2)**

**Di-p-toly-sulfide, ndls (from alc), mp 56.6–57°, bp 175° at 16mm (Ref 3)**

**Di-o,m-toly-sulfide, odorless oil, bp 170° at 11mm (Ref 4)**

**Di-o,p-toly-sulfide, liq, bp 173° at 11mm, sp gr 1.0774 at 15/40° (Ref 5)**

**Di-m,p-toly-sulfide, liq, fr p 28° (Ref 6)**

Other props & methods of prepn are found in the Refs

**Dinitro-ditolyl-sulfide; Dinitrodimethyl-diphenylsulfide or Bis(nitrormethylphenyl)-sulfide, [H₃C₆H₄(NO₂)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 crys (from alc), mp 152° (Ref 7, pp 343 & 1284)

4,4'-Dinitro-3,3'-dimethyl-diphenyl-sulfide, pale-yel crys (from glc acet ac), mp 164°; insol in hot aq solns of Na₂S or Na₂S₂ (Ref 7, p 1339)

2,2'-Dinitro-4,4'-dimethyl-diphenyl-sulfide, dk-red prisms (from acet), mp 125–26° (Ref 7, p 401)

6,2'-Dinitro-3,4'-dimethyl-diphenyl-sulfide, pale yel prls (from ac acet ac), mp 131–32° (Ref 7, p 1438)

Other props & methods of prepn are found in the Refs

**Trinitro-ditolylsulfide — not found in the literature thru 1966**

**Tetranitro-sulfide, Dinitrodimethyl-diphenylsulfide or Bis(dinitromethylphenyl)-sulfide, H₃C₆H₄(NO₂)₂SC₂H₄(NO₂)₂CH₃; mw 394.32, N 14.21%.** Several isomers exist: Bis(4,6-dinitro-2-methyl-phenyl) -sulfide, yel crys, mp 210°; unchanged by warming in nitric acid; was prepd by reaction of 3,5-dinitro-2-bromotoluene with Na₂S (Ref 8, p 373)

2,6,2',6'-Tetranitro-3,3'-dimethyl-diphenylsulfide, yel crys (from glc acet ac), mp 222°; mod sol in chlf, benz, acet & glc 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, p 367) & Ref 10)

4,6,4',6'-Tetranitro-3,3'-dimethyl-diphenylsulfide, Ir-yel prls (from xylene), mp 189–90°; sol in hot alc, acet & xylene; diff 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, p 367); Refs 9 & 12

**Bis(2,6-dinitro-4-toly)-sulfide, yel crys (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 tетranitro derivs were reported

**Hexanitri-l-ditolylsulfide or Bis(2,4,6-trinitro-3-toly)-sulfide, H₃C₆H₄(NO₂)₂S₂C₆H₄(NO₂)₂CH₃; mw 484.32, N 17.35%; crys (from ac acet ac), mp 170°;**
Di(tolyl) - triazenes and Derivatives

**Di( or Bis) (tolyl) - triazenes**

H₃C₂H₄-NH: N=N-C₆H₄.CH₃, mw 225.28, N 18.65%, exists in six isomers:

1,3,5-Di-(o-tolyl) - triazene, yel nldls (from petr eth), mp 51°, It was first prepd in 1887 by Fischer & Wimmer (Ref 1, p703 & Ref 2) by reaction of 2 moles o-toluidine, 3 molesaq HCl & 1 mole NaNO₂ 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

1,3,5-Di-(m-tolyl) - triazene, 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)

2,4'-Di-(o, m'-tolyl) - triazene, pure yel crystls (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)

1,3,5-Di-(p-tolyl) - triazene, lt-yel ndls (from petr eth) or lt-yel, was prepd by reaction of o-toluenediazonium chloride & 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-toluenediazonium chloride & p-toluidine hydrochloride in the presence of Na acetate or Na dicarbonate (Ref 1, p708)

**Dinitro-ditolyl-triazene or Dinitro-dimethyl-diazoaminobenzene,**

H₃C₂H₄(NO₂)₂-NH: N=N-C₆H₄(NO₂).CH₃; mw 315.28, N 22.22%. Four isomers are found in Beil:

5,5'-Dinitro-2,2'-dimethyl-diazoaminobenzene, yel ndls (from alc or glc acet ac), mp 212° (dec); soln in chlf, acet & benz; sl soln in alc; insol in w. It was prepd by treating a soln of 4-nitro-2-aminotoluene in glc acet ac at 0° with a soln of Na nitrite (Ref 1, p703 & Ref 3)

4,4'-Dinitro-2,2'-dimethyl-diazoaminobenzene, golden-yel crystls (from acet), mp ca 237°, prepd by treating 5-nitro-2-aminotoluene in glc acet ac with a Na nitrite soln (Ref 1, p704 & Ref 4)

3,3'-Dinitro-2,2'-dimethyl-diazoaminobenzene, yel ndls, mp 191°; prepd similarly as above cmpd (Ref 1, p704 & Ref 5)

2,2'-Dinitro-4,4'-dimethyl-diazoaminobenzene, dk: red-bm ndls (from alc), mp 163°(dec); 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) Nie- 
mentowski, Ber 22, 2565 (1889)  
3) Witt et al, 
Ber 25, 3155 (1892)  
4) Noeltling, Ber 37, 
2583 (1904)  
5) Meunier, Bull Fr [3] 31, 
641 (1904)  
6) Not found in later Refs thru 1966

**NOTE:** No higher nitrat derivs were found in 
the literature thru 1966

**Di(toly)-trimethylene-diamine.** See Bis- 
(methylphenylamino)-propane in Vol 2, p 
B151-L

**N,N'-Di(toly) -ureas and Derivatives**

Di(or Bis)(toly)-ureas,  
CH₂₃-C₆H₄-NH.CO.NH-C₆H₄.CH₂, mw 240.29, 
N 11.66%; exists in four isomers:  
N,N'-Di-o-toly-urea, ndls, mp 255–56°;  
insol in w; sl sol in hot alc (Ref 1)  
N,N'-Di-m-toly-urea, ndls, mp 223–25°;  
insol in w; sol in hot alc (Ref 2)  
N,N'-Di-p-toly-urea, crystals, mp 266–68°;  
insol in w; sl sol in alc & eth (Ref 3)

Acctg to Davis (Ref 5), 40 parts of 
para isomer dissolved in benz are required 
for complete gelatinization of 100 parts of 
Pyocellulose  
N-o-Toly-N'-p-toly-urea, ndls (from alc), 
mp 263–64°; v sl sol in hot alc (Ref 4)

Other props & methods of prep 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-ditoly-urea; Bis(nitrotoly)-urea;  
Bis(nitromethylphenyl)-urea or Dinitro- 
dimethylphenyl-urea,**  
CH₂₃-C₆H₄(NO₂)₃.NH.CO.NH-C₆H₄(NO₂)₃.CH₂; 
mw 330.29, N 16.96%. Several isomers exist:  
N,N'-Bis(5-nitro-2-methylphenyl)-urea, ndls, 
(from glc acet ac), mp 300–05° (sublimation);  
insol in benz, eth & chlf; sl sol in alc; sol 
in glc 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 glc acet ac), mp 305–10° (sublimation);  
insol in w, eth, chlf & benz; sl sol in alc; 
sol in glc 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, ndls 
(from glc 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, p1004–05 & Ref 2, 
p661)

Refs: 1) Beil 12, 846, 847, 999, 1004  
2) Vitteret, Bull Fr [3], 21, 659, 661, 662, 
663 (1899)  
3) Not found in later Refs thru 1966

**Tetranitro, C₁₈H₁₅N₄O₈, and Hexanitro, 
C₁₈H₁₅N₆O₁₂, derivs of Di(toly)-urea were not 
found in the literature thru 1966**

**Di(triaxyacyclohexylmethyl)-ether.** See 
Bis(1,3,5-triaza-3,5-dinitro-cyclohexylmethyl)- 
ether in Vol 2, pB159-L

**Ditriazolone (called Di[1.2.4-triazolyl]- 
(3.3'), in Gerl. See Bis(b-sym or b-1,2,4- 
triazole) in Vol 2, pB159-R

**3,3'-Ditriazolyl-formamide.** See N,N'- 
Bis(s-triazol-3-yl)-formamide in Vol 2, 
pB159-R

**Ditriazo-mesidine.** See 2-Amino-4,6- 
diazidomesitylene in Vol 1, p A224-L, 
under AMINOMESITYLENES AND DERI- 
VATIVES
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 prep were not described because the ADL reports listed here as Refs 2 & 3 were classified at that time. They are now declassified

\[
\begin{align*}
\text{CH}_3\text{CH} & \quad \text{CH}(	ext{CH}_3)\text{CH} \\
\text{CH}(	ext{CH}_3)\text{CH} & \quad \text{CO} \quad \text{CO} \\
\end{align*}
\]

MW 546.72, N 5.12%; white crystals (from ethyl), mp 128°C (dec); sol in eth & acet; insol in w.

It was prepd by treating 2,4,6-tri-isopropylacetophenone in glacial acet acid with concd nitric acid & aq Na nitrite soln at 60°C (Refs 2 & 3).


Di(2,4,6-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 prep were not described because the ADL rept listed here as Ref 2, was classified

\[
\begin{align*}
\text{CH}_3\text{C}_6\text{H}_2\text{CO} \quad \text{C}_6\text{H}_2\text{CO} \quad \text{CH}_3\text{C}_6\text{H}_2\text{CO} \\
\end{align*}
\]

MW 378.11, N 7.40%; yellow needles (from ethyl), mp 141-45°C; prepd by warming 2,4,6-trimethylacetophenone with concd nitric acid (Refs 1 & 2).

Di(3,5-dinitro-2,4,6-trimethylbenzoyl)-furoxan.

\[
\begin{align*}
\text{O}_2\text{N} & \quad \text{CH}_3 \\
\text{C}_6\text{H}_2\text{CO} & \quad \text{C}_6\text{H}_2\text{CO} \\
\end{align*}
\]

MW 558.41, N 15.05%; white crystals (from chloroform); mp 235°C; sol in nitrobenzene, acet & 2-nitropropane; mod sol in chloroform; sol in acetic & methanol; very soluble in ethyl, glacial acetic acid & concd nitric acid; insol in water. Its heat of combustion is 2655 kcal/mol at constant vol with water liq.

The parent compd is nitrated with fuming nitric acid (d 1.52) below 0°C & sulfuric acid-water soln added dropwise at -5°C, and the reaction mixture poured into ice water. The yield was 81%.


Di(or Bis) (2,4,6-trimethylpyridino)-diazido-copper (II), [Cu(C$_6$H$_5$N)$_2$]$^+$[N$_3$]$^-$, MW 389.94, N 28.74%; greenish-brown crystals, mp - explodes on heating to 198-202°C or when dropped on metallic block preheated to 220-250°C does not explode from impact. It can be prepd by dissolving Cu azide in trimethylpyridine soln, or by adding Na$_3$N$_2$ to the Cu$^{II}$ salt of trimethylpyridine (Ref 2).

Refs: 1) Beil, not found 2) A. Cinarus & M. Straumanis, J PraktChem 162, 312(1943) & CA 88, 1969(1944).

Di(2,4,6-trimethylbenzoyl)-urea. See under Di(2,4,6-trimethylbenzoyl)-furoxan.

Di(or Bis) (trinitrobutyl)-urea. See under Di-(trinitrobutyl)-urea in this Vol.

Di(2,2,2-trinitroethyl)-amine. See under Diethyl-amine in this Vol.
1,2-Di(or Bis) (trinitroethylamo)-benzene. See under 1,2-Di(ethylamo)-benzene in this Vol.

1,4-Di(or Bis)[N(2',2',2',2',2',2'-trinitroethyl)-carboxamide]-piperazine. See under Di-ethyl-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 Diethyl-nitramine 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)-ethylenedinitramine or N,N'-Di(or Bis)-(2,4,6-trinitrophenyl-nitramino)-ethane (Code named Bitetral, Ditetral or Octyl). See under Bis(anilino)-ethane in Vol 2, p B131-R.

1,3-Di(or Bis)(2',4',6'-trinitrophosphoryl-propylene)dinitramine; 1,3-Di(or Bis)-2',4',6'-trinitrophosphoryl-nitramino)-propene or N,N'-[Hexanitrodiphenyl] -propylene-1,3-dinitramine (Code named Methylene Ditetral). See 1,3-Bis(2',4',6'-trinitro-N-nitroanilino)-propene in Vol 2, p B133-L.

Di(trinitrophosphoryl)-urea. See N,N'-Bis-(2,4,6-trinitrophosphoryl)-urea or Dipicryl-urea in Vol 2 of Encycl, p B156-L.

Dittmar 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 Nuponset, Massachusetts. He invented, before coming to the US, Dualine and some other expls, listed under next item.

After Dittmar's 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, NS 3.2, sugar 8.4, Na nitrate 31.2, NC 23.4 & unaccounted 0.6%. b) NG 30.2, NS 4.0, sugar 8.9, Na nitrate 37.9, NC with charcoal 19.3 & unaccounted 0.7% (Ref, p345).

Titan. An expl prep by impregnating vegetable fibers with a soln of sugar and mannite in amylene, C$_5$H$_{10}$O. Amylene is a colorless transparent liquid obtd by dehydration of anilic alcohol (Ref, p768) and Xylaglodine. An expl prep by nitrating a mixture of glycetin, starch, cellulose and mannite (Ref, p812). Ref: Daniel (1902), 208, 345, 768 & 812.

Di(trifluoromethylthio)-fluorobutane or Bis(tri-fluoromethylthio)-fluorobutane, CF$_3$SCF$_2$–CF$_3$SCF$_3$; mw 302.17; liq, bp 80–81°C; 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 $K_2U_2O_7 \cdot 3H_2O$; 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, KClO$_4$ 4, dextrin 5 & Amm hydrogen fluoride (NH$_4$HF$_2$) 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,**

\[
\begin{align*}
\text{NH}_2\text{NH} & \quad \text{NH}_2\text{NH} \\
\text{CO} & \quad \text{CO}
\end{align*}
\]

; 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-Diureylene-butan.** German name for the compd listed in foreign literature as 2.2'.

Dixo-4.3-dimethyl-hexahydro-[imidazolo-4,5-imidazol] β,γ and 7.8-Dimethyl-acetylenediurein,

\[
\begin{align*}
\text{OC} & \quad \text{NH}_2\text{C(Ch)}_3\text{NH} & \quad \text{CO} \\
\text{NH}_2\text{C(Ch)}_3\text{NH} & \quad - \text{mw 170.17} \\
\text{N 32.93%}; \text{ndls or pilts (from water)}; \text{mp} - \text{dec 350–55° with frothing; sol in hot w; insol in eth, chl & benz & very dif sol in boiling alc. It was prepd by Franchimont & Klobbie (Refs 1 & 3) by allowing to stand diacetyl & urea in water. It was also prepd by Biltz (Refs 1 & 4)}
\end{align*}
\]

**Dinitro-β,γ-diureylene-butan, called in Beil 1,3-Dinitro-7,8-dimethyl-acetylenediurein,**

\[
\begin{align*}
\text{OC} & \quad \text{NH}_2\text{C(Ch)}_3\text{N(NO)}_2 & \quad \text{CO} \\
\text{NH}_2\text{C(Ch)}_3\text{N(NO)}_2 & \quad - \text{mw 260.17, N 32.31%}; \text{colorless crysts, mp – darkens at 230°; diffic sol in w & alc; nearly insol in eth, chl & benz. It was prepd by Franchimont & Klobbie (Refs 2 & 3) by allowing to stand 1 part of β,γ diureylene-butan with 5 parts abs nitric acid}
\end{align*}
\]

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 255-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)

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

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 mix in Diver's soln. The Davis process produces mixts which will propagate satisfactorily in small-diam charges. They are soft & pliable and also propagate in regular dynamite cartridges. However, the mixts have no moisture resitance and are not easily waterproofed. Hence, they require watertight containers
Refs: 1) BritP 15852(1905) 2) C.O.


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
Divine's Explosives. American chemist, S.R. Divine, patented in Great Britain 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, 2403991bs of Rack-a-rock, along with 423311bs of Dynamite, were exploded for this purpose in a single blast. Divine's expl was prep'd 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" or "Break-hills" was in the form of a paste consisting of K chlorate 23.5 and NB 76.5%. It was prep'd just before use and loaded into paraffined paper cartridges

Other Divine expls listed in Ref 7 are:

1) K chlorate 83.4, NB 8.3 & petroleum oil 8.3%
2) K chlorate 87.5 & kerosene 12.5%
3) 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 railroads. 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 "explosivo della Rendelck Powder Co" (1904): K chlorate 80, resin 18, asphalt 1 & nitrated wood pulp 1%

Gody (Ref 3, p576) listed as "explosive 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 compds, 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, p553)

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


Divine's Explosive Rack-a-Rock. See in previous item

Divinyl or Divinyle See 1,3-Butadiene in Vol 2 of Encycl, p B365-R

Divinylacetylene, DVA or 1,5-Hexadien-3-yne, H_2:C:CH.C:CH:CH:CH_3; mw 78.11; oily liq, sp gr 0.785, bp ~ decompt at 83.5°. It can be prep'd by passing acetylene into ammoniacal cuprous chloride. It is a basis for synthetic butyl rubber (Refts 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-alanaphthylamine, 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, H₂C:CH.C:CH, described in Ref 6, p 1210-L.


Divinylbenzene (DVB) or Vinylstyrene,
H₂C:CH.C₆H₄.CH.CH₂; 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*, mw liq, easily polymerized; sp gr 0.9289 at 20°; fr p minus 65.9°, bp 199.5°. It can be prep'd (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 Vinyl benzene, described as styrene in Ref 3, p 1081-L.


Divinyl Ether, Divinyl Oxide (DVO), Vinyl Ether or Ethenoxethene,
H₂C:CH.O:CH.CH₂; mw 70.09; colorless liquid, sp gr 0.773 at 20/20°, bp 28.3°; fl p below minus 22°; n₂D 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 & v sol in eth. It can be prep'd 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, νD 1.4117 at 20°, which burned vigorously and could be detonated by a hammer blow (Ref 4).

DVO is used as an anesthetic (Ref 5).


Dixylyl or Tetramethyl-biphenyl. See Bixylyl in Vol 2, p B164-R.

Dixylylamine and Derivatives

Sym-Dixylylamine or Tetramethyl-diphenylamine, (CH₃)₂C₆H₄.NH.C₆H₄(CH₃)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 prep'd 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 decmp 340–45°; prep'd from 4-azido-xylene, bromo-Zn-ammonia & Amm bromide heated in a tube at 300–310° (Ref 2).

Azido, C₁₈H₁₇N₃, Diazido, C₁₈H₁₇N₃, and Dimitro, C₁₈H₁₇N₃O₄, derivs of Dixinylamine were not found in the literature thru 1966.

2,4,6-Trinitro-3,5,3',5'-tetramethyl-diphenylamine, (CH₃)₂C₆(NO)₂₃.NH.C₆H₄(CH₃)₂; mw 360.32. N 15.55%; pale crs, mp 209°; sl sol in alc; prep'd by heating 5-bromo-2,4,6-trinitro-m-xylene with m-xylidine in alc at 150–60° (Refs 3 & 4).
Tetranitrodixylamine, C₁₄H₁₃N₃O₈, not found in the literature thru 1966

2,4,6,2',4','6'-sym-Hexanitroxylyamine or 3,5,3',5'-Tetramethyl-2,4,6,2',4','6'-hexanitrodiphenylamine,
\[ \text{O}_2\text{N.C} \]
\[ \text{C(CH₃)} \text{C(NO}_2\text{)} \]
\[ \text{C} \text{NH.C} \text{C(NO}_2\text{)} \text{C(CH₃)} \]
\[ \text{C(NO}_2\text{)} \text{C(CH₃)} \text{C(NO}_2\text{)} \text{C(CH₃)} \]

mw 493.32, N 19.80%; yel crs (from glc acet ac); mp 222° or bm-yel (from alc), mp 231°(dec) (Ref 5). It can be prepd by treating 2,4,6-trinitro-3,5,3',5'-tetramethyl-diphenylamine with nitric & sulfuric acids (Refs 3, 4 & 5). It forms crst salts with NH₄, K, Rb & Cs (Ref 5)

This cmpd is expt, although no expl props are reported.


DNA. Abbr for Dinitroaniline. See Vol 1, p448-L

DNAs. Our abbr for Dinitroanisole. See Vol 1, p448-L

DNB. Abbr for Dinitrobenzene. See Vol 2, p B46-L

DNDCP. Abbr for 1,3-Dinitro-1,3-diazo-cyclopentane, known also as 1,3-Dinitro-imidazolidine. Described under Imidazolidine

DNF. Abbr for Dinitroethylbenzene

DNEU. Abbr for Dinitroethyleurea

DNG. Abbr for Dinitroglycerin

DNM. Abbr for Dinitronaphthalene

DNP. Abbr for 4,4-Dinitropimelic Acid, HOOC.CH₂.CH₂.C(NO₂)₂.CH₂.CH₂.CO.OH. 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)

DNP. Designation for Bisdinitrophenylformate

DNPS. Designation for Bisdinitropropylsuccinate

DNPT or DPT. Abbr for Dinitropentamethylenetetramine; also called 3,7-Dinitrobicyclo-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-cyclooctane; called in CA 3,7-Dinitro-1,3,5,7-tetrazaboricyclo[3.3.1]nonane. It is described under 1,5-Endomethylene-1,3,5,7-tetraza-2,4,6,8-cyclooctane 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-cyclooctane

DNT. Abbr for Dinitrotoluene

DNT/HV₃. A Fr expl mixt which will be described as HV₂ (Explosif)

Dobgeröt. 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

Dodecanitrocellulose. 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 prep'd 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 un-oiled 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 "Milite 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 large 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 Urbafski (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, ComptRendCongr-InternChimInd 31 7 Liège, 1958 (Publ in IndChimBelge (Suppl), 231–39 (1959) (Exp props of AN-fuel oil system. Max deton was obtd with 6% oil content, 1300ft/sec)
9) Ibid, 16(8), 413–22(1965) & CA 64, 7960 (1966) (Underground uses for AN-combustible oil expls)

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 Yaremko & 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 detm 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:

$$V_g = \frac{V_b(P - w) \times 273 \times 1000}{760 \times T \times a}$$ liters/kg

where:  
$$V_g = \text{Volume of gases formed on expln minus vol of water vapor, calc'd to atm press and temp } 0^\circ C$$  
$$V_b = \text{Volume of bomb in liters}$$  
$$P = \text{abs press after expln (in mm) minus residual press before the expln}$$  
$$w = \text{tension of water vapor saturating the atmos at absolut temp } T^\circ$$  
$$T = \text{absol temp of bomb's body at the moment of expln}$$  
$$a = \text{weight of expl sample (100g)}$$

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 absorbed water is b, wt of sample a, the vol of water vapor at atm pressure and 0°C can be calc'd from the formula:

$$V_{H_2O} = \frac{22.4 \times b \times 100}{18 \times a}$$ liters/kg

and total specific volume:

$$V = V_g + V_{H_2O}$$ 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>
<thead>
<tr>
<th>Explosive</th>
<th>Density g/cc</th>
<th>Specific Volume of Gases in l/kg</th>
<th>H$_2$O liq</th>
<th>H$_2$O gas</th>
</tr>
</thead>
<tbody>
<tr>
<td>Troil (TNT)</td>
<td>1.5</td>
<td>610</td>
<td>750</td>
<td></td>
</tr>
<tr>
<td>Tetryl</td>
<td>1.55</td>
<td>620</td>
<td>740</td>
<td></td>
</tr>
<tr>
<td>Ghekosoghen (RDX)</td>
<td>1.5</td>
<td>700</td>
<td>890</td>
<td></td>
</tr>
<tr>
<td>TEN (PETN)</td>
<td>1.65</td>
<td>550</td>
<td>790</td>
<td></td>
</tr>
<tr>
<td>Ammonal 80/20</td>
<td>0.7</td>
<td>300</td>
<td>720</td>
<td></td>
</tr>
<tr>
<td>(80/20 Amatal)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>


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

Donor (1901). A Sprengel-type expl patented in Russia by Fielder. It consisted of 80 parts of pulverized oxidizer (mixture of K chlorate 70 & K permanganate 30%) and 20 pts of a fuel oil (mixture 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 Fällung 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 yellow 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 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 (Gelatn 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 nitrocmpds 3.0 & woodmeal 9.0% (Ref 1, p 94 and Ref 4, p 47-R)

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, Tmnzl 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 3545°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, ZentrGewerbefhgUnfallverhürt 30, 97 (1943) & CA 40, 4523 (1946) (Freezing of Donarit)  
3) Weichelt (1953), 37–8 & 375
4) PATR 2510 (1958), pp 38 & 47
5) Giua, Trattato 6(I) (1959), 373  6) Urbanský 3 (1967), 263 (Mining expl used in ammo during WWI)

Donarite. A Spanish, non-permissible coal mining expl listed in Vol 3 of Encycl, p C422-L under COMMERCIAL OR INDUSTRIAL EXPLOSIVES


Dope, Balanced. See next item

Dope (Base or Absorbent). In the prep 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 transportation 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 Subdynamite 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 expls.

For expls containing only a small amount 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 containing NG (or NG + NGc) and powdery or coarsely ground dopers are known as Powdery (or Straight) Dynamites.

If a small amount of soluble (low N content) NC (such as collodion cotton) is added to Dynamite, the NG (or NG + NGc) gelatinizes it and the resulting mix is a more or less viscous gel. Such expls are known as Gelatin Dynamites. With larger amounts of NC (7-10%), the resulting gel is so tough that no exudation can take place even without addition of absorbing dopers. Such expls are very powerful and are known as Blasting Gelatins (See Vol 2 of Encycl, p B211-R). With Dynamites containing 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 containing no NC.

The term "dope" also includes the antacidic 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 prepared by heating a mix of sulfur & rosin until the foaming ceased and then pulverizing the resulting solid after its cooling.

It should be noted that in selecting dopers, one must remember that not only the comp 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 dopers 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 CO₂ (Ref 6, p 9).


Doppelzünder (Double Igniter). A German double igniter for acoustical 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 (1943), 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 ob-
served 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 Doppler principle is applied now to electromagnetic radiations in connection with radio 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 between the missile and any point on the ground can be accurately measured by means of a Doppler radio system. By integrating the velocity values from the instant of takeoff, position vectors can be deduced 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 Doppler system transmits a sawtooth frequency-modulated (FM) wave, both the velocity and the instantaneous distance between the missile and the ground station can be determined 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).

Doppler 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 chisel. 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 Doppler Homing Systems, which belong to the active group, may be subdivided into two systems:

a) FM-CW Doppler 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 Doppler shift (Ref 2, pp 451-52)

b) Pulse Doppler 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 between 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 instrument 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)


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 2200–2400 ft/sec and maximum range of 29 miles. Wt of exps was 2000 lbs, wt of propellant 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


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 prep'n a chge of BkPdr, intended to serve as a propln, 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 comps 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)


**Dormont 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, 486 (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.


**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 (Detonationsfähigkeits) 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 0.5g LA/LSt mixture

e) Gallery Tests were conducted with methane-air mixtures and with coal dust

DOUBLE-BASE AND TRIPLE-BASE PROPELLANTS. A description of double-base propellants 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 C53-L to C541-R).

Some ball powders, described in Vol 2, pp B11-L to B16-L, can be made double-base. The description did not include the German propellants 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 propellants containing NC + NG + NGu are described in Vol 2 of Encycl, pp C34 (Table V) and p C37-L.

We are not giving here the description of the German double-base propellants containing NG because there was nothing unusual about them and many were similar to US propellants.

The description given below will be devoted to the problem of gun barrel erosion caused by NG-NG propellants and to the development in Germany, before WWII, of a new class of propellants which reduced the erosion to such an extent that the serviceable life of a barrel increased 4-5 times in comparison with NG-NG propellants.

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 unserviceable, 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 done in Germany that the serviceable barrel life of rapid firing AA gun was only 1700 firings when NC-NG propellant of calorific value of 950 kcal/kg was used, and the muzzle velocity was 3300 ft/sec. When the NG content was reduced in order to obtain the calorific value of 820 kcal/kg, the number of serviceable firings could be increased to 3500. This meant that reduction of 130 kcal/kg in calorific value, doubled (approximately) the life of the barrel. No further improvement in life of AA guns could be obtained when using NC-NG propellants (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 propellants 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 propellant known as "G" Pulver ("G" stands for the 1st letter of the name Gallwitz) by replacing in double-base propellants NG with DEGDN (Diethylene glycol nitrate), it was assumed that it should prolong the barrel life to 2 x 3500 = 7000 firings. This value was called on the assumption that each 130 kcal/kg reduction of cal value should double the life of the barrel, as it was doubled in the case of reduction from 950 to 820 kcal/kg. This theory worked in the case of NG propellants, but did not seem to be applicable when NG was replaced with DEGDN, since 17000 firings were not obtained instead of 7000 anticipated (Refs 1 & 2 and Ref 4, p Ger 43).

One of the earliest "G" propellants consisted of: NC (blend of coal & insol NC's giving an average N content of 12.2%) 61.53, commercial DEGDN 26.37, EtCenZ 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 & 690 kcal/kg. This propellant was used in tubular shape during WWII in 100mm Army Gun designated as K18 (Kanon 1918) (Ref 4, pp Ger 71 & Ger 145).
A flaked propellant for use in various howitzers contained NC (13.0% N) 54.40, DEGDN 44.50, Acaride 0.50, graphite 0.05, MgO 0.05 & K sulfate 0.50% (Ref 4, p Ger 145). A tubular propellant for use in 88mm Army AA Gun contained: NC (12.0% N) 64.08, DEGDN 27.47, EtCentr 5.35, phthalate 0.90, vaselate 1.85, graphite 0.10 & MgO 0.25%; calorific value 730 kcal/kg (Ref 4, p Ger 145). A tubular propellant for various Army guns: NC (12.0% N) 60.55, DEGDN 25.95, EtCentr 3.75, hydrocellulose 3.00, DNT 4.00, α-MNN 2.50, graphite 0.10 & MgO 0.15%; calorific value 730 kcal/kg (Ref 4, p 145). A tubular propellant 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 730 kcal/kg (Ref 4, p 145). A universal propellant 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 900 kcal/kg (Ref 4, p 145).

The commercial DEGDN used in "G" propellants was prepared by nitration of the product known in Germany as "Polyglykol", which was a mixture of DEG (known in Ger as "Dialkylglykol") with small amounts of EG (ethylene glycol), called in Ger Glykol and in English glycol. Polyglykol was a product easily available from non-feed materials.

The advantage of DEGDN over NG included (besides producing much "cooler" propellants) more effective gelatinization for NC’s. This permitted one to prepare more homogeneous propellants 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), 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 propellants during WWII in African Campaign, Gen Gallwitz proposed replacing DEGDN with a less volatile product (only 1.5 times more volatile than NG), TEGDN (Triethylene glycolinitrate), which could be prep'd 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 650 kcal/kg vs 670-690 for DEGDN propellants).

A typical TEGDN propellant 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 650 kcal/kg. Gelatinizing props of TEGDN were found to be similar to those of DEGDN (Reps 1, 2 & 3 and Ref 4, pp Ger 71 & Ger 145).

All "G" propellants were slow-burning and were efficient only in weapons with long barrels, which allowed complete combustion of propellants. 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 combustion of propellant while the shell was still in the barrel (Reps 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 propellants. They are similar to the ones described above.

The Gudolpulver, described in Reps 1, 2 & 3 and in Ref 4, p Ger 81 and mentioned above, was a "G" Pulver in which a large amount of NG (Nitroglycerine) was incorporated. It was actually a "triple-base propellant".

As was mentioned above "G" propellants were unsuitable for use in short-barrel weapons. In order to obtain satisfactory results with such weapons (without resorting to NC-NG propellants, containing 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 flashiness. Such a propellant was obtb in 1937 on incorporating a large amount 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 amounts of NGu (which is not a gelatinizer for NC and is not gelatinized by nitrated glycols) did not produce brittle propellants. In order to have a propellant of good performance, the crystals of NGu should be short and fine and evenly distributed throughout the mass of propellant. Good blending of Gudolpulver ingredients was achieved by kneading in a Werner-Pfeifer apparatus the required amount of NC with nitrated glycol until a uniform jelly was obtb. 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 (Ref 1 & 2 and Ref 4, p Ger 81).

Following are combs of some Gudol propellants:

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, EthPhUret 5.00, K nitrate 4.00, graphite 0.10 & MgO 0.25%; caloric value 730 kcal/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, PhEthUret 1.50, hydrocellulose 4.00, K nitrate 4.00, Acardite 0.40, graphite 0.10 & MgO 0.15%; caloric value 720 kcal/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, EthPhUret 4.50, K sulfate 5.00, MgO 0.25 & graphite 0.10%; caloric 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 propellants 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.

Propellants containing 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 propellant, the breech as well as muzzle flash were practically eliminated and so was the smoke. The erosion of gun barrel was very low (Ref 1, 2 & 3 and Ref 4, p Ger 81)

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 BALLISTICITE, Vol 2 under CANNON PROPELLANT and Vol 3 under CORDITE.


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 det 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 comp & 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 some 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.
Double-Effect Powders (Poudres à double effet, in Fr). A series of exps 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 oxidation in cases of high fluidity of tar.

Pyrodialites were exps 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) Force (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) Candill (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) Fedonoff & Clift 4 (1944), p29

Double Lead Salt of Formic and Nitric Acids, Pb(NO₃)₂(OOCH), "active"; and Pb(NO₃)₂–[Pb(OOCH)₂]₉, "inactive". These two samples were investigated for possible military use as suggested by their originator who also published 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 Picatiny 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)


Double Salt of Cesium Dichromate and Cesium Decahydratedecaborate. 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 comps, which ignite reliably and uniformly and has excellent resistance to impact and to static electricity. The compd has the formula (Cs₂B₁₂H₁₈O₁₂C₉Cr₂O₇. Triethylammonium decahydratedecaborate 3.2, and 2.94 K₂Cr₂O₇ were dissolved in 50 parts water. Five parts 25% nq 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 Cs₂Cr₂O₇ and Cs decahydratedecaborate,
was formed. The product was recrystd 3 times from water, 260-70°C (with discoloration). The product flashed at 290°C. 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 Pentacythritol Tetranitrate pressed at 200 lbs. Above this base charge was loaded 3 grains Pb(NO₃)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 sep 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.

While the soln was well stirred at that temp, 15ml M Pb(NO₃)₂, dild with water to a vol of 50ml, was added dropwise in 15-25 mins to ppt the crystal 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°C. 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 stphnate 16.1 and 1 24.0g, resp. The proprietary name of double salt is Stobonate. 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 Stobonate 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, 15450 (1966) as follows:

Procedures are given for the prep of a new series of primary explosives useful in compns for ammunition primers. Thus, 9g NaOH in 50cc water is added to 25g 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(C₆H₃N₃O₇)₂·2Pb(OH)₂·Pb(C₂H₅O₂)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(C₆H₃N₃O₇)₂·2Pb(OH)₂·2Pb(C₂H₅O₂)₂, 276°C
- Tribasic lead picrate-lead acetate, Pb(C₆H₃N₃O₇)₂·3Pb(OH)₂·3Pb(C₂H₅O₂)₂, 277°C

Monobasic lead picrate-lead formate, Pb(C₆H₃N₃O₇)₂·Pb(OH)₂·Pb(HCO₂)₂, 258°C

Dibasic lead picrate-lead formate, Pb(C₆H₃N₃O₇)₂·2Pb(OH)₂·2Pb(HCO₂)₂, 277°C

Double Salt of Lead Nitroaminotetrozole and Lead Stphnate. 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 (1) are formed by

\[ \begin{align*}
  &\text{O}_2\text{N} \\
  &\text{Pb} \\
  &\text{N} \\
  &\text{Pb} \\
  &\text{N} \\
  &\text{Pb} \\
  &\text{N} \\
  &\text{O}_2\text{N} \\
\end{align*} \]

(1)

as a soln of Pb salt, desirably in excess, with a soln of an alkali or alk earth salt of Stphnic Acid and of Nitroaminotetrozole. For example, 1M aq Mg stphnate and K Nitroaminotetrozole were prepd, the solns having, resp pt 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 Stphnic Acid. The soln was heated in a water bath to a temp of 75-80°C.
Trisbasic lead picrate-lead formate,
Pb(C₆H₂N₃O₇)₂·3Pb(OH)₂·3Pb(HCO₃)₂, 247°C
Monobasic lead picrate-lead propionate,
Pb(C₆H₂N₃O₇)₂·Pb(OH)₂·Pb(C₃H₅O₂)₂, 291°C
Dibasic lead picrate-lead propionate,
Pb(C₆H₂N₃O₇)₂·2Pb(OH)₂·2Pb(C₃H₅O₂)₂, 265°C
Trisbasic lead picrate-lead propionate,
Pb(C₆H₂N₃O₇)₂·3Pb(OH)₂·3Pb(C₃H₅O₂)₂, 255°C
Monobasic lead picrate-lead lactate,
Pb(C₆H₂N₃O₇)₂·Pb(OH)₂·Pb(C₃H₅O₃)₂, 276°C
Dibasic lead picrate-lead lactate,
Pb(C₆H₂N₃O₇)₂·2Pb(OH)₂·2Pb(C₃H₅O₃)₂, 263°C
Monobasic lead picrate-lead acrylate,
Pb(C₆H₂N₃O₇)₂·Pb(OH)₂·Pb(C₃H₅O₂)₂, 268°C
Dibasic lead picrate-lead acrylate,
Pb(C₆H₂N₃O₇)₂·2Pb(OH)₂·2Pb(C₃H₅O₂)₂, 261°C
Trisbasic lead picrate-lead methacrylate,
Pb(C₆H₂N₃O₇)₂·3Pb(OH)₂·3Pb(C₃H₅O₂)₂, 254°C
Monobasic lead picrate-lead methacrylate,
Pb(C₆H₂N₃O₇)₂·Pb(OH)₂·Pb(C₃H₅O₂)₂, 268°C
Dibasic lead picrate-lead methacrylate,
Pb(C₆H₂N₃O₇)₂·2Pb(OH)₂·2Pb(C₃H₅O₂)₂, 278°C
Trisbasic lead picrate-lead methacrylate,
Pb(C₆H₂N₃O₇)₂·3Pb(OH)₂·3Pb(C₃H₅O₂)₂, 283°C
Monobasic lead picrate-lead aminoacrylate,
Pb(C₆H₂N₃O₇)₂·Pb(OH)₂·Pb(C₃H₅NO₂)₂, 274°C
Dibasic lead picrate-lead aminoacrylate,
Pb(C₆H₂N₃O₇)₂·2Pb(OH)₂·2Pb(C₃H₅NO₂)₂, 216°C
Trisbasic lead picrate-lead aminoacrylate,
Pb(C₆H₂N₃O₇)₂·3Pb(OH)₂·3Pb(C₃H₅NO₂)₂, 291°C

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 mèche enroulée", has been manuf from steel and rolled sheet iron by the Forges et Ateliers de Construction Electrique de Jeumont, Paris, 8°

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 on in its target by infrared seeking
device in its nose


**Dowmetal.** Trademark of Dow Chemical Co, Midland, Mich, applied to a series of light and tough metalscontg 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 (Dichoresene). 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 (Dinitropentamethylene-tetramine) 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-Endo-ethylene-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 (reusable) 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


**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: KNO₃ 44.5, NG 35.5, Nitro-cotton 2.5, woodmeal & charcoal 12 & vaseline 5.5%  
Ref: Marshall, Dictr (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 GB Britain before WWI 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) Steitbacher (1948), 129  2) Steitbacher (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  
Note: Giua, Trattato 6(I) (1959), p 401, lists, among others, the following expls developed by Alvisi: a) Craititi (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 C555-L, as Cremonita together with a Cremonita contg AN 80 & cannel coal 20% and d) Mantaniti – mixts of perchlorate with sulfur & carbon. No Dranite was listed by Giua

Dreadnaught Powder. A permissible expl: AN 75–77, 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 enabled for this invention  

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 peller 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-gn 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 39 3) Night Drift Signal AN-Mk 4 and 4) Night Drift Signal AN-Mk 5

Current US military ground smoke & marine signals are described in Ref 4


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, p 605) 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.


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 exch 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 mar ring 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) Baratol 6 Cyclotol 7) 50/50 & 10/90 Pentolite and 1) 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)


Drillingspulver. Get short tubular powder for howitzers (Haubitze) such as the 10cm Haubitze

Ref: H. Brunswig, "Das Rauchlose Pulver", W. DeGruyter, Berlin (1926), p 131

Drip Oil (Dinitrotoluene oil, Liquid DNT, or DNT Low Oil) (Tropol or Dinitrotropol in Ger). A black tarry oil consisting of a mix 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 nitrate 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 crystals) 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 formerly used in the US and other countries when crude TNT (known as Troil) was purified by washing with volatile solvents, such as alcohol or xylene. This took place during WWII 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 distill 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 mix 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 bewt WWII 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 comps 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, Naoum (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 bm-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 crystals 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 I (1917), 259 2) Colver (1918), 178 3) Naoûm, 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, Landaus, 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 Ochard (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).

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.

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 propellant 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 propellant gases on the base of the projectile. This engraving is accomplished partly by cutting and partly by forming.

In recoiless ammunition, the force required to accomplish engraving of the band would interfere with accomplishment of the recoiless 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 51-R & Ref 6, p 35)

There is also the so-called welded overlap 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 51-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 Encyc, 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 devided 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 accord 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).


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).


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 det 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 "Alumund" 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.


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:

\[
M = 1000 + \frac{16000}{\sqrt{7.3 + W/5 - 1}}
\]

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 50000 lb/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 calculated from the formula:

\[
M = \frac{2\pi S}{W_m(D-t) + 2W_0(D-2t)}
\]

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 set forward of filler and b) longitudinal compressive stress in bomb walls due to set forward 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 super-imposed on crushed stone placed on firm ground. SAP-bombs weighing less than 1000 lbs are dropped on a 2-ft thick surface, those over 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

Ref: 1) Hayes (1938), 612–13  
2) Ohart (1946), 208–11  

**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 obt'd if a given number of primers are fired at heights between 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 dctermined 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 dctermined 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 30mm), 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 ratchet 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 explosive substances](image)
THE DEVICE WITH CENTRING RING AND DETAILS

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 will be found in the Refs and Addnl Refs cited below

(One type of Drop-Weight Impact Machine developed in the US during WWII)
3) Ohart (1946), 48-9, 96, 131-32, 210 & 380-81
5) Glossary of Ord (1959), 257 (Setback force) 6) F.D. Altman, "Drop Test of Large Castings of
Experimental Explosive PAX-5", Naval-Proofing Ground, 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, Explosiv 9, 4-13, 30-42 (1961) 8) A. Maček, "Sensitivity

Addnl Refs on Drop Tests:
A) J.J. Fox et al, "Sensitivity of High Explosives", OSRD Rept 1288 (Mar 1943)
C) Hercules Powder Co, "Sensitivity of High Explosives", OSRD Rept 4962 (Apr 1945)
Studies with the Drop-Weight Impact Machine", OSRD Rept 5744 (Dec 1945)
F) J.M. Downard, "Sensitivity of High Explosives", OSRD Rept 6627 (Feb 1946)
6629 (Mar 1946)
H) NDRC Summary Tech Rept of Div 8, Vol 1 (1946), 38-41 (Sensitivity & 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 Sensi-
nitiveness 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. Bultinch, "Improved Methods and Techniques of Testing Impact Sensitivity of
Explosives", PATR 2282 (1956)
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)
Ministry of Aviation TIL T-5194 (Oct 1962)
R) M. Kusakabe & N. Ishikawa, Kôyo-Kyôyo-Kyôkaishi 24, No 6, 12-23 (1963)
[Engl translation Picatinny Arsenal Tech Memo TM-1734 (Jan 1966)]
S) C. Boyars, Western States Sect – Combust
Inst Papers WSCI 65-27 (1965) & CA 64,
4852 (1966) (A review of drop-weight sen-
sitivity testing of expls, with 38 refs)
T) A.J. Clear, "Standard Laboratory Pro-
cedures for Determining Sensitivity, Brisance,
and Stability of Explosives", PATR 3278
D 1555

(Dec 1965), pp 2-7, 32-37 (Impact test procedures & appa- ratus)


**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 re-inforced 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


**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 Drozdov), N.F.** (1862-1954). A Rus ballis- tician, former professor of Mikhail-

**Optimum Gun, called in Rus: "Orudie Naibol'-shego Mogushchegora" (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 \((mv_{g}^{2})/2\). Here \(m\) is mass of the projectile and \(v_{g}\) its starting velocity in the barrel.

Accdg to Prof Serebryakov, Drozdov's formula has advantage over the formula of French ballisticians: \((m_{p}v_{p}^{2})/2\), which expresses maximum total work. Here \(p\) is relative part of consumed proplnt


**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 Vereinigte 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 GB, Britain 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.
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 Bament (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.

The modern "wheel mill", such as described by Marshall, Bament, 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 GB Britain, 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, p240 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).


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, pp 976-78 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 taming 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 from a second line to a second separator, receiver and pump. The cake now approaches the dis-
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:

- Dorco 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 prepq 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 manuf 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 58 and reproduced here as Fig 6. The lower part of drum dips into molten material located in a hollow, steam-
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, p.1164-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.

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, pp.347–50) and a rotary, single-drum flaker is shown in Fig 271, p.348

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), pp.980–81, 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, p.381 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, exps and proplnts which are too sensitive for use in projectiles, unless they contain some moisture. For example, Gun-cotton, 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 expls 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). Exps can also be dried by the methods for drying NC described in detail in Vol.3 of Encycl., p D45-L.

For drying of exps at the plant, Olsen patented (Ref 7) the following method: Grains of an exp, 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 exps 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. du Pont 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°C) was circulated thru it in order to pick up volatile solvents (alcohol & ether). This air went 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°C 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°C. 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 amount 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. Arrihat 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-L 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".

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.
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
(Harding & Co)

FIG 13 – ROTARY KILN, REFRACTORY-LINED, FOR ROASTING AND CALCINING. IN SPECIAL CASES, DRYING IS ALSO ACCOMPLISHED
(Harding & 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, pp 1608-12 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 V (1917), 289 (Drying of NC); 294 (Drying of proplts) 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 proplts) 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 nitrate)  
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 explosives)  
10) Riegel, ChemMach (1944), 351–81 (Driers)  
11) D.B. Broughton, IEC 37, 1884–85 (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)  
16) Kirk & Othmer 5 (1950), 232–65 (Drying)  
17) J.R. Harris, "Freezing and Drying", HafnerPubCo, NY, (1952)  
19) S.P. Artyushin, "Dehydration and Dust Collection in Coal Concentration Plants" (in Russian), Ugleoechizdat, Moscow (1956)  
20) M.J. Belin, "Dehydration of the Product of Coal Enrichment" (in Russ), GosNauch-Tekhizdatel, Moscow (1958)  
21) E.L. Holland-Merton, "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 ed 7 (1965), pp 326–78 (Drying)  
24) S. Shemesh, "Producing TNT by Continuous Nitration", Ordnance 54 (1970), p 527

**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

**DSSP.** Abbreviation 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, \((\text{C}_2\text{H}_5)_2\text{SO}_4\), a blister gas, listed in Vol 2 of Encycl, p C169-L. It was known in France as "Rationite"

**DTA.** Abbreviation 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

**Dueline Stamping 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 the compo to have NG 50, partially nitrated sawdust 30 and K nitrate 20%. This expi 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)  

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, naphthalene 7.8 and tar 7.8; Mixture No 2: K picrate 45, anthracene 45 and tar 10%  

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  
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 exercised 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
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 bores 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, ArmedForcesChem 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 prepard 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 obted 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, Hexanbexol or Melampyrit) (Dulcit in Ger), HO.CH₂(CHOH)₄.CH₂.OH;

mw 182.17, colorless monoclinic crystals, d 1.466 at 15/4°C, mp 188.5°C, 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 obted 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, C₆H₂(OH)₂(NO₃)₂,4;

mw 362.17, N 15.47%; prismatic ndls (from alc), mp 120–30°C. It can be prepard by gently heating Dulcitol Hexanitrate (qv) (Reps 1 & 2)

Refs: 1) Beil 1, 547 2) Béchamp, CR 51, 257 (1860) 3) Not found in later refs thru 1966

Dulcitol Pentanitrate, C₆H₄(OH)(ONO₂)₂;

mw 407.17, N 17.20%; ndls (from alc + w), mp – sinters at 71°C & melts at 75°C; has bitter taste; mod sol in alc, eth, acet & benz; sl sol in petr eth; v sl sol in water. It was prepard by gently heating Dulcitol Hexanitrate (qv) in pyridine (Reps 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 orCCI₄), mp 85–86°C]

Dulcitol Hexanitrate (Nitrodulcite), (O₅N)CH₂.[CH(ONO₂)₄].CH₂(ONO₂)₄ or C₆H₄(ONO₂)₄;

mw 452.17, N 18.59%, OB to CO₂ +71%; wh ndls (from alc), d 1.62 mp 94–95°C (Reps 1 & 3); bp 290–95°C at
Dulitz Explosive (1886). Mixtures of K chloride (or other oxidizer) with various quantities of gel prepd by treating NC with nitrobenzene. Eg: 10 parts of K chloride & 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 CO₂, a Trautz-Nierderl gasometer to measure the amt of gas used either in the determination or in the blank test; a gas buret 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
Dümmeberg (Dueenbeget) 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 BkR, which was prepd by the most careful blending of its ingredients. Beginning in 1902 it manuf'd single-base cannon, smokeless prop., known as DRDC-02 (Dümmeberg Röhrenpulver für Kanonen, 1902). During WWII it was the principal Ger plant for the development and production of double-base, solventless prop., both for cannons and rockets. The total area of the plant was ca. 18 sq miles and it employed 7600 in 1942 (mostly foreigners). This plant also prep'd some of the ingredients used in manuf. of prop., but NC, glycol and acids were obtd from Krümmel Fabrik of Dynamit AG. The personnel at Dümmeberg developed, in collaboration with General Udo Gallivitz, the "cool propellants": "G" or "K" Pulver (Ref 3, p Ger 70) which cont'd DEGDN or TEGDN and also "Gudolpulver", which contained NGu in admn. to DEGDN or TEGDN (Ref 3, p Ger 81) (See also "Cool (or Cooled) Propellants" in Vol 3 of Encycl, p C511-R).

Ref: 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 (Dümmeberg 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 "Dannite". 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. 

Ref: 1) Van Gelder & Schlatter (1927), pp 373 & 942 2) Army Ordnance 16, 364 (May–June 1936) (Describes his contributions as developing an impact-testing machine, de-
developing War Dept Factory at Picatinny Arsenal and organizing the Bureau of Explosives for the American Railway Association)

Dunmite, 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

Duplémite. 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

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

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), Eusthèrè Iténé de 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 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 expn 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 col-
laboration 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 through 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, manuf 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, manuf 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), manuf 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 manuf 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 ("Alvite", "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 "Pour- vex" Extra; various prill-fuel formulations such as "Nilit", 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 manufactured 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 "Acu-dor" 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 "Acu-dor" 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, pp15-16 & 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 seis- mograph sledge charges in very deep holes", a "millisecond (MS) delay connector for delay firing of Primacord", "Detaprine" Primers, and electric squibs for military and commercial uses (Ref 6, pp16 & 87-99; also Ref 7, pp6-7).

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 de-

vices 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 Acu-dor Mark V and MS (Millisecond) Delay (Ref 6, pp91) DuPont's "Detacord" (Explosion-Bonded Clad Metal), Ref 6, op 14-15. See also Vol 3 of Encycl, p D96-L.

DuPont's Detacord (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", Strikolos X; DuPont Gelatin, High-Velocity Gelatin, special Gelatin, Toval Gelatin Dynamite, Gelex and Seismograph Types Gelatin (Seismograph 60% High-Velocity Gelatin, ) (Ref 6, pp33-42). Properties of Dynamites are described on pp 73-80.

DuPont's Electric Squib. See Ref 6, pp 94-5 and Ref 7, pp6-7.

DuPont's Explosive Releases are described in Ref 7, pp14, but not listed in alphabetical index of Ref 6.


DuPont's Flexible Linear Shaped Charge (FLSC). See Ref 7, pp8 & 12; not listed in Ref 6.

DuPont's HD5 (High Detonation Pressure) Primers for water gel expls (Ref 6, pp64-7)


DuPont's Nitrometer is described in Vol 1, pp A373-L to A377-L and its illustration is on p A374-L.

DuPont's Permissible, 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, pp 62-3)
**DuPont's Primers, Non-NG.** See Ref 6, pp 63-8
**DuPont's Pyrocore.** See Ref 7, p9
**DuPont's Seismic Prospecting Explosives**, such as Seismograph 60% "High Velocity" Gelatin; Nitrarnons WW, WW-EL, S & S-EL; Seismogel A; Seismex and Seismex PW (Ref 6, pp 358-62) (See also Seismograph Type Explosives, pp 41-2)
**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, pp 90-1
**DuPont's Stripkolex** (Ref 6, p37)
**DuPont's Submarine (Underwater) Blasting and Explosives Used.** See Ref 6, pp 421-27
**DuPont's Trimex Explosives** (Ref 6, p43)
**DuPont's Water Gel Explosives,** such as Tovex Extras and Pourvex Extras (Ref 6, pp 68-71)

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
4) W.S. Dutton, "DuPont in World War II", Armed Forces Chemical Journal 3, No 7, pp 45-38 (1950) & CA 44, 3178 (1950) (Discussion on the manuf of chemicals by DuPont Co)
6) DuPont's Blasters' Handbook (1966), pp 3-20 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 Salpêtriers 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 experimenter, 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, Chem Ind 46, 427-34 (1940) (Founder of DuPont) & CA 34, 3548 (1940)
Note: In the dictionary: "Nouveau Petit LAROUSSE Illustree", Paris (1953), p 1341, 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, Lammet, was only 4 years his junior

Refs: 1) Anon, Chem&EngNews 30, 3097 (July 28, 1952) 2) Anon, Ordnance 48, 571 (1964) (Obituary)

DuPont, Lammet (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 saltpetre for K nitrate (imported from India) in the manuf of BkPdr. About 1880 he established the Repauno Chemical Co which manuf'd 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), p 4

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 Lammet duPont in 1844 as president of Repauno. He introduced at Repauno Works many improvements in Dynamites, such as introduction in 1893 of Gelatin-Dynamites


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 amounts 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

**Duranickel.** Trademark of DuPont Co for a wrought, “age-hardenable” alloy conatining 94% of Ni. Has greater strength and hardness and high resistance to corrosion which is characteristic of Ni
Ref: CondChemDict (1961), p425

**Duroplex.** Trademark of Rohm & Haas Co for drying and coagulating oil-modified alkyd resins derived from phthalic anhydride, polyhydric alcohols, and vegetable oils (Ref 2). It was used by Aerojet Engineering Corp, Azusa, Calif as an ingredient of some solid propellants, such as RL-223 (Ref 1)

**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 expls (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) Naoum, 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) Naoum, NG (1928), p385
5) Encycl of Expls, PATR 2700, Vol 2, p C14-L, "Drum Camera"
rates of gases by photographing flames as was done in Russia by V.A. Mikhail'son.

Duration Time of Burning of a Rocket Propellant. A term defined by the Aerojet Eng'g Corp, Azusa, 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-die-away portion of the curve would be located approx at 50% of the max value obtainable from the curve.

Ref: RocketEncycl (1959), p140 (Duration, Firing), 165 (Firing Duration) & 596 (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.

Ref: 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 Dulrol (sym-1,2,4,5-Tetrachlorobenzene),** C₆H₄(CH₃)₄Cl, mw 134.21, colorless crystals 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 cryst. It is used in plasticizers, polymers and in fibers.


**Durex.** Trademark of Hooker Chem Corp, for a line of phenol formaldehyde resins and molding compds; also including diacetyl phthalate resins.

Ref: 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

**Durimat-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.

Ref: 1) Daniel (1902), p214  2) Encycl of Expuls, 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 acidproof 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

Duroli See Durene

"Duroli" 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 Smoke. 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 Explosibility of. Dust expln data obtd in laboratory tests by the Bur Mines 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.


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 Bur Mines 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 (corn) 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.


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

![Dust Fuze Diagram](image-url)
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 or 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 charge leaked from the condenser, the remaining charge 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:**

**Dutch Explosives, Ammunition and Weapons.**

No information at our disposal. Attempt to obtain it directly from the "Ministerie Van Defence", Netherlands 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 Duijn 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 1D and 160mm long (closed with a glass stopper), an accurately weighed 4g sample of propellant (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 propellants or at 109–111°C for straight NC propellants. 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 propellants should not register more than 2% loss in 72 hrs and there should be no brown fumes.


**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 Geschützspulver 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, prepped 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. Mendeleev's "Pyro-collodium", which contained NC of N=12.44% and served as a prototype for US Navy proppt 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.


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 proppt RCP, invented in 1884 by his father (See above). He died as the Director of Vereinigen 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 (1843–1905) 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). Steetbacher (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. Steetbacher, "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)

Duxite 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°C(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, An 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 (1917), 374  2) Barnett (1919), p36(Duxite); 123–25 (Rotherham Test); 182–84 (Ballistic Pendulum Test)

Duxite T4. French designation for Trimethyl-enetriinitramine (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) calculated 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 without formation of polyethylene glycols or dihydroxy polymers.


Dymnyi Porokh (Russ for Smoke Powder). There are three types of Russian powders known under this name:

a) Chomy Porokh (Black Powder) (BkPdr).
A mix of KNO₃ 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 gain burning ca 0.64m/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 propellants 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% (Ref: 1) Gorst (1957), pp 156-59 2) Vasilevskii (1957), p26

Dyneget. 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, 49140(1969).

Dynamite or Nitromagrite. A Brit 80% Dynamite contg, as an absorbent, magnesia
alba (a hydrated Mg carbonate). It was patented in Great Britain in 1878 by E. Jones and authorized for manufacture in 1879. No factory was built, however, because the House of Lords declared that all explosible 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 shear deformation. A perfect fluid has no shear stresses despite shear deformations in the fluid (Refs 2, 3, 4, 6 & 7)


NOTE: See also Fluidics in Ordnance in Volume 6 of Encyclopedia

Note: LX-04-1 consists of HMX 85 & Viton A (Vinylidene fluoride hexafluoro propylene copolymer) 14%. PBX 9404: HMX 94, NC 3 & CEF (Tris-β-chloroethylphosphates) 3%. PBX 9011: HMX 90 & Estane (Polystyrene polyurethane of adipic acid, 1,4-butanediol, diphenylmethane diisocyanate) 10%.

RX-08-AZ: HMX 80, FEFO (Fluorodinitro ethyl formal) 13-16 & Polyester 3-6%


Dynamite Industry is Fading Away. Since the appearance on the market after World War II of ANFO (Amm nitrate Fuel oil), which sells at 4 to 6x a pound vs five times as much for Dynamite, many Dynamite 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%.


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.


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 $5 \times 10^8$ in/in/sec. The "dynamic" tensile strength of these expls is ca four times greater than their "static" strength.
DYNAMITE
AND SUBSTITUTE

(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 manufactured in US are LF (low-freezing) because they contain some NGc (Nitroglycerol), known also as EGDN (Ethylene glycol 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 explosion mixtures containing 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 mixtures 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 prep in 1833 of impure Nitrostarch (NS) by Bracconier. The next step was prep 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 prep in 1846 of NC of high N content (known as Guncotton), independently by C.F. Schünbein (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 prep 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 prep 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 pelter, 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 prep, naming their product Pyroglycerin or Gliolit. 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 Petersburg against the combined Anglo-French-Turkish Fleet. This barrage of E. Nobel proved to be successful (Ref 27, p 9 & Ref 37, p 311).

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, pp 1-7; Ref 27, pp 8-13; Ref 35, pp 196-97 and Ref 37, pp 285-87.

Accdg to Stettbacher (Ref 27, p 9), 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, pp 9-10), 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. Ghen, 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 "Pochaand" (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 Pochaand.

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 obtid 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 Malarsee. He was allowed, however, in 1865 to build a plant at Krummel, 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, pp Ger 218 & Ger 219).

Later in 1865 the Swedish Govt gave A. Nobel permission to build a plant in Vinterviken 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 infer- sorial 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 mix 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 "Guhrydynamite" 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), Pauleilles (France), Schlebusch near Köln (Germany), Bilbao (Spain), Isleten (Switzerland), Avigliana, near Torino (Italy), Trafalga 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 throughout 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 Lamont 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 Guhry dynamite 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 expl. 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 ("nitro-lignin") 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 Scely and mixtures of pulverized AN with sawdust or charcoal were proposed in 1867 by Swedish inventors Norbin & Ohlsson and also by Björkman. When these mixtures were combined with NG, Dynamite-like expls Aminikkret and Serain were obt'd (See Ref 58, pp A306-R to A307-L). The Serain of Björkman, 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 Séraine 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 Norbin & 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 Seranit, 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, p4) 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 Masconetcong tunnel, 1 mile long, near Eastern, 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 Ammon-gelatin 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, p19)

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 amount 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 DNBs. Other substances proposed in 1876 were amyl- and isoamyl nitrates. 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 brittleness 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 Greisenaup Mine, Germany, was caused by frozen Dynamite. This disaster produced the renewal of research on antifreezes and in 1903 the SA de Poudes et Dynamites of France introduced DNT & TNT as antifreezes. These combs 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, p629 (quoted from Ref 15, pp15-16). 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 CO₂ = 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, pC266-L) under the name of 2-Chloropropane-1,3-diol-1,3-dinitrate or β-Dinitrochlorohydrin, (O₂N·CH₂·CH(Cl)·CH₂(NO₂)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 CO₂ 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, p371). 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 classic work in ZAngewChem 18, pp11 & 53 (1905). In this work he calculated the molecular lowering of the fr p of NG by a great number of compps 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 Tetranitroglycerin (TeNDG), Polynitroglycerin (PNG) or Nitropolyglycerin (NPG), (O₂N·CH₂·CH(NO₂)₂·CH(OH)₂·CH(ONO)₂·CH₂(NO₂). Its prepn and properties are described in Naúm (Ref 15, pp199-202) and in Vol 5 of Encycl under Diglycerol, pD1261-L.

Accdg to E.M. Symmes (translator of Naúm's book), DGTeN was used in the USA to a very large extent beginning in 1912 until about 1926 (Ref 15, p202, Footnote 5). It was not used, however, in Germany where it was invented. Its manuf was patented by the Zentralstelle für wissenschaftlich-technische Untersuchungen, Neubabelsberg, near Potsdam (Ref 15, p19).

In the years following 1906, other substances were proposed as antifreezes: a) Mixed glycerin esters, such as Dinitroacetin with Dinitroformin [Veazio Vender, SS 2, 21 & 195(1907)]

b) 1,3-Butylenglycol Dinitrate, patented in 1911 in the USA and described in Ref 15, p375-76
c) Mixed Ethylene-propylene Glycol, prep 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, p375, Footnote of Translator)
The most important antifreezes proved to be Nitroglycol (NGc) or Glycoldinitrate (GeDN), also known as Ethyleneglycol Dinitrate or Dinitroglycol, \( (\text{O}_2\text{NO})\text{CH}_2\text{CH}_2\text{ONO}_2 \). Its prep 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 EngP 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 NGc 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 NGC) 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 strength 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>
<thead>
<tr>
<th>Component</th>
<th>Gelatin</th>
<th>Dynamite</th>
<th>German</th>
<th>German Straight</th>
<th>French</th>
<th>Blueing</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>II</td>
<td>I</td>
<td>II</td>
<td>I</td>
<td>II</td>
<td>I</td>
</tr>
<tr>
<td>Safety Explosives</td>
<td>Swiss (French)</td>
<td>Gelatin</td>
<td>German</td>
<td>German Straight</td>
<td>French</td>
<td>Blueing</td>
</tr>
</tbody>
</table>

**TABLE 1**

<table>
<thead>
<tr>
<th>Proprieties</th>
<th>NACl</th>
<th>NAOH</th>
<th>NaOH</th>
<th>KC1O4</th>
<th>NH4NO3</th>
<th>KNO3</th>
<th>N2O5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Velosity of Gran - m/sec</td>
<td>7000</td>
<td>6000</td>
<td>24</td>
<td>24</td>
<td>24</td>
<td>24</td>
<td>24</td>
</tr>
<tr>
<td>Total Block Casting - m</td>
<td>490</td>
<td>490</td>
<td>490</td>
<td>490</td>
<td>490</td>
<td>490</td>
<td>490</td>
</tr>
<tr>
<td>Transit Test Value - cC</td>
<td>8.5</td>
<td>8.5</td>
<td>8.5</td>
<td>8.5</td>
<td>8.5</td>
<td>8.5</td>
<td>8.5</td>
</tr>
<tr>
<td>Density - g/cc</td>
<td>1.27</td>
<td>1.27</td>
<td>1.27</td>
<td>1.27</td>
<td>1.27</td>
<td>1.27</td>
<td>1.27</td>
</tr>
<tr>
<td>O2 or C02 %</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>
1) A higher volatility than NG, but not so high as to cause appreciable evaporation in the cartridge 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, p 380)

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, p 1), 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, p 3).

Properties of these Dynamites are given in Ref 16, Tables 2 & 3, pp 5 & 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 prep 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 & actacid. 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.

Acedg 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)

<table>
<thead>
<tr>
<th>Class and Grade of LF Dynamites</th>
<th>Composition, %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>NG with NPG</td>
</tr>
<tr>
<td>25% Gelatin Dynamite</td>
<td>22.5</td>
</tr>
<tr>
<td>40% Gelatin Dynamite</td>
<td>32.7</td>
</tr>
<tr>
<td>60% Gelatin Dynamite</td>
<td>50.3</td>
</tr>
<tr>
<td>60% Ammonia Gelatin</td>
<td>36.1</td>
</tr>
<tr>
<td>40% Straight NG Dynamite</td>
<td>39.3</td>
</tr>
</tbody>
</table>

---


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. Gragertoff. 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 Glycerol

Nitroglycerin (NG) or Glycerol Trinitrate,

\[ C_3H_5(ONO_2)_3, \text{ mw 227.09, N 18.50%, OB to CO}_2 +3.5%, \text{ OB to CO +24.5%; colorless oil; sp gr 1.591 at 25^\circ, np 1.4713 at 25^\circ; mp 13.2^\circ (stable form) and 2.2^\circ (labile form); bp decomp at 145-150^\circ; hydrolisis 0.005% acid at 60^\circ in 5 days, hygroscopicity 0.06% at 30^\circ and 90% RH; specific heat 0.356 cal/g/\text{gC} for liquid and 0.315 for solid; viscosity 36.0 centipoises at 20^\circ; volatility 0.11 mg/cm/hr at 60^\circ; very sl sol in w; sol in alc or in ether; breathing of its vapors causes a headache.]

NG is prepd by nitratation 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. Nitratation is usually conducted at 25^\circ 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^\circ. 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:

- Brissance 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^\circ in 5 secs
- Flammability - Its explosives can be ignited by flame but not as easily as those with Nitroglycerol (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^\circ - 4% 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 Guhdynamite 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^\circ - less stable than Nitroglycerol (Ref 15, p226)
- KI Test at 81.1^\circ - 10+ minutes

Power (Strength) by Ballistic Mortar - 140% of TNT

Power by Trauzl Test - Naoûm (Ref 15, p227) gives 5900 cc for 10 g sample; TNT produces expansion ca 300 cc

Rifle Bullet Impact Test - 100% explosions

Strength - See Power

Temperature of Explosion. Accdg to Brunswig (as quoted in Naoûm) 3470^\circ 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

Nitroglycerol (NGc), Glycol Dinitrate or Ethylene-glycol Dinitrate, \((\text{CH}_2\text{O})_2\text{C}_3\text{H}_4\text{O}_7\text{N}_3\text{O}_9\), mw 152.07, N 18.42%, OB to CO, 0.0%, OB to CO, +21.04%; nearly colorless; slightly syrupy liquid; sp gr 1.485 at 25°, nD 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 prep by nitration of ethylene glycol, \((\text{CH}_2\text{O})_2\text{H}_2\) with a mixed nitric-sulfuric acid in the same apparatus that is used for prep of NG. The glycol is prep 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: 2000 & 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 Sensitivity w/Kast Apparatus – 1 drop on filter paper exploded at 20 to 25cm fall of 2kg weight (Ref 15, p225)
Initiation Sensitivity – More sensitive than NG to initial impulse; can be exploded by cap No 1 (Ref 15, p226)
K1 Test at 75° – More stable than NG (Ref 15, p226)
Power (Strength) by Trauzl Test – 650cc for 10g sample vs 900cc 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 Ata (Ref 37)

Class I. Nongelatinous (Powdery) Nitroglycerin Explosives with an Inactive (Inert) Base, such as Gubrodynamite, 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 30 mm 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 Naoum (Ref 15, pp266-73). Illustrations on pp267 thru 271 of Ref 15 show various Dynamite Cartridge Packages.

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 1.6 g/cc (1.5 is the highest practical)
- **Detonation Equation**: accord to Pérez Araujo (Ref 37, p320): \(2C_3H_5(ONO_2)_3 + 2.48SiO_2 + 6CO + 3H_2O + 3N_2 + 0.50_2 + 2.48SiO_2\)
- **Detonation Rate**: 5650 m/sec at density 1.3 and 6650 to 6800 at 1.5 g/cc
- **Explosion Temperature or Deflagration Point**: 180°–182°, when heated slowly and 240° when heated at the rate of 20° per minute (Ref 15, p277)
- **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 Sensitivity**: detonates when rubbed between iron or other metal plates (Ref 15, p276)
- **Gas Volume on Explosion**: 536,8/L/kg (Ref 15, p276)
- **Heat of Explosion**: ca 1200 kcal/kg with water liquid (Ref 15, p276)
- **Impact Sensitivity**: with 2 kg weight ca 7 cm and 20 cm for frozen expl (Ref 15, p276)
- **Initiation Sensitivity**: GD can be detonated, when not frozen, with a cap contg 0.6 g MF but requires at least 1.0 g MF when in frozen state (Ref 15, p277)
- **Power**: See Strength

Propagation of Detonation (Sensitivity by influence). Cartridges 30 mm in diam can propagate detonation over a space of 30 cm 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°. Accordg 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. 10 g GD under sand tamping gave a net expansion of 320 cc vs 550 cc for liquid NG. This
means 55% NG (Ref 13, pp278-79). Rinkenbach gave 97% TNT (Ref 62, p367)
Temperature of Explosion. Accdg to Brunwig (as quoted by Naoum) 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 Naoum (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 "Magnesialny" (See under Historical); magnesia alba, in "Dynamagnite" or "Nitromagnite"; magnesium sulfate, in "Nobel's Ardeer Powder"; pulverized mica, in "Mica Powder"; tanninite in "Dynamite de Vonges"; and Na carbonate, crystalized, 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 bores 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°C. 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 & Mining (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 Gex 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 Grisoumite and in some Amer Dynamites; cork, charcoal (in "Carbdynamite", described in Ref 60, p C52-L); naphthalene (in Reineischemdynamit) 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 absorption 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 add'l 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 cartridgeg. 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 USButMines 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, Collodial 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...
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 compo.

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>
<thead>
<tr>
<th>Components, % and Properties</th>
<th>Strength, %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>20</td>
</tr>
<tr>
<td>Nitroglycerin</td>
<td>20.2</td>
</tr>
<tr>
<td>Na nitrate</td>
<td>59.3</td>
</tr>
<tr>
<td>Carbonaceous combustible material</td>
<td>15.4</td>
</tr>
<tr>
<td>Sulfur</td>
<td>2.9</td>
</tr>
<tr>
<td>Antacid</td>
<td>1.3</td>
</tr>
<tr>
<td>Moisture</td>
<td>0.9</td>
</tr>
<tr>
<td>Count (Density)</td>
<td>102</td>
</tr>
<tr>
<td>Ballistic pendulum test, % TNT</td>
<td>83</td>
</tr>
<tr>
<td>Detonation rate, m/sec</td>
<td>3600</td>
</tr>
</tbody>
</table>
### Table IV

<table>
<thead>
<tr>
<th>Components, % and Properties</th>
<th>Strength, %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>25</td>
</tr>
<tr>
<td>NG/NGc—90/10</td>
<td>15.0</td>
</tr>
<tr>
<td>Collodion Cotton</td>
<td>14.0</td>
</tr>
<tr>
<td>Na Nitrate</td>
<td>60.0</td>
</tr>
<tr>
<td>AN (coated)</td>
<td>4.5</td>
</tr>
<tr>
<td>Sulfur</td>
<td>1.0</td>
</tr>
<tr>
<td>Marble Dust</td>
<td>1.5</td>
</tr>
<tr>
<td>&quot;A&quot; Pulp *</td>
<td>4.0</td>
</tr>
<tr>
<td>&quot;B&quot; Pulp **</td>
<td>4.0</td>
</tr>
</tbody>
</table>

* "A" Pulp was a wood pulp with a low absorptive value
* "B" Pulp was a wood pulp 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 92–93% 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 cong 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).

According to Steertbacher (Ref 27, p333), 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 exps, 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 pro-
properties, as described in Ref 37, p325. For this a small specimen (ca 0.2 g) of NC is placed in a crucible containing 10 g of NG which is heated on a water bath for 13 mins with constant stirring of the contents with a wooden spatula. Then the crucible is cooled for 15 mins 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 "cannistering house", where it is extruded into paper cannisters (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 exps 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" Gelatin 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" Gelatins are claimed to retain their high velocities even under extremely adverse conditions. Their densities are about 10% lower.

Table V

<table>
<thead>
<tr>
<th>Gelatin Dynamites (USA)</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Components, % and Properties</th>
<th>Strength, %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>20</td>
</tr>
<tr>
<td>-----------------------------</td>
<td>----</td>
</tr>
<tr>
<td>NG</td>
<td>20.2</td>
</tr>
<tr>
<td>Na nitrate</td>
<td>60.3</td>
</tr>
<tr>
<td>NC, soluble</td>
<td>0.4</td>
</tr>
<tr>
<td>Carbonaceous combustible material</td>
<td>8.5</td>
</tr>
<tr>
<td>Sulfur</td>
<td>8.2</td>
</tr>
<tr>
<td>Antacid</td>
<td>1.5</td>
</tr>
<tr>
<td>Moisture</td>
<td>0.9</td>
</tr>
<tr>
<td>Ballistic pendulum test, %TNT</td>
<td>70</td>
</tr>
<tr>
<td>Rate of detonation, m/sec</td>
<td>4000</td>
</tr>
</tbody>
</table>
than the regular DuPont Gelatins, but they are comparable in sensitiveness to shock and friction (Ref 63, p38).

Aging (aging) of Dynamites is discussed in detail in Vol 1 of Encycl (Ref 58, pp A110-R to A112-L).

The so-called Tosal 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 PATR2510 (Ref 56, p Ger 67). Some of them are listed here in Table VI.

Acdng to Stegbar (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 prep’d by mixing the ingredients at ca 40°C (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>
<thead>
<tr>
<th>Table VI</th>
<th>Gelatin Dynamites (German &amp; Swiss)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Components, % and Properties</td>
<td>Designation</td>
</tr>
<tr>
<td></td>
<td>No 1</td>
</tr>
<tr>
<td>NG (or NG + NGc)</td>
<td>62.5</td>
</tr>
<tr>
<td>NC</td>
<td>2.5</td>
</tr>
<tr>
<td>Vegetable meal</td>
<td>8.0</td>
</tr>
<tr>
<td>TNT &amp; DNT</td>
<td>—</td>
</tr>
<tr>
<td>Na nitrate</td>
<td>27.0</td>
</tr>
<tr>
<td>K perchlorate</td>
<td>—</td>
</tr>
<tr>
<td>Alkali chloride</td>
<td>—</td>
</tr>
<tr>
<td>Oxygen Balance to CO₂, %</td>
<td>+4.4</td>
</tr>
<tr>
<td>Density</td>
<td>1.55</td>
</tr>
<tr>
<td>Trauzl Test, cc</td>
<td>400</td>
</tr>
<tr>
<td>Pb Block Crushing, mm</td>
<td>20.0</td>
</tr>
<tr>
<td>Detonation Rate, m/sec</td>
<td>7000</td>
</tr>
<tr>
<td>Heat of Explosion, kcal/kg (H₂O vapor)</td>
<td>1235</td>
</tr>
<tr>
<td>Temperature of Explosion, °C</td>
<td>2950</td>
</tr>
</tbody>
</table>
### Table VII
**Gelatin Dynamites of Apache Powder Co**

<table>
<thead>
<tr>
<th>Components, % and Properties</th>
<th>Strength, %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>30</td>
</tr>
<tr>
<td>NG/NGc - 90/10</td>
<td>26.0</td>
</tr>
<tr>
<td>Collodion Cotton</td>
<td>0.2</td>
</tr>
<tr>
<td>NaNO₃</td>
<td>57.3</td>
</tr>
<tr>
<td>&quot;A&quot; Pulp *</td>
<td>–</td>
</tr>
<tr>
<td>&quot;B&quot; Pulp **</td>
<td>–</td>
</tr>
<tr>
<td>Sulfur</td>
<td>8.0</td>
</tr>
<tr>
<td>Oat Hulls (dry)</td>
<td>–</td>
</tr>
<tr>
<td>Starch (dry)</td>
<td>7.5</td>
</tr>
<tr>
<td>Flour</td>
<td>–</td>
</tr>
<tr>
<td>Marble Dust</td>
<td>1.0</td>
</tr>
<tr>
<td>Detonation Rate, m/sec</td>
<td>2210</td>
</tr>
<tr>
<td>Detonation Rate, ft/sec</td>
<td>7245</td>
</tr>
</tbody>
</table>

* "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**

<table>
<thead>
<tr>
<th>Components, %</th>
<th>Strength, %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>25</td>
</tr>
<tr>
<td>NG</td>
<td>22.5</td>
</tr>
<tr>
<td>Collodion Cotton</td>
<td>0.3</td>
</tr>
<tr>
<td>NaNO₃</td>
<td>62.0</td>
</tr>
<tr>
<td>&quot;A&quot; Pulp *</td>
<td>–</td>
</tr>
<tr>
<td>&quot;B&quot; Pulp *</td>
<td>–</td>
</tr>
<tr>
<td>&quot;C&quot; Pulp **</td>
<td>–</td>
</tr>
<tr>
<td>Flour (hard wheat)</td>
<td>6.2</td>
</tr>
<tr>
<td>Wheat Middlings</td>
<td>–</td>
</tr>
<tr>
<td>Corn Meal</td>
<td>4.0</td>
</tr>
<tr>
<td>Sawdust</td>
<td>–</td>
</tr>
<tr>
<td>Sulfur</td>
<td>4.0</td>
</tr>
<tr>
<td>Marble Dust</td>
<td>1.0</td>
</tr>
</tbody>
</table>

* "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, p940).

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, p945 of Vol I 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 CO2 +1.5%, density 1.44, Trauzl test value 485 cc, Pb block crushing 21.0 mm, detonation rate 7000 m/sec, heat of expln (H2O vapor) 1300 kcal/kg and temp of expln 2770°C.

Note: This expl, called "Ammonia Dynamite" (Forsite) by Naoum (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 WWII 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

<table>
<thead>
<tr>
<th>Components, %</th>
<th>Designation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>A</td>
</tr>
<tr>
<td>NG</td>
<td>10.0</td>
</tr>
<tr>
<td>Drip Oil</td>
<td>-</td>
</tr>
<tr>
<td>AN (fine)</td>
<td>77.0</td>
</tr>
<tr>
<td>AN (coarse)</td>
<td>-</td>
</tr>
<tr>
<td>Soda (screened)</td>
<td>3.0</td>
</tr>
<tr>
<td>Soda (pulverized)</td>
<td>-</td>
</tr>
<tr>
<td>Pulp</td>
<td>10.0</td>
</tr>
<tr>
<td>Corn Meal</td>
<td>-</td>
</tr>
<tr>
<td>NaCl</td>
<td>-</td>
</tr>
<tr>
<td>NH4Cl</td>
<td>-</td>
</tr>
<tr>
<td>(NH4)2SO4</td>
<td>-</td>
</tr>
<tr>
<td>Marble Dust</td>
<td>-</td>
</tr>
</tbody>
</table>
Class V. Gelatinous Nitroglycerin-Ammonium Nitrate Explosives, known as Ammonia Gelatin Dynamites. The so-called Special Gelains manufactured by the DuPont Co differ from “DuPont Straight Gelatin” (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 Gelatin”, but they are somewhat lower in velocity of detonation and slightly less water-resistant. They are manufactured 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 Gelatin” 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 Gelatin, 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 Gelatin Dynamites are also listed in Table 14 of Ref 62 and in Ref 66, p78-84. Germans used before WWII several types of Ammonia Gelatin containing large proportion of DNCH (Dinitrochlorhydrin), (O2NO)CH2.CH(ONO2).CH2Cl. A mixture of DNCH with 15–20% NG was known as “Nitrochlorin”. Following is an example of such low-freezing Dynamites: Deutsche Ammongelatine: Dinitrochlorhydrin 30, NC 3, DNT with TNT 10, AN 45, NaN3 10 & woodmeal 2%; its density was 1.45, brisance by Kast formula B2x10^6 vs 86x10^6 for TNT, detonation rate 6900 m/sec, Trauzl test value 400 cc, heat of explosion 1100 kcal/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 Gelatin 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 Wetzersprengstoße 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 permissible expls 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 permissible 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, p.276), 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" (p240).

D. Coyote Tunnels, Blasting of is briefly described in Vol 3 of Encycl (Ref 64, pC551) and in Ref 63, pp 240-44. 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, pp 245-64. 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, pp 279-84, 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, pp 285-98, 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 permisibles are strong enough to produce adequate breakage and displacement in soft rock or coal (p297)

In Chap 20 of Ref 63, pp 299-312, is described the development of "Strip Mining of Coal" with recommended expls: "ANFO-P", "Nilire" 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, pp 313-26, 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 "stopping 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, pp 327-50. The recommended expls by DuPont Co for

### Table X

**Quarry Powders of the Apache Powder Co, Benson, Arizona**

<table>
<thead>
<tr>
<th>Components, % and Properties</th>
<th>1LF</th>
<th>2LF</th>
<th>4LF</th>
<th>6LF</th>
</tr>
</thead>
<tbody>
<tr>
<td>NG</td>
<td>7.2</td>
<td>8.3</td>
<td>7.5</td>
<td>4.5</td>
</tr>
<tr>
<td>Drip Oil</td>
<td>0.8</td>
<td>1.7</td>
<td>1.5</td>
<td>0.5</td>
</tr>
<tr>
<td>NaNO₃ (pulverized)</td>
<td>-</td>
<td>-</td>
<td>22.0</td>
<td>-</td>
</tr>
<tr>
<td>NaNO₃ (screened)</td>
<td>11.0</td>
<td>24.0</td>
<td>22.0</td>
<td>66.0</td>
</tr>
<tr>
<td>Sawdust</td>
<td>10.0</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>NH₄NO₃ (coarse)</td>
<td>65.0</td>
<td>50.0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>NH₄NO₃ (fine)</td>
<td>-</td>
<td>-</td>
<td>27.0</td>
<td>-</td>
</tr>
<tr>
<td>&quot;A&quot; Pulp (low absorptive value)</td>
<td>-</td>
<td>5.0</td>
<td>6.0</td>
<td>-</td>
</tr>
<tr>
<td>Sulfur (pulverized)</td>
<td>5.0</td>
<td>-</td>
<td>8.0</td>
<td>-</td>
</tr>
<tr>
<td>Sulfur (screened)</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>14.0</td>
</tr>
<tr>
<td>Corn Meal</td>
<td>-</td>
<td>-</td>
<td>5.0</td>
<td>-</td>
</tr>
<tr>
<td>Coal (pulverized)</td>
<td>-</td>
<td>10.0</td>
<td>-</td>
<td>14.0</td>
</tr>
<tr>
<td>Marble Dust</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Ballistic Pendulum, % TNT</td>
<td>98.0</td>
<td>88.0</td>
<td>85.0</td>
<td>69.0</td>
</tr>
<tr>
<td>Lead Block Crushing, mm</td>
<td>4.1</td>
<td>5.0</td>
<td>8.75</td>
<td>4.8</td>
</tr>
</tbody>
</table>
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.

C. 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 acoustic 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 depth 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 5" 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, p6.45), 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 manufacture 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 comp 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, p6.45).

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% “Hi-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 Gottard Tunnel, 14920 meters long, connecting Italy with Switzerland, used low-freezing Dynamite and construction was completed in 6½ years between 1873 & 1880.

c) Arlberg Tunnel, 10240 meters long, in Tyrol (Austria), used Dynamite and was constructed in 3 years, between 1880 & 1883.

d) Simplon Tunnel, 19770 meters long, connecting Italy and France, used Blasting Gelatin and Dynamite, and was constructed in 6½ years, between 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 Drilling (p477–
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 rec
recommended expls: 60 or 80% "Hi-Velocity Gelatin";
h) Blasting Log Jams (p481), with recom
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, at
tempts were made to apply them either by them
selves 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, mil
itary cratering, military demolition, military exca
vation 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
propels 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 Pro-
pellants" 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, con-
taining 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
Piroksilinovoe Shashki consisted of compressed
NC and are probably still used.

Because NG and its mixtures (such as Dyna-
mites) are very sensitive to friction and impact
(especially by rifle bullet), they found no satis-
factory use as bursting charges in ammunition,
although many attempts were made to do so. One
of the earliest attempts was by a Russian officer
Petrushkovskii, 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 BkRf used at that time. This
idea was, however, abandoned after several
expls 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 Smaiianinoff for loading shells up to 203 mm. No premature took place at muzzle velocity up to 654 m/sec (See "Americanite" in Vol 1 of Encycl, p A168-L).

When 7 to 10% of CC (Collodium Cotton, NC of ca 12% N) was incorporated with NG, the resulting hard gel was less sensitive than Guhr-dynamite, 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, pp 233-34). 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, p 934), 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 caliper, firing 600-pound shells at a muzzle velocity of 600 ft/sec. Some field guns of that caliper were built, but proved unsatisfactory because their range was only 1950 yards.

Another variant was Hiram Maxim Gun (Ref 14, p 937), 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 Ana reported (Ref 37, pp 333-34) that Americans used large caliber pneumatic guns, "canones de dinamina" in their coastal defenses and also during the Spanish-American War (1898). One of the American warships "Veiusbus", 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, p 327).

Although Dynamites proved to be unsuitable as bursting charges in 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 Cutting Explosive in Vol 3 (Ref 64), pp C553-R & p D57 and in Pica-tinny 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 PicArsenTechRepts, 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 WWI was based on NS (Nitrostarch) instead of NG. Its compn and properties are given in Ref 52, p 207; Ref 66, p 7-85 and in Vol 3 of Encycl under Ref 12, listed on p D60-R. Trojan demolition explosives were tested at Pica-tinny 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 4570 m/sec and relative effectiveness 0.86 of TNT block with velocity 6400 m/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 WWII for loading trench mortar shells and hand & rifle grenades. Its comp 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 Expns (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% Colloidin 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 35 g sand crushed vs 43 g for TNT; Detonation Rate 6000 m/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 Naoun (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, Anm 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 (13.15 N) 3.0%. Properties: Density 1.79 g/cc; Brisance 22.3 g sand crushed; Detonation Rate 2600 m/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 48 hrs 0.17 and no expln in 100 hrs; Hygroscopicity, 90% RH, 30° gained in wt 2.25% in 26 days; Impact Test with 2 kg wt, PicArsn App 7 inches; Rifle Bullet Impact Test in std cast iron bomb 100% explns; Vacuum Stability at 100° 3.66 cc 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, t alc 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 Hantite, also developed by Ottosen. 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. Baldwin, 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) compo is listed in Spec MIL-D-45413A (MU), 15 Aug 1962: RDX 75%+10, TNT 15%+1, Grade SAE No 10 Engine Oil plus Polyisobutylene 520, 5% & Cornstarch 520.5%

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. Velicky & M. Epton deals with development of Nipolit—Type Explosives. One compo 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) 24.5
- graphite glaze

- 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 & brassing. The material is fairly rigid in large diameters but somewhat flexible in small diameters.

The method of prep by 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 (70/15/5/3 RDX/talc/di-(2-ethyl-hexyl) 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/contour starch/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 (Triperoxyetriol) 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 48.0g 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

Fricitn Sensitivity – unaffected by steel or fiber shoe

Gap Propagation in Air – max distance 2.5 inches for expln

Gas Volume – 611cc/g

Heat of Explosion – 625cal/g

Hygroscopicity – 0.31% gain in 30 days at 71° & 95% RH

Impact Sensitivity – PicAtsn App 22 inches with 2 kg weight

Initiation Sensitivity – dry material explodes by No 6 Electric C the 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°C – 0.90 cc/40hrs

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.

References on DYNAMITE and SUBSTITUTES
Explosives", Reinhold, NY (1958), pp 11-12 (Waterproofing of AN and other hygroscopic dope in Dynamites is achieved to a certain degree by a film of NG or of NG-NC gel. 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 blastingexplos (e.g. Nitrocellulose gels), higher cost than for BIbR, and higher sensitiveness (which sometimes is desirable))

57) M. Giua, "Trattato di Chimica Industriale", UTET, Torino, 6(I) (1959), pp 338-45


60a) B. Belkovski & G.T. Dyakov, "Vzryvny Veshchestva: Teoriya, Khimiya, Tekhnologiya i Uprorba" (Explosive Substances: Theory, Chemistry, Technology and Use), Tekhnika, Sofia (Bulgaria) (1962), 339pp 60b) V.A. Assonov, "Vzryvchatye Veshchestva i ikh Primenenie v Gomoy Promyshlennosti" (Explosives and Their Application in Mining Industry), Moscow, GosNauchno-TekhIzdatLit po GomDelu, 1963, 139pp

60c) E.L. Gómez, "Estudio de los Explosivos Industriales", Interiencia, Madrid (1963), pp 129-

Note: 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 PROCEEDURES

(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 conng NG and NGc, it is necessary to warn that both of these substances (especially NGc) cause headaches on inhaling their vapors and cause 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 \( = W/V \).

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 amount 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. BerMin 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, 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 prep 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 prep of KI-starch paper, is in BerMin Bull 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 exp1 submitted to the BerMin 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 BerMin Bull 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 BerMin 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 BerMin (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 Dautriechg or Chronograph Methods described in Vol 3 of Encycl, pp C31L to C31B. Mettegag- and Dautriechg Methods have been used at the BerMin 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 BerMin Bull 346 (Ref 5) pp79–84

**Explosion-by-Influence Sensitivity** or Air-Gap Sensitivity has been detd at the BerMin 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 & B 300

**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.

1A. 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 CCl₄ 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-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 CCl₄ to the flask containing 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°C 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:

\[ \text{% Moisture} = \frac{[(A - B) + (0.11\times)\times 100}{\text{Wt of sample}} \]

*(0.11) is constant correction in grams compensates for water present in CCl₄ 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 CCl₄ are used in distillation in lieu of a 250-ml flask and the 150-ml CCl₄ 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 amount of moisture, use less than 50 g samples.

1B. Moisture by Karl Fischer Method

According to Pierson (Ref 11, p1353), the Karl Fischer Method, as described on pp 1290-93, 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 detection of small amounts 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 prepared as described below.

Karl Fischer Reagent used in Method 101.4 of MIL-STD-650 (Ref 10a, p2) is prepared in the following manner: Tum on the hood (to avoid inhaling pyridine vapor which is odorous and toxic). Take 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, according to the equation derived in 1939 by Smith, Bryant & Mitchell and later described by R.W. Freedman in AnalChem 28, 247 (1956):

$$\text{SO}_3 \cdot \text{C}_9 \text{H}_5 \text{N} + \text{I}_2 \cdot \text{C}_6 \text{H}_5 \text{N} + \text{C}_6 \text{H}_5 \text{N} + \text{CH}_3 \text{OH} + \text{H}_2 \text{O} \rightarrow \text{C}_6 \text{H}_5 \text{N} \cdot (\text{H}) \cdot \text{SO}_4 \cdot \text{CH}_3 \cdot 2 \text{C}_6 \text{H}_5 \text{N} \cdot \text{H}_2 \text{O}$$

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 mix makes a visual dem of the end point difficult. Therefore, the endpoint can be determined 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

![Fig 2. Wiring Diagram](image)

1.5 volt dry cell

40 μ potentiometer

Galvanometer

Pt electrodes

Fig 2. WIRING DIAGRAM

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 solution 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 the end point is approaching, because the local excess of KFR's cause momentary deflections of the galvanometer.

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 "Aquattator" of Precision Scientific Co is shown here on the left hand side of Fig 5.
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 made 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
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 prep’d as described in Bofors Manual (Ref 8, p17-L) in the following manner: Place 15 g of Mg chips or clippings in a 5-liter flar-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$_2$C$_4$H$_4$O$_7$·2H$_2$O) 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 prep’n, 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 No 21).

**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, pp 1291ff), 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, p 1291) 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 P₂O₅ 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 atmo-
spheric 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 arrange-
ment 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 toward lower values, the re-
agent 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 titra-
ting cell (beaker) and titrating with KF reagent
to the change of color specified
Calculation:

Water Equivalent of KF Reagent in mg/ml =

\[
\frac{mg \text{ of } H_2O \text{ in 10 ml water-in-methanol soln}}{\text{ml of KF reagent used}}
\]

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 re-
agent with water-in-methanol soln.
Repeat the titrations until the ratios from 3

successive titrations agree within 5 ppt. Then
standatize 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$_2$C$_4$H$_6$O$_6$·2H$_2$O (weighed
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 re-
agent until a moderate excess is reached, and
back-titrating with water-in-methanol soln. Con-
vert the weight of dihydrate used to milligrams
of water (each mg of dihydrate contains 0.1556 mg
H$_2$O) 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-metha-

nol soln})]
\]

\[
r = \text{ratio of volumes, KF reagent per water-in-

methanol}
\]

Determination of Moisture in Dynomites

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 0.050 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 
- \( W \) = 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(Nitroglycerol or Ethyleneglycol Dinitrate) and DEGDN(Diehydrene, glycol 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-3150S, p486 of E.H. Sargent & Co catalog (See our Fig 1). An alumnum crucible is used in place of extraction cup S-31615 or S-31625. If elemental sulfur is present, a 2nd extraction is made with CS₂.

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-CS₂ 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 CS₂, 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 prep'd by dissolving in 90 ml of distd w, 5 g Cd₃ and
**Figure 2 Determination of Ether Solubles**

1. **Ether Extract**
2. **CS₂ Extract (if sulfur present)**
3. **Combine extracts**
4. **Nitrocompounds Absent**
5. **Nitrocompounds present**
6. **Split Sample**
7. **Acetic Acid Treatment** *(Removes Explosive oil and Nitrocompounds)*
8. **Grease (Oils & Resins) + Sulfur**
   - **Acetone Extraction**
     - **Extract**
     - **Residue**
   - **Grease**
   - **Sulfur**
9. **Methyl Sulfuric Acid Treatment** *(Removes Explosive Oil)*
10. **Ether Extraction**
    - **Residue Discard**
    - **Extract**
      - **Nitrocompounds, grease, trace sulfur**
      - **Wash with water + NaHCO₃**
        - **Water Layer Discard**
        - **Ether Layer Evaporate**
      - **Acetone Extraction**
        - **Grease**
        - **Nitrocompounds**
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 indicates 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°C, 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:**

\[
\% \text{Ether solubles + Moisture} = \frac{(\text{Wt loss of cruc}) \times 100}{\text{Wt of sample}}
\]

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 CS₂, place in a water bath and extract for 2 hours

*Caution:* Avoid breathing the CS₂ vapors

n) Remove the extractor from the water bath, cool, and place the crucible on an edge of a steam bath to evaporate residual CS₂

o) Dry in an oven for 2 hours at 95°C or overnight at 70°C, cool in a desiccator and weigh.

**Calculation 2:**

\[
\% \text{Ether} - \text{CS}_2 \text{ solubles} + \text{H}_2\text{O} = \\
\% \text{Ether solubles} + \text{S} + \text{H}_2\text{O} = \\
\frac{(\text{Wt loss of crucible})\times 100}{\text{Wt of sample}}
\]

Save the crucible with residue

q) Transfer the CS₂ 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-CS₂ extract to 200 ml with ether and withdraw 100 ml into a beaker for the determination 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 & lr 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-CS₂ 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+NGe) and nitrocompd 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:

% Grease(oils & resins) = \[ \frac{(Wt \text{ gain of beaker}) \times 100}{Wt \text{ of sample}} \]

% Explosive Oil (NG+NGc) = Calculation 2 (Step 1p) minus the sum of: Calculations 3 & 4 (Step 2f), Nitrobody (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:

% Grease = \[ \frac{(Wt \text{ gain of 50-ml beaker}) \times 100}{Wt \text{ of sample}} \]

% Sulfur = Calculation 3 (Step 2f) minus % Grease

*Note F:* Sulfur can also be det by oxidizing it with bromine water, followed by pptg with Ba chloride soln and weighing as BaSO₄

3. Nitrocompounds Determination

a) Take the 100-ml ether-CS₂ extract retained for the det of nitrocompounds in the first beaker of step 1t, 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 sulfartic 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 NaHCO₃ 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 comps

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:

% Nitrocompounds = \[ \frac{(Wt \text{ gain of beaker}) \times 100}{Wt \text{ of sample}} \]
III. WATER (OR WATER-ALCOHOL) SOLUBLE CONSTITUENTS. DETERMINATION IN COMMERCIAL BLASTING EXPLOSIVES

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 amount 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 1 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 ether extraction tube of Standard Method No 30)

Note B: Extract NS (Nitroestarch) exps 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 exps 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:

\[
\text{% Water (or water-alcohol) solubles} = \frac{\text{Loss of weight}}{\text{Sample wt (50 g)}} \times 100
\]

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 amount of salts present in soin 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:

\[4\text{NH}_4\text{NO}_3 + 6\text{HCHO} \rightarrow (\text{CH}_2)\text{N}_2\text{O}_4 + 6\text{H}_2\text{O} + 4\text{HNO}_3\]

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 (0.8 g 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 of CO\textsubscript{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\textsubscript{2}-free water contained in a 150-ml beaker until the first appearance of a pink end point

Normality of NaOH soln = \[\frac{1000 \times (\text{Wt KHC}_8\text{H}_4\text{O}_4)}{204.2 \times (\text{ml NaOH})}\]

Standard sodium hydroxide, 0.1N. Dilute 50 ml of 0.5N NaOH to 250 ml with CO\textsubscript{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 75ml dist w and contg 4 drops of 1% phenolphthalein in a 250-ml beaker. Continue titrating to the disappearance of the pink color

\[
\text{Normality of HCl} = \frac{(ml \, \text{NaOH}) \times (N \, \text{NaOH})}{ml \, \text{HCl}} = \frac{25.00(N \, \text{NaOH})}{ml \, \text{HCl}}
\]

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-titrlate with 0.1N HCl to pH 8.5 (glass-calomel electrode system)

\[
\% \, \text{AN} = \frac{([ml \, \text{NaOH} \times N \, \text{NaOH}] - [ml \, \text{HCl} \times N \, \text{HCl}]) \times 0.08005 \times (\text{Tot extract}) \times 100}{([ml \, \text{Aliquot}] \times (\text{Sample wt}))}
\]

where Tot extract = 250ml, Aliquot 25ml & Sample wt 50 g


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

\[
\% \, \text{NaCl} = \frac{(ml \, \text{AgNO}_3) \times (N \, \text{AgNO}_3) \times (0.05845) \times (\text{Tot extract}) \times 100}{([ml \, \text{Aliquot}] \times (\text{Sample wt}))}
\]

where ml of AgNO₃ 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 concent 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 conc 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 Meker 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

Cool in a desiccator and weigh as total nitrates

\[
\% \, \text{Total Nitrates} = \frac{(\text{Wt of residue in dish}) \times (\text{Tot extract}) \times 100}{(ml \, \text{Aliquot}) \times (\text{Sample wt})}
\]

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 9b 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

\[
\% \, \text{ZnO} = \frac{(\text{Wt of residue after ignition}) \times (\text{Tot extract}) \times 100}{(ml \, \text{Aliquot}) \times (\text{Sample wt})}
\]

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:

\( \text{c) Gently heat the residue of step 9b over a burner until the evolution of nitrogen oxides from the decompn of Zn nitrate has ceased} \)

\( \text{d) Cool and weigh the remaining material as Na nitrate and Zn oxide} \)

\( \text{e) Treat with } w, \text{ filter the insol ZnO on a Gooch crucible, and wash} \)

\( \text{f) Dry the crucible, ignite, cool and weigh} \)

\( \% \text{ ZnO} = \frac{(\text{Wt after ignition}) \times (\text{Tot extract}) \times 100}{(\text{ml Aliquot}) \times (\text{Sample wt})} \)

where \( \text{Tot extract} = 250 \text{ml, Aliquot} = 50 \text{ml and Sample wt} = 50 \text{g} \)

11) \( \text{Sodium Nitrate. Calculate it from equation:} \)

\( \% \text{ NaNO}_3 = \frac{\% \text{ Total nitrates} - \left[\left(\% \text{ ZnO} \times 2.33\right) + \% \text{NaCl} \times 1.194\right]}{\% \text{ Total nitrates}} \)

where \( \% \text{ Tot Nitrates} = \% \text{ ZnO in step 9b and } \% \text{ NaCl in step 8} \)

12) \( \text{Sodium Salts as Sodium Sulfate.} \)

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 Meker 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:

\( \% \text{ Na}_2\text{SO}_4 = \frac{\left(\% \text{ZnO} \times 1.984\right) + \left(\% \text{NaCl} \times 1.215\right)}{\% \text{ZnO}} \)

where \( \% \text{ZnO} = \% \text{ NaCl from step 8} \)

Calculate: \( \% \text{ NaNO}_3 = \% \text{ Na}_2\text{SO}_4 \times 1.197 \) and compare the value with that obtb in step 11

IV. ACID SOLUBLE CONSTITUENTS DETERMINATION IN COMMERCIAL BLASTING EXPLOSIVES (Using Bur-Mines 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°C 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 expi 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 1.

e) Save the crucible with residue for further operations as outlined in step 1.

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 distilled water.

b) Add conc 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 filtered, while hot, thru the above alundum crucible.

e) Rinse the contents of crucible with hot distilled water, 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 desiccator and weigh.

h) Calculate % acid solubles as in step 1.

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 3c. 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 flocculent aluminum hydroxide 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.

% Aluminum =

\[
\frac{(Wt \ Al_2O_3 \times 0.52923) \times (Tot \ extract) \times 100}{(Sample \ wt) \times (Aliquot)}
\]

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_6H_4ON)_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 ppt.

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 distilled water. Dry at 120 to 140°C, cool and weigh. The ppt is Al(C₆H₅ON)₃ and contains 5.87% Al

\[ \text{% Aluminum} = \left( \frac{\text{Wt Al(C₆H₅ON)}_3 \times (0.0587) \times (\text{Tot extract}) \times 100}{(\text{Sample wt}) \times (\text{Aliquot})} \right) \]

where Tot extract = 250 ml, Aliquot = 50 ml and Sample = 50 g

6. Determination of Antocid (Calcium Carbonate)
   a) Take the filtrate in 400-ml beaker (See step 4b) and heat to boiling
   b) Add 7 ml of saturated ammonium oxalate (soly 11.8 g per 100 g water at 50°C), stir well and place on the steam bath for an hour or more. This will ppt Ca as oxalate, CaC₂O₄
   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 water to remove excess ammonium oxalate. It is not necessary to remove at this time the CaC₂O₄ which adheres to the 1st beaker.
   d) Save the filter paper with adhering CaC₂O₄ and also the filtrate in beaker No 2 for further determinations.
   f) Add 100 ml distilled water and 5 ml concd sulfuric acid to the 1st beaker and scrub the walls to dissolve any adhering CaC₂O₄.
   g) Heat to boiling and titrate to a pink color with 0.1N KMnO₄ (See Note D below).
   h) Transfer the filter of step 6d containing the remainder of CaC₂O₄ to the 1st beaker, stir and continue titration until a faint pink color persists for one min.
   i) Calculate as follows:

\[ \text{% CaCO₃} = \left( \frac{\text{(ml KMnO₄)} \times (0.250)}{\text{(ml KMnO₄)} \times (0.05005) \times (\text{Tot extract}) \times 100}{(\text{Aliquot}) \times (\text{Sample wt})} \right) \]

where Tot extract = 250 ml, Aliquot = 50 ml and Sample = 50 g

Note D: For preparation of standard 0.1N potassium permanganate, dissolve 3.250 g KMnO₄ in 1 liter of hot distilled water and allow the solution to stand 12 hrs or longer. Filter thru a fine porosity, sintered glass crucible to remove MnO₂ and store in a dark bottle.

Note E: For standardization of KMnO₄ solution, transfer 0.3 g of dried (at 105°C) Na oxalate to a 500-ml beaker and add 250 ml dilute (5:95) sulfuric acid, previously boiled for 10-15 mins and cooled to 27°C. Stir until oxalate has dissolved and gradually add KMnO₄ solution, with stirring, at the rate of 25-35 ml per minute until the appearance of a pink color. Usually 39-40 ml are required. Heat the solution to 55-60°C and complete the titration until a faint pink color persists for 1 min. Add the last 0.5-1 ml dropwise, allowing each drop to become decolorized before the next is introduced.

\[ 2\text{KMnO}_4 + 5\text{Na}_2\text{C}_2\text{O}_4 + 8\text{H}_2\text{SO}_4 = \text{K}_2\text{SO}_4 + 2\text{MnSO}_4 + \text{H}_2\text{O} + 10\text{CO}_2 \]

Normality of KMnO₄ = \[ \frac{\text{Wt Na}_2\text{C}_2\text{O}_4}{\text{(ml KMnO₄)} \times (0.05005)} \]

7. Determination of Antocid (Zinc Oxide)
   a) Before proceeding with this test, see if there is any zinc present in the acid extract: Take 10 ml of acid extract (See steps 1a & 1b) or 2a & 2b), neutralize it with dilute NaOH solution and then add 1 ml of glacial acetic acid. Pass in H₂S 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 400-ml beaker No 2 and add 50 ml of water (water-alcohol) extract (See Standard Method No 32, step 10).
   c) Acidify with hydrochloric acid, add 25 ml of mercuric ammonium thiocyanate precipitating reagent and allow the precipitation to take place overnight.

Note: For prepn. of precipitating reagent dissolve 32.0 g Ammonium thiocyanate and 27 g mercuric chloride in 500 ml distilled water. 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 10 ml of precipitating reagent in a liter of distilled water.
   e) Burn the crucible with ppt to ZnO and calculate:

\[ \text{% ZnO} = \left( \frac{\text{Wt of ppt in crucible}}{\text{(ml KMnO₄)} \times (0.05005) \times (\text{Tot extract}) \times 100}{(\text{Aliquot}) \times (\text{Sample wt})} \right) \]

where Tot extract = 250 ml, Aliquot = 50 ml 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 CCl₄ 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 CCl₄ as in step 1b and repeat operations of steps 1c and 1d.
   f) Wash the residue in the crucible attached to suction flask with 20ml hor CCl₄ 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 2hrs under 70°C, cool in a desiccator and weigh.
   h) Calculate wt loss of residue in the crucible and:

\[
\% \text{ Sulfur} = \frac{\text{(Wt loss in crucible)} \times 100}{\text{(Original sample wt = 50 g)}} + \frac{\text{wt loss in crucible}}{\text{Original sample wt = 50 g}}
\]

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 CCl₄, 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°C for 2 hours, cool and weigh.
   j) Calculate:

\[
\% \text{ NC} = \frac{\text{(Wt loss of acetone extraction)} \times 100}{\text{(Original sample wt = 50 g)}}
\]

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 additional 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 Dynamite.
d) Estimate the proportion of the constituents of absorbents found by microscopic examination.
e) The percent absorbents present is calculated 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 permissible: 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); tamarind seed, vegetable ivory & vegetable meal; woodpulp (Has long, thin, cohesive fibers).

Note B: Microscopic examination of absorbents is described in Bureau of Mines Bull 96 (Ref 2, pp74–5). For prep of glass microscope 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 slide and Petri dish to the alundum crucible of step 1a.
b) Ignite with a bumer 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.

Pierson’s Procedure D–1 (Ref 11, p1353 & Ref 17, Method D90–1) Moisture by Carbon Tetachloride 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 CCl₄ instead of CS₂, 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 1.0 g. accurately weighed, of the filtered dried expl oil (from the ether extraction into a tared 100-ml beaker. Add 10 ml of freshly prepd methylsulfuric acid (obtd by gradually adding, under the hood, 5 ml of concd sulfuric acid to 5 ml 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 40 ml of distd w and transfer the soln to a 250-ml separatory funnel. Extract with 50 ml of pure...
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 NaHCO₃ soln., followed with 25 ml of distilled water. 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 calculated from the equations:

\[
\text{\% LF compds} = \frac{\text{grams of LF compd} \times 100}{\text{grams of oil taken} \times (1.0 g)}
\]

\[
\text{\% LF compds on basis of original sample} = \frac{\text{\% Ether sol-(\% Moist+\% Oils+\% Resin+\% S)x\% LF compds}}{100}
\]

Pierson (Ref 11, p1356) gives the following equation:

\[
\text{\% Nitrocompounds} = \frac{\text{grams Nitrocompound} \times \text{\% Ether soluble}}{\text{grams of explosive oil weighed}}
\]

Colorimetric Tests for Nitrocompounds (Ref 11, ppi356–57)

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 between 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 (NH₄)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 de-nitrated to their respective alcohols by action of methanol and concd HCl. On oxidizing the residue of polyhydric alcohols with periodic acid, H₂IO₆, 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. Ethylene glycol, diethylene glycol and diglycerin do not react with H₂IO₆ 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

<table>
<thead>
<tr>
<th>Nitroglycerin Plus:</th>
</tr>
</thead>
<tbody>
<tr>
<td>NGc (Nitroglycerin) (EGDN)</td>
</tr>
<tr>
<td>DEGDN (Diethylene glycol Dinitrate)</td>
</tr>
<tr>
<td>TNDD (Tetranitrodiglycerin)</td>
</tr>
<tr>
<td>SON (Sugar Octonitrate)</td>
</tr>
<tr>
<td>SON-EGDN</td>
</tr>
</tbody>
</table>

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.
methanol soln to contain ca 0.25 g of NG and place in a tared (to 0.1 g) 250 ml Erlenmeyer flask for denitrification. 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 bromocresol 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 3 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 H₃IO₆ (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 =

(\(A-B\) x N NaOH) x 0.2271 x 1.087 x 100

where: A = ml NaOH required to titrate the sample
B = ml NaOH required to titrate 25 ml of H₃IO₆

soil 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: Bromocresol green-methyl red mixed indicator is prepd by adding 0.4 g methyl red and 0.2 g bromocresol green to 100 ml 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 H₃IO₆ in one liter of distd w, filtering thru a tittered glass funnel any insol material and storing a filtrate in a dark glass bottle provided with a glass stopper. For standardiza-

tion of H₃IO₆, 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 bromocresol 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 (Nitroglycerol) 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 H₃IO₆ Method

C = % LF Compounds

If no LF comprds 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:

NH₄NO₃, NaNO₃, NaCl, Mg(NO₃)₂, Chlorates & Perchlorates and Other Salts (such as ZnO, Ca(NO₃)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 Mg₃P₂O₇ as the final product or by titrating an aliquot of the water extract with EDTA (Ethylenediamine-tetraacetic 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 AgNO₃ 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 110⁰, 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% H₂SO₄ soln and boil the soln until the odor of SO₂ is no longer perceptible (usually 10 mins). Add 2 ml of 1:3 nitric acid, followed by 50 ml of 0.1N AgNO₃ 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 110⁰, cool in a desiccator and weigh. Save the filtrate for the perchlorate test. Calculate as NaClO₃ or KClO₃

\[
\% \text{NaClO}_3 = \frac{\text{Wt AgCl} \times 0.4078 \times 100}{\text{Wt of sample}}
\]
\[
\% \text{KClO}_3 = \frac{\text{Wt AgCl} \times 0.5202 \times 100}{\text{Wt of sample}}
\]

in which: \(\% \text{NaCl} = \frac{a}{b}\) x 1.821

\(\% \text{KCl} = \frac{a}{b}\) x 1.644

Perchlorates. Since perchlorates are not reduced by H₂SO₃, 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 Na₂CO₃, mix and heat over a bumer until a clear melt is obtd and then for 15-20 mins more. Cool the melt, dissolve in dilute HNO₃ and determine the chloride by ppt with AgNO₃ soln

\[
\% \text{NaClO}_4 = \frac{\text{Wt AgCl} \times 0.8543 \times 100}{\text{Wt of sample}}
\]
\[
\% \text{KClO}_4 = \frac{\text{Wt AgCl} \times 0.9667 \times 100}{\text{Wt of sample}}
\]

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 Bur Mines 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 Tetra-chloride 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-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 buming 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 buming 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 comds, such as SON Sucrose Octanitrate, LF (Low-Freezing) Compounds, TeNDG (Tetranitrodiglycerin), NGc (Nitroglycerol), 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 ½ to ¾ full of the Dynamite sample. The amount will depend on the expl oil content, which is determined from the ether extraction conducted as follows: Pour in 50 ml of ether, cover the funnel with a watch glass, let stand to impregnate the sample and then apply suction. Repeat with 3 additional 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 insolubles substances from the explosive oils, using a very small, fast filter paper (such as No 41 Whatman) into a rubber-stoppered bottle containing the exp oil previously obt from dem of ether extract. Store it in a CaCl₂ 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: Tetranitrotdiglycerin (TeNDG) and Diethylene glycol Dinitrate (DEGN) (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 5.0 g of the filtered expl oil into a large test tube, add 50 ml 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 distilled water and separate by decantation. Dissolve the residue in 30 ml of ether and add an excess of anhyd Na₂SO₄ to take up the remaining w. Either decant or filter the etheral soln, evaporate to dryness and place in a vacuum desiccator for one hr. A partial identification of compnts can be made on the basis of a nitrogen dem: TeNDG contains 16.19% N while DEGN 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 exp oil in methanol and add a little NH₂SO₃ (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 Cl₂O 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 ingrednts 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 benzene-ligroin. Treat chromatographically, using a silicic acid column. The NG, NGc and similar exp 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 C389 to C398) (See also Ref 18, pp4–6).

Mixture of NGc and NG, separated from other interfering ingrednts by suitable solvents or by a chromatographic method, may be quantitatively analyzed by IR (infrared) spectrophotometry, as briefly described by Pierson (Ref 11, p1363–64).

Note: The outline presented by Pierson is based on the method of W. Hoff, 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 Wrappers 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>
<thead>
<tr>
<th>Ingredient, %</th>
<th>Type I</th>
<th>Type II</th>
<th>Type III</th>
<th>Type IV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Explosive Oil</td>
<td>163% *</td>
<td>25%</td>
<td>29%</td>
<td>50%</td>
</tr>
<tr>
<td>NH₄NO₃</td>
<td>62%</td>
<td>13%</td>
<td>28%</td>
<td>28%</td>
</tr>
<tr>
<td>NaNO₃</td>
<td>10%</td>
<td>47%</td>
<td>31%</td>
<td>39%</td>
</tr>
<tr>
<td>Antacid &amp; Fuel combustible and</td>
<td>antacid are left to the discretion of the manufacturer:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Rate of Detonation, ft/sec, minimum</td>
<td>10000</td>
<td>12000</td>
<td>16000</td>
<td>16000</td>
</tr>
</tbody>
</table>

* May be NS (Nitrostarch)

Requirements and Tests:
1) Moisture Content shall not exceed 2% when determined in accordance with a suitable method that appears in a standard or textbook reference and previously approved by the contracting officer.

2) Stability. The Dynamites shall cause no discoloration of Kl-starch test paper for a minimum period of 10 mins, when tested at 82.5±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 not be lower than indicated in Table, when determined by the DuPontnie Method (which is described in Vol 3, p C311-R) or by other methods, such as Mettegang’s, described under Chronograph 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 IIIa – 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 ARMCP 706-177 (1967), pp122–24. It was never standardized nor manufactured. Therefore, no Specification was issued.


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-24B 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 conr 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 1½ 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 \(-70^\circ\text{F}\).

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 throughout 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 (5400 m/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 4.5 ft long shall cause complete detonation throughout 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 throughout 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 3 x 3 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 100 ft. The block is placed vertically with one of the 3 x 3 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 obt 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 2.0 g of sample in a tared filtering crucible and install it in a Bailey-Walker Extraction Apparatus (such as No 9-650 of Fisher's catalog) (See Fig 1), contg carbon tetrachloride. Heat on a water-bath (bp of CCl₄ 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 CCl₄ washings. Transfer the crucible to a Fisher Filterator (such as shown in Fisher catalog as No 9-788) (See Fig 2), and wash with ca 100 ml CCl₄ using suction. Transfer the washings to the 250-ml volumetric flask contg the filtrate with three 25-ml washings, fill with addnl CCl₄ to the mark and save for the TNT determination. Remove the crucible from the filterator, dry for one hour in an oven maintained at 70°F, cool in a desiccator and weigh. Save the crucible with
residue for RDX and Guar Gum determinations

2) TNT Determination by the Titinous Chloride Method. It is based on the following reaction:
   \[ \text{Hg} \text{C}_6 \text{C}_6 \text{H}(\text{NO}_2)_2 + 18 \text{TiCl}_4 + 18 \text{HCl} \rightarrow \]
   \[ \text{Hg} \text{C}_6 \text{C}_6 \text{H}(\text{NH}_2)_2 + 18 \text{TiCl}_4 + 6 \text{H}_2 \text{O} \]
   and standardization of \( \text{TiCl}_3 \) solution is based on the reaction:
   \[ 6 \text{TiCl}_3 + 3 \text{K}_2 \text{Cr}_2 \text{O}_7 + 14 \text{HCl} \rightarrow \]
   \[ 6 \text{TiCl}_4 + 2 \text{KCl} + 2 \text{CrCl}_3 + 7 \text{H}_2 \text{O} \]
   a) Preparation of 0.2N Standard Titanous Chloride Solution from 20-Percent Solution. The
   method is the same as used for prep of 0.15N soln described in Vol 1, pp A415-R & A416-L
   of Encycl, except that a larger proportion of commercial 20\% \( \text{TiCl}_3 \) soln is used
   Procedure. For each liter of soln mix 150 ml
   of 20\% \( \text{TiCl}_3 \) soln with 100 ml 38\% HCl and dilute with distilled, 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 \( \text{TiCl}_3 \) 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 distilled, oxygen-free, water
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 \( \text{TiCl}_3 \) Solution with \( \text{K}_2 \text{Cr}_2 \text{O}_7 \) Procedure. Weigh accurately ca
   0.35 g of pure dried \( \text{K}_2 \text{Cr}_2 \text{O}_7 \) (Natl Bur Standards 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 distilled
   water to dissolve the dichromate and sweep
   out the air by passing in a current of \( \text{CO}_2 \) (or \( \text{N}_2 \))
   during 5 mins. Add 50 ml of 10\% \( \text{H}_2 \text{SO}_4 \) soln
   and titrate with 0.2N \( \text{TiCl}_3 \) soln (to be standard-
   ized) from buret \( C \) of Fig 3, connected by
   tube to Kipp Apparatus \( K \). The current of \( \text{CO}_2 \)
   (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 5.32\% Ba diphenylsulfonate indicator soln, as
   specified in Method 708.1 of MIL-STD-286B
   (1957). 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-titrature the ferric thiocyanate complex with 0.2N \( \text{TiCl}_3 \) soln from
the same buret until the light blue color re-
appears. Determine the mls of \( \text{TiCl}_3 \) 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

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 TiCl₃ soln using the following equation:

\[ N \text{ of TiCl}_3 \text{ soln} = \frac{A}{(0.04904B)}, \]

where: \( A = \text{wt of } K_2Cr_2O_7 \text{ and} \)

\( B = \text{corrected reading of buret (Ref 19, p 3)} \)

The normality of TiCl₃ 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 titanic chloride and ferric ammonium sulfate proceeds accdg to the following equation:

\[ 2\text{TiCl}_3 + \text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O} \rightarrow \]

\[ \text{TiCl}_4 + \text{Ti}(\text{SO}_4)_2 + \text{FeCl}_2 + \text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 + 24\text{H}_2\text{O} \]

Prepare 0.15N ferric ammonium sulfate soln in the same manner as described in Vol 1 of Encycl, p A 415 for prepn of 0.05N soln, but make it three times stronger by using ca. 75 g of \( \text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O} \) for each liter of reagent.

For its standatization with TiCl₃ 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 \( \text{CO}_2 \) thru the flask, titrate from buret C with 0.2N TiCl₃ 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 \( A = \text{vol of TiCl}_3 \text{ soln used; } N = \text{its normality; } B = \text{vol of ferric ammonium sulfate soln used (45 ml)} \)

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 CCl₄ and dried, to the titration flask F. Evaporate the CCl₄ to dryness with a slow stream of air and then sweep the air off with a stream of \( \text{CO}_2 \) from a cylinder (or from a Kipp generator). While continuing to pass \( \text{CO}_2 \) 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 \( \text{CO}_2 \) cylinder) an accurately measured excess of 0.2N TiCl₃ 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 \( \text{CO}_2 \) 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 \( \text{CO}_2 \), 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 \( \text{CO}_2 \) from an empty flask F, and, while continuing to pass \( \text{CO}_2 \), add 25 ml of glac AcOH, 25 ml of 15% HCl soln and 20 ml of 0.2N TiCl₃ 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% \( \text{NH}_4\text{SCN} \) soln.

Calculation:

\[ \% \text{TNT} = \frac{1.261[(AN - BF) - (CN - DF)]}{W} \]

where: \( A = \text{ml of TiCl}_3 \text{ soln added to flask; } N = \text{normality of TiCl}_3 \text{ soln; } B = \text{ml of ferric ammonium sulfate soln added from buret C; } F = \text{its normality; } C = \text{ml TiCl}_3 \text{ soln added to blank; } D = \text{ml of ferric ammonium sulfate soln used for blank; } W = \text{wt of sample represented by the aliquot taken (ca 2.0 g)} \)

3) RDX Determination

Insert the filtering crucible (contg the residue after extraction with CCl₄ 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 3N sulfuric acid. The formation of a reddish-brown color indicates the presence of RDX. Some addnal 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 \( A = \text{wt of crucible with contents before acetone extraction} \); \( B = \text{wt of crucible with residue after acetone extraction} \); \( W = \text{wt of sample (ca 2 g) before carbon tetrachloride extraction} \)

4) **Gluar Gum Determination**

Percentage of Guar Gum, used since 1968 to replace cornstarch, is calculated from the equation:

\[ \% \text{Gluar Gum} = \frac{(B - C) \times 100}{W} \]

where \( B = \text{wt of crucible with residue after acetone extraction} \); \( C = \text{wt of empty crucible} \); \( W = \text{wt of sample (ca 2 g) before carbon tetrachloride extraction} \)

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):
4) C.A. Taylor & Wm.H. Rinkenbach, "EXPLOSIVES: Their Materials, Constitution and Analysis", Bull 219 (1923), pp116-118 (Physical examination of Blasting Explosives); 118-119 (Dope-size test); 119-43 (Chemical Analysis of Blasting Explosives)
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), Ausschützung (Exudation); Chemische Analyse (Asche, Kieselschuur, Nitroglycerin, Vaselin, Fett (Grease), Kamfer, Anorganische Salze, Schwefel (Sulfur), Zellulose, Nitrozellulose, Kollodiumwolle (Collodion Cotton), Harz (Rosin), Nitropolyglyzerin und Dinitrochlorhydrin) (Ref 3, pp 449–63)

**Italian.** Prove delle dinamiti: Acidità, Umidità (Moisture), Stabilità, Esadizione; Dosatura (Determinazione della nitroglicerina e della materia inerte; nitrocellulosa solubile, resine, anfora, e nitroderivati aromatici (Ref 1, pp 310–12 & Ref 7, p 465)

**Russian.** Ispytaniye dinamitov: Kislomost' (Acidity), Vlaznost' (Moisture) Stoykost' (Stability), Eksudat siya; Khimicheskii analiz (Nitroglitsazin, Nitroglikol, Pitoksilin, Interniya veshchestva, etc) (Ref 6, pp 37, 38, 94, 95)

**Spanish.** Pruebas de las dinamitas: Asidade, Humedad, Higroscopicidad, Estabilidad, Exudación; Analisis quimico [Nitroglicerina, Nitrocelulosa, Nitroclorhidrina, kieselschuur, carbón, serrín (sawdust), cuolin, etc] (Ref 4, pp 758–63 & Ref 5, pp 271–76)


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 BatMines 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 C461. 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, EnggNews 59, 692(1908) & CA 2, 245(1908) (Magazine and thaw house for Dynamites)

2a) Editor, Engg & 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 mass 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 nitrate of succinyl 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 Tetranitroglycerin are gelatinized with NC in the presence of 0.5 to 2.0% "gelatinization accelerator", such as ethylene glycol monoethyl ether)

8) IG Farbenindustrie AG, FrP 848652 & 848740(1939); CA 6114 & 5912(1941) (Low-freezing Dynamites contg glyceryl monoacryl derivatives)

9) F. Schwarzer, SwissP 228654(1943) & CA 43, 2437(1949) (Dynamite prep 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 nitroaliphatic paraffin, such as Nitromethane, Dinitoethane, 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 polyethylene glycol ether contg at least 8 carbon atoms joined together and at least 2 ethyleneoxy groups joined together. Example: polyethylene glycol 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 HeNMe and RDX distributed in a mixture of Dinitro cuene, Dinitro-tetra fluore benzene and nitrate product of sec-butylnitrobutane)

14) J. Whetstone, USP 2460375(1949) & CA 43, 2776(1949) (A granular Dynamite is prep 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$_3$)$_2$·4H$_2$O, Pb(NO$_3$)$_2$, Mg(NO$_3$)$_2$·6H$_2$O 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 rye meal 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 Cheddite & Dynamit at Liestal and Isleton. It manufd Sprenggelatine (Blasting Gelatin) (with or without NGc) and safety Dynamites, such as Telsit and Gelatine-Cheddite. c) Société Suisse des Explosifs, at Gamsen-Briague. 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) (Powder Dynamites are rendered cohesive by incorporating small quantities of polybutenes. E.g. NG 6.0, AN 80.4, Na nitrate 5.0, wood pulp 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, TransInstMining-Engs 110, 355-600 (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, wood meal 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 14651 (51) & CA 46, 11689 (1952) (Waterproofing of a Dynamite may be achieved by incorpora-
ing 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, 11705 (1954) (Colloidal Dynamite consisting of: TNT 9.0, NC 1.0, AN 52.5, urea 8.0, urea nitrate 24.2 & GuN (Guainidine 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 compg contg pelletized sulfur).

22) R. Clogau, USP 2676877 (1954) & CA 48, 1322-23 (1954) (Gelatinous Dynamite compg a surface-active agent, such as tri-ethanolamine 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-C$_6$H$_4$(CO$_2$Bu)$_2$ 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 nitrocompounds 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, FrP 1114381 (1956)& CA 53, 11840 (1959) (Explosive oils by nitrification of ethylene)

29) E.J. Russell, USP 2821466 (1958) & CA 52, 6795 (1958) [Water-resistant Dynamites prep 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 prep 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 explosion expl units resistant to hydrostatic pressure prep by heating thin metal cans contg AN 75% (at least) with usual fuels, sensitzizers 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 73886 (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 H₂S-treated ferric or ferrous ferrocyanide and their complexes)

34) F. Brown, USP 2879149 (1959) & CA 53, 11840 (1959) (A low-cost expl prep 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.J. Coursesen & 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) (Explosables castable at < 110° are prep by combining the eutectic mix containing 49% NaNO₃ and 51% Mg(NO₃)₂ with other ingredients, e.g. AN 50.05, 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%)


42) N.A. Hamilton, USP 2932251 (1960) & CA 55, 3983 (1960) (A self-contained, water-proof, expl cartridge is made from 90–96% granular AN, contg 3–7% TNT and 1–3% bindet (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 compd 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, KogyoKakuyaku-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 & BaSO₄ 5%)

46) P.E. Wilt & R.D. Nelson, USP 2968541 (1961) & CA 55, 7843 (1961) [Nondusty Nitro-starch 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 mix 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 299737B (1961) & CA 56, 1664 (1962) (Blasting expls consisting of porous AN prepbd by special process, fuels & sensitizers)

48) Watenaux & LeGall and État français, FrP 1242324 (1961) & CA 56, 2628 (1962) [Permissible expls prepbd 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% NaNO₃)

50) W.H. Eckels, USP 2999744 (1961) & CA 56, 1664 (1962) (A plastic expl prepbd 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, NaNO₃ 36.4, wood pulp 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, NaNO₃ 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, prepbd 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 prepbd 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 prepbd by intimately mixing AN >60, a nitrate of an alkylamine (such as that of mono-, di-, or triethanolamine ≥ 8, H₂O up to 15% and dispersing throuth 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. Zentman & Th. J. Reid, BritP 883918 (1961) & CA 56, 11875 (1962) [An explosive prep by stirring 24.5 parts granular poly (Me methacrylate) of mol wt 1000000 with 75.5 parts 98% HNO₃ 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 NaNO₃), sawdust & Fe₂O₃ are suitable for mud-capping and for use under water]

60) A. Berthmann & E. Ludolph, GerP 1096813 (1961) & CA 56, 3716 (1962) [Waterproofing of Dynamites by octadeyl 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 prep by AN shielded by coating with a mineral fat melting above 90° and solidifying at -20°. One of such fats was prep 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 sensitzers]

63) Ph.G. Butts & C.R. Fignerhood, GerP 1109777 (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 & a ballistic modification agent 2–10% by wt. The agent may be GUN, NGU, RDX, or 2,4-Dinitrophenyloxyethanol, single or together. The binder may consist of a mixed polymer of butadiene with acrylic acid, acrylonitrile or vinylpyridine]

64) E. von Holt, GerP 1110562 (1961) & CA 57, 10098 (1962) [An expl prep by mixing 100 parts NC (of N=12.2%) (previously moistened with H₂O) with 40 ps TeNMe, 20 ps Diglycol Dinitrat & 0.5 part stabilizer]

65) B.B. Enoksson, GerP 111079 (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'-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, NaNO₃ 56, NH₄Cl 32.5, carboxymethyl cellulose 1, and a synthetic sillic acid (Hi-Sil 233) 0.5% gave a semigelatinous, stable expl safe against coal dust and mine gas]

68) A. Berthmann & E. Groesch, 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 (1961) & CA 57, 6201 (1962) [Gelatinous expls prep by blending NC with bis(methyl nitro) 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, KogyoKaiyakukyokaishi 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)


73) Hercules Powder Co, BritP 893590 (1962) & CA 57, 1142 (1962) [A Blasting Explosive suitable for underwater use is prep by mixing 25 parts NaNO₃ with 16.8ps H₂O & 2.1ps of
Na carboxymethyl cellulose, followed by heating to 50–60°C (to remove air bubbles), cooling and mixing with 41.9 ps AN & 14.7 of a fine grade propellant]

74) Th. Lan, GerP 1125333 (1962) & CA 57, 11441 (1962) (Plastic safety expls contg Tri-nitroaniline and NGc gelatinized with NG)

75) A. Berthmann et al, GerP 1123963 (1962) & CA 57, 2489 (1962) (Water-contg expls prep as H₂O-in-oil emulsions of NG or TNT, and with 1% emulsifier and stabilizer added. E.g. 50 ml NG, 50 ml H₂O, 0.5 g Zn hydroxystearate & 0.5 g Pyroxylin)

76) H. Aarfot, NorWP 99360 (1962) & CA 57, 8792 (1962) [Explos consisting of AN & fuel oil, part of which may be replaced by sawdust, soot or nitrated hydrocarbons, Al stearate may be used as thickeners of fuel oil. These expls may be incorporated into NG based expls]

77) A.G. Iroda, BelgP 613336 (1962) & CA 57, 14045 (1962) [Expl prep 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)


80) E.M. Scott Jr, USP 3016488 (1962) & CA 58, 407 (1963) (Explos provided by mixing 70 parts uncoated AN prills, 30% of NaN₃ and 6% 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 NaN₃ 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. Enochson, USP 303903 (1962) & CA 57, 1298 (1963) [An expl or propult compn is prepd by coating AN with a C₁₂₂₀ 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 304412 (1962) & CA 57, 12780 (1962) (AN-cyanide expls, such as contg AN 81, K₃Fe(CN)₆ 4 & asphalt 15%)

86) S.R. Brinkley Jr & G.V. von Eisle, 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) (Explos 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) (Explos aliphatic and aliphatic-aromatic compns contg trinitrophenyl groups)
90) C.O. Davis et al, USP 3052578 (1962) & CA 57, 15404 (1962) [AN-based nitrocarbonate-type blasting explosive, which provides high efficiency in the ability to eliminate all voids in a bore hole, are prepared by blending AN 80, said aqueous AN 15 & petroleum oil 5%. A small amount of N(C₂H₄OH)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 obtained by allowing droplets of a molten mixture of AN & NaN₃ (or other inorganic nitrate) to fall into and churn a hydrocarbon fuel which is a liquid below the mp of the nitrates. As a result of this the droplets crystallize inwardly from the surface to form gels, the outer surfaces and inner cavities of which are covered and filled, resp., with fuel. This fuel is pulled thru the entirely impervious walls as cooling and shrinking takes place. Examples of fuels: kerosene and DNT]

92) O. Osborn et al, USP 3053710 (1962) & CA 58, 6642 (1963) [Mixtures of Mg hydride (up to 50%) with inorganic 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 AN & PETN 7-20, AN 75-90 & combustible matter 3-10%. One quarter of AN may be replaced by NaN₃]

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 prepared by mixing 2 moles hexamethyleneetramine, 1 mole Al(NO₃)₃.H₂O and 40% KClO₃ 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 containing particulate NC & AN with sufficient water to give such viscosity that the particulate material remains in suspension and hence in uniform distribution throughout 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, Tetly, PETN, RDX or HMX up to 25% AN may be replaced with alkali metal nitrates, such as NaN₃]

97) F. Frutiger, BelgP 614511 (1962) & CA 58, 1298 (1963) [Plastic AN expls contg no NG but a plasticizer (such as MNBz, DNBr 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) [Desensitization of NG or other nitric esters by polyglycols]


104) R.W. Van Dolah et al, Ibid, pp 77-89 & CA 58, 5444 (1963) (Fumes from AN-hydrocarbon mixtures)

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 NaClO₃ 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, JAN 0.1, AN 71, wood flour 1 & Fe₂O₃ 0.2%].

110. ZakladyChemiczne 'Krywald', PolP 45, 579 (1962) & CA 58, 8846 (1963) (Rock Blasting expl, such as contg AN 76, ferro-silicon 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 crystals with liquid fuel to above the crystal transition point. Upon cooling, fissures form in the AN particles and the liquid covers these new surfaces, as well as the old ones and this causes the improvement. E.g. AN 54.9, NGc 22, Colloid Cotton 1, NaNO₃ 10, DNT 6, TNT 5, wood flour 1 & Fe₂O₃ 0.1%).

113. Z.G. Pozdnikov, Vzryvnoye Delo, Nauchn-TekhnGomObshchestvo, 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 20/N390 & CA 61, 6849 (1964) (Modern Russian nonpermissible explosives and methods for their improvement) [included are Ammonites No 6, 7, 9 & 10: Igdonits, Granulits, and Granulated Granulits. They are described later in this Volume].

115. M. Aoki & Sh. Motoya, JapanP 4444 (1962) & 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 (1962) & 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 H₂O 8, gum arabic 10 & 150 mesh NaCl to form a paste. The paste is then dried at 50° & 30mm for 3 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 Explosives consisting of an HE (such as TNT or RDX), sand AN & guar gum with its cross-linking agent borax. Part of AN can be replaced by NaNO₃).

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 propllt) 23-75% oxidizing salt (such as AN with/without NaNO₃), 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 922939 (1963) & CA 58, 13705 (1963) [The crys water-sol ingredients of expls are waterproofed by spraying their surfaces with a soln of hydrophilic materials (such as CaCl₂ or ZnCl₂), followed by spraying with a soln of ionic waterproofing agents (such as stearic acid, NH₄-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 prepared by treating AN expls with a soln containing 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 detonation velocity of AN expls are obtained by heating AN crystals to ca 85° and then cooling them to ca 25° with agitation, to achieve crys 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 prep from fertilizer-grade AN prills (with or without NaN₃O₃) and small amts of fine Al, Fe₂O₃, 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 NaClO₃ (or KClO₃) & PhNO₂ with atomized Al & PbO₂ are incorporated in mixt of above patent]

126) E. von Holt, GerP 1147159 (1963) & CA 59, 4970 (1963) [Homogeneous solid expls prep 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 polyaline nitrates, such as NG or NGc. Al grindings may be incorporated]

127) H. Riedl & H. Schluster, GerP 1148924 (1963) & CA 59, 3712 (1963) (Non-exudable Gelatin Dynamite is prep 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 prep by mixing of 8-10 mesh fertilizer-grade AN 58-83, Driver's Liquid (and soln of AN in anhyd NH₄) 14-42 & 0.5-5%. Spensol D (a soln of AN 60, NH₄ 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 prep 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, NaN₃O₃ 22-24, TNT (RDX or proplnt) 28-32, water 8-15, a gelling agent (such as polycrylamide) 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 prep by mixing highly dispersed AN with pectized sodioseulose or NC in volatile solvent, and evapg solvent with simultaneous granulation of the mass]

132) D.L. Coursea & 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 22, NaN₃O₃ 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(1963) & CA 59, 13763(1963) (Powdered AN expels prep by mixing ingredients, such as AN 60, NaN₃ 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 prep by heating nonporous pellet 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 for suitable blasting purposes)

135) J.R. Hradil & M.F. Hradil, BritP 936042(1963) & CA 59, 13765(1963) (Exp compn contg a light metal sensitiz. E.g. AN prills 50.7, Mg billet scalings 23.1, coarse Al chips 17.2 & urea 9.0%)

136) J.D. MclIvaine, USP 3108917(1963) & CA 60, 1534(1964) (TNT-tetraborate gelled, aqueous explosive slurry which does not gel up to 24 hrs after prep is provided by a mixt of 15–40% particulate TNT, 60–40 Comp B or smokeless propplt; 31.5–77% granular AN (alone or in admist with NH₄ chlorate or perchlorate); 8–26% water; 0.1–2% monogalactan and 0.002–0.2% (calcld as Ba₂O₃) 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. Tamoishi & K. Oishe, JapanP 161487(1963) & CA 60, 7866(1964) (Conventional Ge-latin-Dynamites show a decrease in rate of deton and propagation of deton when the pressure of the surrounding medium becomes higher than 5kg/cm³. Incorporation of 1.5 to 2.5% of Fe₂O₃ pdr renders Dynamites detonating perfectly at pressures far higher than 5kg/cm³)

139) W.A. Gey & R.W. Van Dolah, USP 3110640(1963) & CA 62, 1508(1965) [Exp mixts of 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 NH₄ perchlorocaprylate, an ether of dihydroperfluorobutanol, or poly(chlorotrifluoroethylene)]

140) W.O. Urenbach & L.L. Udy, USP 3113059(1963) & CA 60, 7866(1964) (Slurries contg Al, water, AN & other ingredients, such as NaN₃, TNT, ferrophosphorus, smokeless proplt, 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 NH₄ 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 C₈-18 non-aromatic hydrocarbon oil, 5 ps DNT oil and 3 ps MNT oil)

143) I. Hayashi & T. Sato, JapanP 161477(1963) & CA 60, 9094(1964) (A gelatinous Dynamite can be prep 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 prep requires less time than the conventional method)

144) T. Sato, JapanP 3445(1963) & 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(1963) & CA 60, 9094(1964) (Commercial non-NG expls are prep by mixing 0.5% polyethylene glycol stearate 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 5951(‘63) & CA 60, 7866 (1964) [AN-fuel expls obtd by repla-
   cing part of the oil by phenol, cresol, xylene, aniline, etc]

147) I. Hayashi & T. Sato, JapanP 16147(‘63)
   & CA 60, 9094 (1964) [A gelatinous expl prep-
   ed by dippng 1.2 parts of NC in ca 1 part emul-
   sion consisting of water, nonionic paraffin & surfactant
   (such as Tween). After mixing the product was kneaded with AN 63.8, starch
   or wood fllr 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 incorp
   orating in fomulations contg AN, Amm
   perchlorate, Na nitrate, nitrocompound, etc
   up to 5% of N,N'-methylene-bis-amide or a
   methylolamide of a C₁₂₋₁₆ 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
   MeNO₂ (preferred), EtNO₂, PrNO₂, BuNO₂,
   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) (Explos which
   can be prepd at the site and poured into a
   borehole are provided by mixts contg lower
   nitroalkanes (such as MeNO₂, etc) and a sensi-
   tizer (such as aralkyl amine, etc). Absorb-
   bents (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, NH₄SCN 8, & Diesel oil 2%
   or AN 86 & NH₄SCN 14% Omission of NH₄SCN
   resulted in compns not cap-sensitive)

153) Nitroglycerin Aktiebolaget, BelgP 628844
   (1963) & CA 61, 1701 (1964) [Explos having
   little physiological effect and low impact sen-
   sitivity contain large amts of 1,2- or 1,3-
   propyleneglycol Dinitrates (in lieu of NG &
   NGc) and of Ams & Na nitrates, together with
   small amts of NC, DNBr (or DNT), wood flour
   & chalk]

154) N.S. Bakarevitch, VzyrnoyeDelo, Sobmik
   No 52/9,189-9(1963); ReferZhKim 1964, Abstr
   No 7/433 & CA 61, 13116 (1964) [Sensitization of
   Russian permissible AN expls with liquid
   nitro esters such as 60:40 mixt of NG & DEGN]

155) Norsk Sprangstoffindustri A/S, NorwP
   102157 (1963) & CA 62, 8926 (1965) [Powdered
   expls prepd by coating AN grains with fusible
   organ compds (such as a paraffin, wax, resin,
   aromatic or aliphatic nitrocmpd, nitramine,
   etc), previously heated to above their mp's.
   Cooling was achieved with air at 20°]

155a) I. Fukuyama & T. Kai, KogyoKayaku-
   Kyokaishi 24(3), 140-47 (1963) & CA 63,
   6677 (1965) (Two types of NG contg expls
   with the salt pairs NH₄Cl/KNO₃ and NH₄Cl/
   NaNO₃ 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 NH₄Cl/NaNO₃ 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 an 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) [Explos comps 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) 2–33%].

159) Dow Chemical Co, BritP 952837 (1964) & CA 61, 528 (1964) [Explos comps contg ammonia, such as 85 parts of a soln (consisting of AN 69.8, liquid NH₃ 23.8 & water 6.4%), 7.5 ps Mg shavings and 7.5 ps Al shavings].

160) E. Saerze, GerP 1167615 (1964) & CA 61, 4144 (1964) [Explos 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. Carlevalo, 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, NaN₃ 15.0, ferrophosphorus 20.0 & guar gum 0.5%].

162) J.S. Logan & J.A. Zaslowsky, USP 3135635 (1964) & CA 61, 5445 (1964) [Explos compn prepd by tumble mixing 186.2 g AN (100% <40-mesh) with 13.8 g yellow solid (100% <100-mesh), obtld in pyrolysis of boron hydride B₂H₆].

163) Ch.M Saffer Jr, USP 3135637 (1964) & CA 61, 5446 (1964) [AN-aliphatic hydrocarbon-urea clathrate explos].

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 autoexaction 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) [Explos contg AN powd (coated with ca 0.1% of mono- or di-octadecylhydrazine, stearylhydrazine, etc) 100 parts, NG c 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% Tetrytrotro-2,3,5,6-dibenzo-1,3a,4,6a-tetrazapentalene or Tetrytrotro-2,3,4,5-dibenzo-1,3a,6,6a-tetrazapentalene and 2–5% TNB or TNT. Other ingredients, such as KClO₃ or PbO₂ may be present].

168) P.H. Cardwell, GerP 1171786 (1964) & CA 61, 8124 (1964) [Sed soln of AN in liquid NH₃ 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) [Slurred 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 NaN₃)].

170) A.H.B. Weeks, CanadP 688577 (1964) & CA 61, 9350 (1964) [A Blasting Explosive consisting of AN (or its mixt with up to 45% NaN₃) 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, NaN₃ 22, TNT 15, water 12.7, guar gum 1 & sugar 4%].

172) T. Yoshida et al, KogyoKenkyuKyokaishi 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) [Explos 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 & Chem Inds Ltd, BritP 970975(1964) & CA 61, 15924(1964) (Expl compl prep 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 amount of water is prep by FGAN prill coated successively with paraffin, a mixt of fissionite with Al 145 Petroleum Wax, mica and Al)

177) J.S. Logan & J.A. Zasłowski, 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) (Slurred Blasting Explosives contg AN & Al, such as: AN 51.7, Al pd 4.0, NaN3 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 compl contg pyridazineinedione combustion catalyst)

180) C.W. Vriessen, USP 3155552(1964) & CA 62, 395(1965) (A castable expl compr provided by curing a mixture contg 50-80% powdered hydrazine nitrate, 13-40% binder (such as a polyester of mol wr 500-5000 derived from polyhydric alcs and polybasic acids), and 3-10% curing agent (such as a diglycicyd ether of bisphenol A or F), powd metals (such as Mg, Al, Ti, etc) 5-15%, and minor proprn 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 2,4-dimethylenebis(2-chloroaniline), n-phenylenediamine & tris(dimethylaminooethyl) phenol may be present also)

181) Imperial Chemical Industries Ltd, Neth- Appl 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 w 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 prep 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, m-MNT, DNT, etc). The solids are introd 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 15-25 ps Al powd, can be initiated by a bridge wire)

184) A. Koenig, FrP 1358222(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 1-4 ps colloidal grease of up to 130° Engler viscosity (such as naphtha or petroleum thickened with stearic acid etc) and glycerol which contains 1-4 ps 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 prep'd by heating to 115°C 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 NH₄NO₃ 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-ethyhexylic acid was added to kerosene and dissolved by stirring and heating to 120-150°F. To the jelly-like soap obt'd 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. Necker, 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(hydroxy-methyl)-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 HNO₃, AN and an insensitive carbonaceous fuel which is immiscible with HNO₃ 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) [Slurred Blasting Explosives which remain pourable up to 24 hrs after mixing are prep'd 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 Na₃Cr₂O₇, KCr₂O₇, ZnCrO₄ or BaCrO₄) 0.005-0.5% of cross-link delaying agent (such as tannic, 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 prep'd 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°C 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) [Explos contg 20-80% urea & 80-20% AN are safe and easily handled]

196) K. Malkovsky & R. Meckir, 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. Souetag, 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°C with an inorganic nitrocompd]

197a) Institut National des Mines, BelgP 645374 (1964) & CA 64, 4856 (1965) [Ion exchange safety expl: alkali metal nitrate & NH₄Cl in molar ratio of 1:1, 1:1.5 & 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, (CH₂)₆N₄·2NH₂NO₃, mp 122° and sol in org solvents can be prep'd 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 crys of above complex. The crys 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 (CH₂)₆N₄·2NH₂NO₃, prep'd by cryst from ether & or melts, is a weak and insensitive explosive. When hammermilled and boostered with a Tetryl pellet in a steel tube a deton vel 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 parts 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. Hadel, USP 3177102 (1965) & CA 63, 426 (1965) (Solid expl compns produced by mixing 70–73% by wt AN in soln with NH₃ 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, or -m, or p-MNT 15.5 & wood flour 6.2%; c) K nitrate 79.9, PbNO₃ 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. Rinkerbach & 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 NaNO₃ 20.8, ZnO 0.3 & mineral oil 0.3%).

205) S. Hodgson, CanP 707268 (1965) & CA 63, 1650 (1965) (Blasting expl prep'd 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 prep'd by mixing 6 parts of a light fuel oil with 94% high-density microprills and adding 5% 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% CH₃CHCOOH (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 RCln with n=1 to 10) mixed with a C₁₀ hydrocarbon). For greater expl power, a nitroalkane (such as MeNO₃) may be introduced into the sensitizing mixt. For example 83.6 parts prilled AN, mixed with 16.4 parts liquid sensitizer (consisting of 63% ClCH₂Cl₂, 20% MeNO₃ & 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 scalings, 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 obt. Part of the AN may be replaced by another expl

210) Jack Linz, 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-aminoalkylmopholine (combustion catalyst having a C<sub>1-4</sub> 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 nitrophenyl 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 mix consisting of 1-10% of the Na salts of methyl- (I) and dimethylnaphthalenesulfonic acids and 90-99% of silico-alumina (II) contg SiO<sub>2</sub> 60-80, Al<sub>2</sub>O<sub>3</sub> 7-14 & Na<sub>2</sub>O 3-10%. Thus 5 parts I and 95 parts II were dry-mixed with rapid agitation and the mix sealed and stored for 24 hrs. When 3 pts of this mix were tumbled 10 mins with 97 pts AN, the resulting mix remained free-flowing during a prescribed caking test]

212) Canadlns, Ltd., BritP 1001216 (1965) & CA 63, 11242 (1965) [Antistatic sensitized AN/Fuel Oil or AN/FO/Metal compns are obt by incorporating a quaternary ammonium salt contg ≥ 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 metalized 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. Criap, USP 3202556 (1965) & CA 63, 12964 (1965) [Blasting expls contg gel-galactomannan gum are described. The gum is prepd by stirring 2 parts guar gum into 198 pts water until the gum is uniformly dispersed and is free of lumps. Antimony oxide (0.2 pts) is then stirred in, followed by 2 pts 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) ≥ 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, NaN<sub>2</sub>O<sub>3</sub> (14-mesh) 29.4, NaNO<sub>3</sub> (90% thru a 20-mesh and 50-75% thru a 100-mesh screen) 11.0, corn flour 2.9, S 5.0 & guar flour 1.0%]

215) Canadlns, Ltd., BritP 1002671 (1965) & CA 63, 17791 (1965) [An improved slurred, non-segregating 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 (mangoolactans, 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. Zaslawsy, USP 3214307 (1965) & CA 63, 17791 (1965) [Blasting gels which have low sensitivity and are substantially devoid of noxious fumes can be prepared by successively mixing into 40-70 parts AN (as a 50% aq soln), K₂Cr₂O₇ 0.5-1.5, guar gum 1.0-3.0, powd Al, Mg or their mixts 10-25 and water 20-40 pts. Alternatively, the guar gum thickener may be added stepwise to permit its hydration. The mix 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 NaNO₃ 24.2, bagasse 1, "Hydroseal 38" 3, ZnO 0.3 & water 16 pts. Density 1.47, ballistic pendulum 9.5, sensitivity in 1.25-in pipe 3 gm PETN, rate of deton at 1.47 in 2.0-in pipe 4830 m/sec, and crater value (cubic ft earth moved/lb slurry) 10.2]}

218] C.L. Schwager, USP 3222322 (1965) & CA 64, 4857 (1966) [Exp slurry: Nitrocellulose 28.2, AN 45.1, NaNO₃ 15.5, Hake Al 1.9, No 5 fuel oil 0.3, Na carboxymethyl cellulose 0.7, guar gum 0.7, ZnO 0.8, anhyd Na₂SO₃ 1.2 & water 7.7% is prepd at 60°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 >3000 m/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 comp consisting of a major amt of 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. Seguitti, IndMinerari (Rome) 16, 289-90 (1965) & CA 64, 7959 (1966) [Uses & props of AN-oil expls: Max expl effect is obtb from mixts contg 5-8% combustible oil & grilled, spherical AN with 85% being less than 30 mesh & 70% less than 80 mesh. In prep 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 20 mm. A mixt of AN 60 & NaNO₃ 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 obtained by slurrying with water but stronger fuses are reqd for deton]

221] T. Seguitti, 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 1 kg AN-oil (5.7%) mixt, 20 liters of CO and 4 liters of NO₂ are liberated; where as 1 kg of Dynamite liberates 7 liters of CO & 4 liters of NO₂]

222] W.E. Gordon, BritP 1014071 (1965) & CA 64, 7962 (1966) [Exp compn: AN 90, hexamethylene tetramine 9 & abietic acid 1% when melted at 145°C, 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 18700 ft/sec at d 1.24 gm/cc to 13400 ft/sec at d 1.44 gm/cc]

223] W. Dick & O.A. Gorrton, BritP 1016462 (1966) & CA 64, 9502 (1966) [Safety expl: NG 8.5, NC 0.1, AN 10, NaNO₃ 36.5, NH₄Cl 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 polymethylacrylate added to a 60/40 NG/Nitroglycol mixt having a viscosity of 11 cp at 20°C pve after 90 mins a completely transparent product having viscosities of 450, 2100 & 4250 cp, resp]

225] T.D.S. Schott, JournInformCorpsGrasInd-Chim, HuilesInd, Acides Gras, Lipochem, Paris 1965, 73-81; discussions 81-3 (in Fr);
& CA 64, 12227 (1966) (A review of application of fatty amines as lumping inhibitors for explos., 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: prepbd by adding AN to 13 parts HCONH₂ to form a supersatd soln at 25°, guar gum 1, water satd with AN 12, NaNO₃ 10, finely divd Al 20, AN 20-25 parts, followed by 400 cc of a mixt contg 200 cc of AN in liq NH₃ and water to yield a very viscous liq of d 1.2 gm/cc)

228) K. Ito, Kogyo Kayaku Kyokaishi 26(3) 140-41 (1965) & CA 64, 17344 (1966) (Sensitivity of Kuro(Black) Carlit: In Japan, comm explods contg NH₄ClO₄, ferrosilicon & wood meal are called Kuro(black) Carlit. Surface activated NH₄ClO₄ with surfactants or a mixt of NH₄ClO₄ & Na₂Cr₂O₇.2H₂O has a higher rate of decomn and reactivity. Consequently, the deton rate & sensitivity to initn of Carlit contg such NH₄ClO₄ are larger than those values of Carlit without such NH₄ClO₄. The effects of such NH₄ClO₄ 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 propncs. Polymers used include: poly(Methacrylic acid), 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 NH₄Cl-KNO₃ compns) are obt'd by the addn of a halogenated org inhibitor (such as hexachloroethane, hexachlorocyclohexane, DDT, chloridron & iodoform). The reaction betw the inhibitor and the oxidizing component produces an inorg halide which inhibits the methane-air reaction. Adjn 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, NH₄Cl 27.0, KNO₃ 59.75, C₃H₆Cl₁₀ 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 2350 m/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 5590 m/sec)

232) Poudrieres Réunies 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, NaNO₃ 54, NH₄Cl 33.5, Ca stearate 0.5 & other additives 2.5%; its unconfined deton vels in 30 and 90 mm chges were 1665 and 1544 m/sec, resp. A similar KNO₃-based compn had 1500 and 1340 m/sec)

233) SERADEX, BelgP 658124 (1965) & CA 64, 4857 (1966) (Safety expl: NG 11, carbohydrate 5.7 & AN/NaCl intercrys mixt 83 parts. This Dynamite did not cause expln of atm when fired in CH₄ & coal powd, at 3% in a small mortar or at 9% for a suspended chge of 1.5kg)

234) R.M. Kaltenebach, BelgP 659035 (1965) & CA 64, 522 (1966) (Low-density expl prepbd 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/Nitroglycerol (40/60) 9, inert material (silicic acid/Zn stearate (0.55p/0.15p) 0.7, KNO₃ 58.9 & NH₄Cl 31.4 parts. The detonation 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 40–93 preferably at least 60% particulate AN (consisting of large and small particle of sizes given in patent), 10–50 preferably 5–25% 1600y 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 prep'd from the above materials and contg 20% Al was fired under water, the wave received at the sensor had an initial pressure of ca 540 kg/sq cm; this fell to 70 kg/sq cm in ca 0.9 millisecond and to 0 in slightly less than 2 milliseconds. A similar compr contg no Al produced a pressure of ca 420 kg/sq cm which fell to ca 70 in 0.4 millisecond and to 0 in 2.1 milliseconds]

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, NaNO₃ 0–38, NG proplnt 0–55, NC proplnt 0–38, TNT 0–16.6, water 8.8–14, and surfactants (nor named) 0.8–1.2%; sp gr 1.50–1.75 g/cc. Nitrol is a powder 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, MineraMet (Mexico) No. 35, 93–132 (1965) (Spain) & CA 66, 2947 (1967) [Some general notions on explosives and the role they fulfil in the seismic process]


241) J.D. Ferguson, USP 3235423 (1966) & CA 64, 12455 (1966) [Stabilized ag slurry blasting comp: Ethylene glycol, (CH₂OH)₂, 18 was dissolved in water 15, pine oil 0.1 stirred in & guar gum 1 part and a trace of CH₃COOH added. This was followed by successive addn of ground AN 8.9 fine NaNO₃ 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 detonation 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. Burtler, USP 3235424 (1966) & CA 64, 12455 (1966) [High density blasting expl: AN 25.96, NaNO₃ 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 comp of d 1.6 g/cc and detonation rate 5240 m/sec in 2.5-in diam pipe (after 24 hrs under w at 300 psi gm). Another mixt AN 11, NaNO₃ 23, proplnt 30, water 10, guar gum 1 & ferrosilicon 23% had a calcd d of 2.01 gm/cc but reqd a 500 gm Pentolite booster for detonation 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, NaNO₃ 8.4, water 16, guar gum 0.7, propellant (NC contg 12–13.5% N) 25.0, NaHCO₃ 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 NaNO₃ 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 20 psi thru 1.25 in 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 5.5 km/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, tetra-ethylammonium or morpholine) is used, but up to 40% may be used with Nitromethane. A modified Trauzl Pb 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-vinylpyrrolidinone was subjected to y-ray irradiation at 0.16–0.32 megard/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 azodisobutyronitrile 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 prep 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 94.0p AN prills, and 1.0 p 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, Z.ErzebergbauMetallhüttenw 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)


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 US-BurMines. 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 amounts increased the shock sensitivity of AN-fuel oil mixts. Transition to detonation was obt 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 charge several diam away.


257) J.S. Wilson et al, USP 3237189 (1966) & CA 66, 2948-R (1957) (Water-resistant grease-like coating for blasting expls was prep by mixing 562g of AN prills and 5.9g of powd AI 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 NH₄OH (28% NH₄) was added. The resulting product was a pourable slurry of ca 1.2g/cc. A sample of slurry placed in water for prolonged period remained unchanged.)

258) H.R. Nicholls & W.J. Duvall, USBurMines-RepInvest 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 charges 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 crystals (79-91.2% parts) encased by a fuel (20-8.3% pts) 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, Ammon T & polyisobutylene. The compns contg coated Al possessed improved sensitivity to deton with densities up to 1.43. Generally the coatings were applied in amounts of 2-45g to 4500g powd Al. Thus, a compn of AN 37.8, NaN₃ 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 diam long. Such mixts sustained deton in chges as small as 2.5 in diam, while mixts contg uncoated Al failed at 4 in.]
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 Propint for TNT; Na 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 expst 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) CanadInds Ltd, Brit P 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 C1-6 secondary and tertiary aliphatic amines and C12-21 aliphatic acids); up to 0.5% acid acceptor (such as Ca, Na or Amn carbonates and Et3N), 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. Mecri, 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 rests 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 slurred 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 NH4Cl 20, powd AN 47 & poly(vinylicloride) molding powd 3%, followed by heating to softening point and pressng 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 expls 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 conng 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, NaNO₂ 10, DNT 15, NG 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 prep'd on the site by mixing 92-94% granular AN with 8-6% of a pasty suspension of fine Al, Si, Zn, Sb, Zr or Sb₂S₃ 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 flp ca 72°). The paste congt 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 soin 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 & 6.8x10³ 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 MeNO₂ or its mixt with a lower alc. A hydrogen-ion indicator was incorprated 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 84pts AN, 16 pts MeNO₂ (or its mixt with MeOH or iso-ProOH), which contained 0.0065% by wt of diethylaminobenzene. 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 sinergetic sensitizing efct of I & II is obt'd 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 obt'd]

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 prep'd by reaction of an org polyisocyanate with a nitric acid partial ester of a polyhydroxy compd including a component contg ≥ OH groups reactive with the isocyanate. For example, equal parts by wt of anhydroeneheptitol 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 prep of expanded graphite are given. For making such expls, an aq slurry was prep 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 prills and the remainder in soln]

278) C.W. Eilo, USP 3261732 (1966) & CA 65, 10419 (1966) [The prep is given of a novel aq slurry blasting agent which is stable when particulate Al and an AcOH-ZnO stabilizer are present. For its prep are mixed: propelallant 30 parts, water 18.0, pine oil 0.1 & ZnO 0.033. Then one-third of 21.9 ps AN prills & 1.0 guar gum are added followed by the remaining AN and 13.5 ps Na nitrate. Finally 14.0 ps Al are added followed by 0.033 ps 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 2,268 kg 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 4.0%. 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 Gluechau 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. Lebailif, FrP 1429285 (1966) & CA 65, 12058 (1966) [A compn, whose combustion velocity can be controlled is obt 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 Nittate-Carbon), its expl properties, and uses 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
NaNO₃), and halogenated hydrocarbon (such as CCl₄ 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, NaNO₃ prill 25.1, MeOH 4.9, gelled CCl₄ 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 ~0.86 & total energy 0.437.

285) J.F. Craig et al, Brit P 1035290 (1966) & CA 65, 15144 (1966) (Addn to Brit P 1002671 & CA 63, 17791c) [Slurred blasting compn stable in storage, consists of an aq slurry of inorgan oxygen-donor salt(s), particulate org exsp(s), gel-forming polysaccharides, finely divided Al, and small ams 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, ethylene glycol 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, Brit P 1037567 (1966) & CA 65, 13450 (1966) (Addn to Brit P 990706) (See NethAppl 6601151, CA 67, 3877b) [Exp compns prepd by mixing particulate AN with a phenol and a HCHO source, carrying out the condensation and evap the resulting water. The condensation could be accelerated by raising the temp and by addg p-CH₃C₆H₄SO₃H. Inhibitors (such as urea, MgO or (NH₄)₂PO₄), 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 propelnt 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 pressd 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 prep 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 HNO₃ soln of K, Na or Amm nitrate, a C-based fuel not miscible with the HNO₃ 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 HNO₃ soln 30.0 parts, AN 47.0, NaNO₃ 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% 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 boresholes with free-running explosive, such as consisting of 30 parts AN, mechanically mixed with 6 parts Diesel oil contg 1% "Marvelan" (which served as an
antistatic) (Comp 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 decompt 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 proportions that the resultant cryst lattice contained 0.5–2.0% of such salts isomorphously substituted therein. Thus a cocrys. 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 cocrys 98:2–Amm perchlorate:K permanganate was 75 cm)

292) DynamitNobelAG, NethPatAppl 6516252 (1966) & CA 65, 18418 (1966) (A ternary mixt of NH₄Cl, alkali nitrates and alk-earth carbonates in an approximate molar prop 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, NGe 3.48, KNO₃ (78% <0.1 mm) 41.28, NH₄Cl (65% <0.1 mm) 36.40 & CaCO₃ (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 (KNO₃ 59.71 & NH₄Cl 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 addct having 3–9 ethylene oxide units, quaternary cationic, amine cationic, anionic or nonionic), and 0.01–2.0% of at least one thickening additive (gel-forming mannogalactan, water-swelling cellulose ether, Na silicate, colloidal SiO₂ or Al₂O₃). 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(NO₂)₂CH₂OH where R & R' are lower-alkyls or hydroxyl-alkyls (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 nitro-alkanol-water soln to the AN previously 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 nitr rate such as NaNO₃ and addn of S in such a way that the NaNO₃/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 NaNO₃, 0.1 stabilizer, 1.5 guar gum, 2.1 particulate Al, 9.0 sulfur (NaNO₃/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 3287753 (1966) & CA 66, 4576m (1967) (Slurry blasting agents contg nonexplosive liquid fuel are provided by suspending a solid oxidant (such as NaClO₃ or NaClO₄ 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 als, polyhydric als & ketones or an aq dispersion of nonexp]
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, add of a premix of NaClO$_3$ 41.5, AN pill 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 add of stabilizer (Na$_2$CO$_3$) 1.0 part provided a stable blasting slurry, d 1.55, which detonated in 5-in diam cbge 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, 63344e), 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 propnt 25.0, AN pill 31.0, NaNO$_3$ (granulated) 19.4, ethylene glycol 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 Triton or Comp B booster), may be sensitized by the inclusion of TNT, Tetrol or finely divided Mg or Al. A suspension of guar gum 1.5 in ethylene glycol 3.0 & AOH 0.3 part was formed by mixing at RT for 3 m.in and added to the basic ingredient mixture. As the slurry is poured into a container or a borehole, variable amts of NaHCO$_3$ (up to 0.14%) suspension in glycol are added into the exp1 stream and air is pumped under pressure into this stream at the same time. This produces an aerated expl slurry.

299) P.L. Swisstuck, USP 3288661 (1966) & CA 66, 39497t (1967) [An aerated aqueous expl 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$_4$, K, Na, Ca, Ba, etc), 4–35% water, 4–60% sensitizer (such as TNT, Pentolite, PETN, smokeless propnt, RDX or Tetrol), Al and Mg, 0.5–2.0% thickeners (such as guar gum, CMC-cellulose & starch), 0.6–5.0% surfactant (such as diethanolamides of coconut & lauric acids, triethanolamine salts, alklypoly(oxethylene), aryalkylsulfonates & mixed dimethylene amine oxides). Presence of

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 mls AN pill (8–20 mesh). These materials exhibit improved fuel oil absorption and detergent 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. Hoberstoffer & 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, BergJuetenmaennMonatsch (Austria) III(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 (1), 4.4–18.7% water, le: set amts of addl fuel (such as wood flour), oxidizer (such as NaNO$_3$ or K$_2$Cr$_2$O$_7$) and thickeners (such as okra gum and cellulose ethers gum)].

304) I.N. Chernov, Fix-TekhnProblRazrab (erroneously spelled Razrab in CA) Poleyiskop, 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 soins of salts, solid NaCl & KCl, ice, sand, powd gypsum, etc. After deton the bomb was cooled, contents extracted with water and the resulting soin 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 deton of crit 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 83/15 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 NH4Cl, (NH4)2SO4, and NaCl, resp]

307) B.N. Kukib, VzryvnoyeDelo 1966(60/17), 83–96(Russ) & CA 67, 13422x (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, DEGeCD, 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 & DEGeCD was similar to pure NG. Pure DEGeCD 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 bew 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 SiO2 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 Pobedit (described later in this Volume) showed that CH₄-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 Metanots and Minorits (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, 55795e (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 obt. 2) The strongest expls are mixts with a near zero oxygen balance 3) Addn of powered metals strengthen the expls but not as much as was reported in the literature 4) The behavior of water-filled expls at temps near frp 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 Igdonits (described later in this Volume) were conducted with mixts prepd from various fractions of granulated AN. Their specific surfaces were cld 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 Dekomman 1 and Dekomman 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 & 0.75), 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% CH₄ 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 symathetic 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 BaSO₄ or MnO₂. To reduce the humidity, 0.5% CM-cellulose was added to A-3. The expl 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 symathetic 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 props of Russian explosives Ammonits and Igdonits]

317) S.A. d'Explosifs et de Produits Chimiques (SAEPC) NethAppl 6606580 (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 BaSO₄ could be added to make the mixt suitable for use in mines. The names of surfactcs used are not given in CA.]

317a) ICI of A & NZ, Ltd, BritP 1033480 (1966) & CA 66, 77934c (1967) [Fluid or plastic compns prep'd 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 NaN₃ 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, KōgyōKayakukyōkaishi 27(5), 288–94 (1966) & CA 66, 11753af (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, 14614v (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 obt'd with Akremi in boreholes and chambers. The problem of 3-month storage of Akremi and its application at −10°C 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, Gun-cotton, Kuro (Black) Corlit (NH₄ClO₄ 66–70, FeSi 8–12, woodmeal 15–19, and heavy oil 3–7%), Permissible Powdered Dynamite, Black Powder, TNT and AN-FO. Both donor and acceptor were contained in a waterproof wrapper] (See also AddnRef 228)

319a) S.B. Wright, USP 3296041 (1967) & CA 66, 87227p (1967) (Continuation-in-part of USP 3173817; CA 62, 12968g) [Granules of org nitrata and nitramine HE, of high bulk d, were prep'd by mixing an az suspension of binder (polyamide, polisylobutylene 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°C in 500g BuOH was added to 470g diaminotriphenyl benzene (II) in 2000g water (at 70–5°C), while stirring at 500–600rpm. The resulting emulsion was agitated to give the desired granule size, BuOH evapd, the slurry cooled to 50°C, 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 6.67 lb "Corvus" oil per 100 lb Al. K dichromate was used as cross-linking agent. One of the final composites consisted of AN 32.7, water 17.3, NaNNO₃ (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, NaNNO₃ 16.0 & "Jaguar 100" guar gum 0.9 part), 20% pellitol & 5.0% precoated Al.

322) G.L. Griffith, USP 3300348 (1967) & CA 66, 67518f (1967) [Exp1 oninitrates, such as prep. by conitration with HNO₃-H₂SO₄ of corn starch mixed with pentaerythritol, to form NS/PETN, had relatively low impact sensitivities, higher initiation sensitivities and detonation rates than mechanical mixes 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. McCoy & E.C. Knowles, USP 3301721 (1967) & CA 66, 77931z (1967) [Hydrazine gel composes 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(M ethacrylate). Thus a soft gel was prep. It contained hydrazine 65.20, polyacrylamide 0.67, microglass fibers 0.80 & Al powder 33.33%. A similarly made gel comprised of hydrazine 58.31, 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 prep'd from 38–73% AN, 25–60% insol HE sensitizer, and 1–3.5% hydrocarbon binder and water-proofing 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 prep. by mixing 49 wt% of a 60 wt% AN soln at 120–80°F for 2–3 mins with a premix contg 25% granular NaNNO₃ 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 0.1 millimole Zr per g gum 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 composes having pouring d=0.5 & ca twice exp1 power of O-balanced expls (as shown by crating values and operational results but not by conventional test methods) are described. One formulation was prep. by mixing 81.5% AN (alone or contg a small amt of dendritic inorg nitrates), 1.5% mineral oil, 1.0% diamonaceous 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 slurtries contg sulfur and esp those contg 15% Al or other light metal were stabilized against decomp and resultant loss of effectiveness at elevated temps by incorporating into such slurtries of (preferably ≥ 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, NaNNO₃ (grained) 20.0, Al 20.0, water 12.0, guar gum 0.5, Na₂S₂O₃ 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 exps, such as listed in USP 2930695 (CA 55, 846h) and CanadP603514 (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 prep by making AN and NaN₃ particles adhere to and around TNT prills, which are held together by a binder consisting of CaSO₄ (ligna) and poly(ethylene oxide). The resulting expl was suitable for use in boreholes containing water]

330) G. Nanquin & E.A. Demelenne, RevIndMiner 49(1), 57–64 (1967) (Fr) & CA 66, 97066x (1967) [Improved Belg permissible expls with ion exchange were prep'd using an equiv mix of KNO₃ & NH₄Cl 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 KNO₃+NH₄Cl when compared with Belg char Broke 418 (which contained the equiv mech mix 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 Charbrit and the amt of rock broken was 20% higher]

331) H. Logan & R.F. Knott, USP 3306789 (1967) & CA 66, 117566c (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 41.8 ps 98% HNO₃ was cooled to 23° and seeded with NH₄NO₃/2HNO₃ mcrystals, then mixed with 28 ps 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 homogenuous* 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 containing Al of particular size distribution and processes for their production were described. A 1000-lb batch of expl was prep'd 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 ~12 ps water (satd at 60°F with 17.6–20 ps AN, based on total batch). After agitating the mix to provide homogeneous mass, it was allowed to stand for >15 mins to allow the gum to swell and NaN₃ 10, fine grain Al 20 and fine grain AN 20–5 ps were added successively with agitation. Finally a mixt of 200ml satd soln of AN in liq ammonia diluted 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, 1175568v (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, prep'd by simply mixing 95 parts MeNO₃ with 5 ps 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 mix of sorbitol hexanitrat 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, tributyl-acetylcitrate 25–30, pigment (1 p lampblack & 8 p chrome yellow) 0.3–0.8, and DPH 4.4 about 0.4% added. The mixt is prep'd 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 mortarl 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, 14–18 (preferably 5–14%) sensitizer such as hexamethyleneetetramine mononitrile (I), dinitratine or a mixt of the two, 45–65% AN, 5–20% NaN0₃ 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, NaN0₃ (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 NaN0₃ in this comp was replaced with AN, the props became sp gr 1.20, pH 4.9 & deton vel 3650m/sec]

337) J.A. Atbie, USP 3321344 (1967) & CA 67, 66206m (1967) [Aq slurry-type expl compns contg AN and an expl sensitizer (such as TNT, DNT or smokeless propnl) have attained commercial acceptance. These compns have been improved by inclusion of gelling agents (such as the cross-linked, partly hydrolyzed polyeacrylamide described in USP 3097120 & CA 59, 8540c). This invention provides expl slurries which remain pourable even when sub. 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, 55839w (1967) [Self-destructive expl cartridge suitable for use in seismic, underwater exploation, 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 case 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 Xanthomonas hydrophilic colloid mixed with aq soln of inorganic nitrate (such as AN) and borax are described. Xanthomonas is a hydrophilic, exocellular, high-moiwt colloid prep'd 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), K₂HPO₄, 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 1g 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, 8355ot (1967) [In org 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 cyst org material (such as glucose, trioxane, sorbitol, PA or Me$_2$NH$_4$ nitrate). The grains could also be made hydrophobic by use of water-sol or compatible substances (such as urea-H$_2$CO resins, cresol, polyvinyl, etc). Thus 10g powd urea-H$_2$CO condensate, 80g glucose and 10g Al powd were added to a soln of 40g trioxane in 100g water at 50$^\circ$, the mass mixed with 1kg granular AN and dried at 50$^\circ$ to yield Al-coated AN granules.

34(4) W.L.K. Schwoyer & Th.P. Dowling, USP 3332349 (1967) & CA 67, 83563(e) (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$_2$O$_3$) 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, NaN$_3$ 5, bagasse 5, sawdust 2, nut meal 2, oat hulls 2, vermiculite 2, Al$_2$O$_3$ 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 0.13–1.5 lb/ft of drill hole required for the specific presplitting operation]

34(6) A.N. Barton et al, USP 3336981 (1967) & CA 67, 92494y (1967) [A stable gel of fuming or concd nitric acid can be prep by the addn of 1–5% by wt of a polymer of N- vinyl-2-pyrrolidone cross-linked in the vinyl group. The prep 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 3265572 (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 a, a'-azobisisobutyronitrile, was mixed with 156 parts red (or white) fuming nitric acid. A gel stable for >1 year formed in 15 mins]

34(7) 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 mix 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$_2$, or furfuryl alc), and enough inert liquid to force the previously injected liquids into the formation, where they will mix and explode]

34(8) 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$^\circ$F (>80% strong) to 125$^\circ$F, with rapid agitation to obtain slurry of fine-grained crystals. Other inorg nitrates could be mixed with NS, as well as fuels, thickening agents, gelling agents and antacids. For example, a sard
soil containing 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 obsd. To 64 parts of this was added a mixt contg wet NS 22.4%, milled NaNO₃ 10, ZnO 0.8, bituminous coal 1.5, No 5 fuel oil 0.5 and guar gum 0.8 part. The expd slurry obtd after mixing had the following characteristics: d 1.44, ballistic pendulum value 10.10, sensitivity (in a 1.5-inch pipe) 10 g Pentolite, and deton rate 4987 m/sec]  

349) J. Calzia, FrP 1467939 (1967) & CA 67, 92489a (1967) (Methods of coating exps with polyacrylonitrile to improve their expl characteristics, are described)  

350) W.T. Jones & J. Wilby, BritP 1082641 (1967) & CA 67, 101578h (1967) (Exppls 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 ≥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) (Exppls 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 ams of balloons to sensitize NMe were in the range 1.5 to 10%. Fuels, oxidizers and thickeners could be incorporated)  

352) SERASDEX, FrP 1479383 (1967) & CA 67, 118759r (1967) (Exp safe for use in presence of natural gas, such as comprising AN (porosity 0.035 cc/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 igni- 

353) I. Toth & L. Nemeth, HungP 153385 (1967) & CA 67, 75039m (1967) [Moisture-proof exps, 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 CH₄. Compn of additives was SiO₂ 60, P₂O₅ 25 & B₂O₃ 15 mole %]  

354) J.P. Konrat, FrP 1469198 (1967) & CA 67, 75040c (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 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); Addo 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 chrmat are described. These mixts remained stable in storage and did not darken, as for example mixt made from AN 37.5, NaNO₃ 8.0, TNT 20.0, Al powd 17.0, glycol 0.9, water 16.0 parts to which was added mannogalactan 0.6 & ZnCrO₄ 0.1% showed only slight darkening after 33 days storage at 30°C, 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 det- 

357) Dynamit Nobel AG, NethAppl 6611745 (1967) & CA 67, 55838b (1967) (Four powd expls safe for CH₄-coal dust-air mixts were obtd by
mixing NG, Ngc, AN, KNO₃ & CaCO₃ 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 & 5.3; 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°C.

357a) Dynamit Nobel AG, NethAppl 6612512 (1967) & CA 67, 55837s (1967) [Plastic and gelatinous safety expls prep'd 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, NaNO₃ 27.5, AN 28.8, CaCO₃ 10.7 & 50% soln of Ca(NO₃)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 fire damp: 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°C, 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% fire damp, safe in a small mortar and 3% fire damp, and safe in a free firing test of 1000m in a 9% fire damp 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 cmpd (such as CCl₄, CHCl₃, C₂H₅Cl₃, 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 polyvinylsulfonate. Preferred thickeners were malagolactans (such as guar gum or carob beans). As a dispersant ethylene glycol was used, as a stabilizer NH₄ lignosulfonate, and as a fuel Diesel oil. For example, the expl compn contg granular AN 79.7, Diesel oil 2.4, CCl₄ 8.0, water 7.0, guar gum 0.5, ethylene glycol 1.0, and NH₄ 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 Teryll, 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 crosslinked 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 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, NaNO₃ 14, hexamethylene-tetramine 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/m³ bulk density). The plastic foam of 0.2mm dispersity was homogenized with the expl components pre-ground 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, 110246x (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, NaN3 15 & water 18 parts was heated to 120°F and treated with 0.8 ps carboxymethyl cellulose for 5 mins. After mixing 8 ps 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 3 mins 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) [PENTB-based mixts useful as expl sensitizers were described in which the impact sensitivity of the PENTB was substantially reduced (without affecting its deton sensitivity) by incorporating 5-55% trimethylolmethane (TMeEt). Mixts contg <30% TMeEt were granular solids, while those contg ≥30% were extradable. A dry stick powder contg 70:30-PENTB/TMeEt 13.5, grained AN 79.7, NaN3 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, 118752b (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 (prep'd at the site) was mixed in proportion 15:11 with MeOH and dissolved in a 73% eq 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 ≤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 bentonite and org comps (also serving as fuels) which are preferred for use with oil as they release water in the mixer. They included polyurethane, nylon, cellulose, poly(vinyl)chloride, 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 Na (Nitrostarch) (23% water) 17.0, AN (ground prills) 53.0, NaN3 (granular) 15.0, ZnO 0.6, Al (flaked) 1.75, Na2S2O4 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 1.25 x 14-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, 4591 v (1968) [Stabilized blasting expls obtd by using AN contg an anticaking agent and subsequent sensitization to produce expl compns having resistance to crys
degradation. This objective is achieved by combined use of 0.01–1.0% of a C₈–C₂₂ 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 addition of sensitizers, such as NG, TNT and NC. The amines, mentioned above, are more effective than 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, NaN₃ 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 9300 ft/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 cong 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, NaN₃ 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 comps, 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, NaN₃ 18–21, fuel (hydrocarbon oil, TNT, powd Al or ferrophosphorus) 8–37 and water 10–20 parts. Included in each component of the foregoing compn are 0.2 to 0.3 parts guar gum, 0.4 to 1.0p polyacrylamide having a mol wt of 5×10⁶ 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 comps are described consisting of an aqueous slurry of inorg oxidizer salt (such as AN or NaN₃), 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) [Nitrocarbonate blasting slurry comps contg flaked Al and DNT as sensitzers, are described. Prior-art inorg oxidizer salt expl comps of the ag-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, Japan P 7105 (‘67) & CA 68, 5149w (1968) [Polyimidotetrasylsulfonylhydrimide R[CH₂CH(CH₃ONO₂)₃]H₄m (I) were hardened by the adn of diisocyanates, or diepoxy compons in the presence of amines. Thus, 50g of I(R=OCH₂CH₂O), m = 2 & n = 1), prepd by adding (CH₂OH)₂ to glycylid nitrate. 0.5g Et₃N, and 30g tolyene 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 compons].

377) Wm. Dick & O.A. Garton, 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 or NGGP 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+NaNO₃) 30–50 and a flame quencher (such as NH₄Cl or NaCl) 10–30%. Thus a Blasting Explosive that would not ignite at atm pressure, contg 80/20–NG/NGGP 20.0, NC 0.3, AN 14.0, NaNO₃ 32.0, NH₄Cl 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, deten 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 adn of aq soln of glycol. Thus, the expl contained NG (with or w/o NGGP) 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 ferroite red 0.1% by wt].

379) K.K. Andreev & V.M. Rogozhnikov, TrMosk-Khim-Tekhnol-Inst No 53, 163–76 (1967) & CA 68, 31686m (1968) [Effect of addns of some combustibles on the combustibility of AN and Amm perchlorate and mixtures based on them. The main conclusions were as follows: The adn of small amt of finely dispersed Al to these mixts sharply increased the possibility of the transition of burning into expls. 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), 4–18 (1967) & CA 68, 41759a (1968) [Use of expl suspensions of AN in admixt with TNT or Dynamite in some Bulgarian mines is discussed].


382) T. Yoshida, KogyoKagakuKyokaishi 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-Tekhnol-Inst 53, 321–38 (1967) (Russ) & CA 68, 51476y (1968) [The effect of some catalytic addtives (NH₄Cl, KCl, BaCl₂, NaCl & K₂Cr₂O₇) on the combustion rates of Dynammons ( Dinammony, 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 addtives (such as K₂C₂O₄, CaCO₃, SiO₂, LiCl, NaCl, KCl, BaCl₂, NH₄Cl, K₂Cr₂O₇, (NH₄)₂O₂O₄, KNO₃, KClO₄ & graphite on Amatol (Ammotol in Russ) contg AN 80 & TNT 20%].

384) W. Cybulski, ArchGom (Polish) 12(3), 189–225 (1967) (in Engl) & CA 68, 14612s (1968) [Testing of safety of 5 types Polish mining expls (comps 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 Izobret.Prom.-
Obraztsy, Tovarnye Znakii 44(15), 194 (1967)
(Storage-stable, shock, resistant cylindrical
or flat charges are composed of AN 58.83,
NaNO₃ 14.80, KCl 3.76, NH₄Cl 9.32 & TNT
18.29 parts)

(386) J.D. Chriple, 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 ≥2 terminal unsatd CH₂ groups
in 30–98%aq HNO₃ is described. An expl
compsn was prep'd from 70%aq HNO₃ 73.2,
acrylamide 5.6, N,N'-methylenebisacrylamide
0.4, Ag nitrate 0.4, (NH₄)₂SO₄ 0.1 & DNT
20.3%. It had O balance -0.7%, density 1.3
and deton velocity 7050m/sec]

(386a) G.L. Griffth, USP 3361603 (1968) &
CA 68, 51486x (1968) [Inorg oxidizer salt
explo compsn contg paper sheet particles as
pouring density reducers, are described.
Such compns comns of mixts of a nitrates,
perchlorate or chloride of NH₄ and alkali- or
alkali-earth metal up to 81%, an expl sensiti-
zizer, such as NS (Nitrostarch) (preferred),
TNT, PETN, Comp B, etc 5–30 & ground
paper 4–16%. Up to 30% (preferably 0.5–
20%) conventional carbonaceous fuel or
0.5–15%metal (Al, Al alloys, ferrosilicon
and ferraphosphorus) fuels, 0.3–2.0% antacid
0.5–30% liquid slurryng agent and small
amts of thickeners (such as guar gum, CMC,
psyllium seed mucilage, pregelatinized
starch, silica aerogels, Al₂O₃, tetrapiglitite,
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. Griffth, USP 3361604 (1968) &
CA 68, 51487a (1968) [Explosive slurries
contg an oxidizer salt and particulate vege-
table 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 NH₄ and alkali- or alk-earth me-
tals, 25–30% of sensitizing expls, such as
NS (preferred), TNT, PETN, Comp B, etc,
0.1–30% of finely divided pulp vegetable
matter, and 7–5% 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% pro-
vided a good expl slurry, d 1.35, ballistic
pendulum value 10.00, viscosity 300cp and
deton rate >3600m/sec]

(388) V.F. Starokazhev et al, VzryvnuyeDelo
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 NaNO₃-NH₄Cl 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 in-
creased. 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 phleg-
matized RDX and then, later, with pure RDX.
Such mixts, as for example, contg: RDX 35,
course AN 38 & inert matter 27% ensured de-
ton 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 pos-
sible to reduce crit diam to as low as 24mm]

(390) V.I. Chikutov, Ibid 1967 (63/20), 169–77
& CA 69, 4080y (1968) [Resins-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-creosol-
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, VzvyinoeDelo 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 comp 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 > 0.3m, the diam of the fissures kept < 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. (Comps are not given in CA). It was decided that the excess pressure of CH₄ 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 compg 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 edgs of different wts. The relative increase of the coarser particles of the active compn in the Ammonite mixt lead to increased resis to firedamp. An increase in the NaCl content of the coarsely dispersed expl also increased resis to firedamp]

395) L.D. Khotina, VzvyinoeDelo 1967, (63/20), 217–19 & CA 69, 4082a (1968) (Effect of sensitzers 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 liquid nitroester)

396) A.N. Zakhaenko & 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. Kolesnikchenko, Ibid 1967, (63/20), 232–37 & CA 69, 11861g (1968) (Industrial testing of mining expl Aquafot at Kal’makinsk and Kurgashinkansk mines showed that the expl is effective, inexpensive and easy to use in both dry and wet quarries. The comp of Aquafot is given in the Section which follows these AddnRefs)

398) M. Gregor et al (Czech), IndChimBelge 1967, 32 (Spec No, Pt 2), 373–76 & CA 71, 31916y (1969) (Stabilization of AN with dolo- mite has advantage over stabilization by chalk)

399) O.A. Gurton & Wm. Dicks, IndChimBelge 1967, 32 (Spec No, Pt 3) 511–14 & CA 70, 49149c (1969) (An expl Dynogex, 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, 79647f (1969) (Thermal decompo of inorg salt-based expls compg 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 Carbornit B, Energit A and Nobelit B. Comps of these expls are not given in CA.

401) C. Fosseé, Ind.Chim.Belge 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 Sterrebeck, 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 expnl data permitting more accurate understanding of the kinetics of the different priming methods).

402) G. Nenquin & G. Fally, Ind.Chim.Belge 1967, 32(Spec No, Pt 3), 606–09 (Fr) & CA 70, 49150h (1969) [The combstn of various mixts of NH₄ salts and alk or alk-earth nitrats, and esp NH₄Cl-KNO₃ (which constitutes the base of ion-exchange safety expls) was studied. It was found that combstn depends mainly on the acid linked to the NH₄ mol and that the acid liberated during reaction reacts with the nitrate to form O and N oxides, which support combstn of NH₄. In NH₄Cl-KNO₃ mixts, max combstn pressure was obtld with the 1:5:1 mixt. Conc 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 de-tonability, a significant reduction in conc 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, Ind.Chim.Belge salts (such as nitrates of NH₃, 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 NH₃, 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, HCONH₂ 10, Na lignosulfonate 2.0 & guar flour 0.7% by wt could be fired in a 3 x 24-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, NaNO₃ 14.7, Al powd 3.9, powd coal 5.3, acrylamide 1.9, and methylenebis(acrylamide) 0.2 part were dispersed in 73.8 ps 65% of an AN soln held at 54° and 0.2 part (NH₄)₂S₂O₃ stirred in. After 2 mins, a medium-to-firm gelled blasting agent was obtld. 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/m³ 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/m³ lead to a sharp increase in the amr 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, NaNO₃ 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 NaNO₃ 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 49.2% 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 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) [Liq expl mixts of polynitrogompds and of aliphatic carbomates, suitable for mining and quarrying, are described. Polynitrogompds suitable for use in these expls were: polynitroparaffins such as C(NO₂)₄, NG, NGc, DGTEN, PETN & TNT and for carbomates (and their mixts) the following were listed: methylcarbomates, n-propylcarbomate, iso-propylcarbomate, vinylcarbomate, isopropylacetylene, etc. Mixing of org polynitrogompds with carbomates produced very sensitive or spontaneously detnating expls. As an example was cited a homogeneous mixt prepd by adding slowly to 18.4 parts isopropanolcarbomate (agitated in a reactor by bubbling a stream of N₂), 78.4 parts of C(NO₂)₄. C(NO₂)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 Ccontg substances.]

413) Wm. D. McFerrin, USP 3378417 (1968) & CA 69, 4117r (1968) [Inorg nitrate expls of any desired expl composition 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 Aln chloride formed from large grains. These grains were obt by the double decom, in the presence of water vapor, of Al 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 sysms 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<Fc<Cu<Ti<Sn<Sb<Al. The activation energy of the ignition reaction was calc'd from the ignition delay time at different temps)

405) Marcel Vereauteren, AustrianP 277027 (1967) & CA 72, 45655y (1970) (In the presence of for manuf expls, AN was crystallized from concd or saturated solns in the presence of 0.1-5% by wt C₆-C₁₅ primary amino alcs and their salts added in amts 0.4-1.0%. Thus a mixt of 25g ndecylamine acetate and 75g triethanolamine acetate was added to 10kg of an ag 80% soln of AN at 65° and the soln allowed to crystallize for 24 hrs without stirring. The crys formed were filtered off and pressed to obtn a crys 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 Lorraine iron mine)

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-antracids (such as CaCO₃ or MgO) up to 0.4, and materials (such as rosin, metallic resinsates, hexamine, waxes or mineral oil) 0.4% could also be present. A mixt listed in CA contg AN 70.68, NaNO₃ 8.91, Al powd 1.57, NS
12.09, CaCO₃ 0.2, wood flour 6.54 & C black 0.01% was a blasting expl of d 1.06–1.10, sensitive to a No 10 cap. When 4.45 parts water was mixed with 95.55 parts 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 19.6 parts of Na borate decahydrate, the d became 1.16 and the expl became sen-
itive to a No 16 cap.

408) R.B. Clay et al, USP 3367805 (1968) & CA 68, 8009x (1968) [Inorganic nitrate aqueous slurry blasting compns contg nitrate and having a high-area lyophobic surface are described. These compns are com-
posed 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 20-50 m²/g together with a lyopho-
bic surface capable of forming void reaction sites. To this mix a phosphite inhibitor was added to prevent premature reaction between 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 can be pumped into boreholes consists of AN 38.0, NaN₃ 15.0, water 17.0, a phosphate stabilizer 0.1, Al powd
1.1, S 6.0, gilsonite 4.0, guar gum and partic-
ulate Al 17.7% by wt. The first three in-
gredients 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 predetermined particle size distribution (as per cent 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 ² openings), while the re-
tained material must pass a 100 mesh (0.0059-²

410) J.W. Francis, USP 3379586 (1968) & CA 69, 4116a (1968) [Gelled Ammonium Nitrate-

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
(≈1.3) and bulk strength, good water-resistance and good sensitivity to initation. 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 H₃ClO₃ in mixes with alkali or alk-earth nitrates, chlorates or perchlorates; as an inorg sensitizr fine Al (up to 5%) (or when mixed with up to 40% coarse Al, up to
10% ferrosilicon or ferrophosphors was included); (ferrophosphors could be used without Al); up to 6% thickener, 5% water, 10% of a water extender such as glycol, glycerol, MeOH, EtOH, HCONH₂, or HCONMe₂, and the conventional fuels. Thus, the expl slurry prepd by mixing AN 17.75, Ba(NO₃)₂ 19.60,
NaNO₃ 14.7, TNT 19.6, ferrophosphors 11.75,
water 12.73, ethylene glycol 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. Kuek, 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 mixes 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 admix 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 comple-

414) J.W. Francis, USP 3379586 (1968) & CA 69, 4116a (1968) [Gelled Ammonium Nitrate-

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
(≈1.3) and bulk strength, good water-resistance and good sensitivity to initation. 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 H₃ClO₃ in mixes with alkali or alk-earth nitrates, chlorates or perchlorates; as an inorg sensitizr fine Al (up to 5%) (or when mixed with up to 40% coarse Al, up to
10% ferrosilicon or ferrophosphors was included); (ferrophosphors could be used without Al); up to 6% thickener, 5% water, 10% of a water extender such as glycol, glycerol, MeOH, EtOH, HCONH₂, or HCONMe₂, and the conventional fuels. Thus, the expl slurry prepd by mixing AN 17.75, Ba(NO₃)₂ 19.60,
NaNO₃ 14.7, TNT 19.6, ferrophosphors 11.75,
water 12.73, ethylene glycol 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. Kuek, 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 mixes 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 admix 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 comple-

414) J.W. Francis, USP 3379586 (1968) & CA 69, 4116a (1968) [Gelled Ammonium Nitrate-

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
(≈1.3) and bulk strength, good water-resistance and good sensitivity to initation. 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 H₃ClO₃ in mixes with alkali or alk-earth nitrates, chlorates or perchlorates; as an inorg sensitizr fine Al (up to 5%) (or when mixed with up to 40% coarse Al, up to
10% ferrosilicon or ferrophosphors was included); (ferrophosphors could be used without Al); up to 6% thickener, 5% water, 10% of a water extender such as glycol, glycerol, MeOH, EtOH, HCONH₂, or HCONMe₂, and the conventional fuels. Thus, the expl slurry prepd by mixing AN 17.75, Ba(NO₃)₂ 19.60,
NaNO₃ 14.7, TNT 19.6, ferrophosphors 11.75,
water 12.73, ethylene glycol 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]
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 inorganic oxidizing salt (AN, NaNO₃, Ba(NO₃)₂, or their mixts), a sensitizing (TNT, RDX/TNT, PETN/TNT, EDNA/TNT, or smokeless propellant), a thickener (guar gum), water, and a polymeric solvent (iso-PrOH, a polyhydric alcoh or an amide). These solvents could replace water to lower the Fp of the mix. Air or other gas could be introduced to decrease the density. Thus, a typical compn consisted of 75-mm, M-1 smokeless propellant 25, AN 43, NaNO₃ 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, 45033c (1968) [Method of prep a strong porous 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 have a high shock energy and low susceptibility to dust explosion, are described. For example, water 17.0, AN 28, and NaNO₃ 12.6 parts, were heated to 100°F and treated with 25.0 ps smokeless propellant and 14.0 of Si powder. The pH was adjusted to 4.5–5.0 with H₂O₂ and a dispersion of 0.7 ps guar gum in 3 times its weight and oil content ca 6.7%, is described.]

420) R.F. Preckel, USP 3390029 (1968) & CA 69, 45034c (1968) [Expl compn consisting of an inorganic oxidizing salt suspended in an organic 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 NH₄ClO₄ 60–70, Al 30–20, and HCONMe₂ 10% were tested for crit diam for propagation of detonation. In all cases, the results were obtained 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 NH₄ClO₄ 72, Al 16, and HCONMe₂ 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 detonation with velocity in the order of celled values was obtained. Similar systems contg no solvent had crit diam ≥ 20 inches.]

421) H.R. Fee, R.W. Lawrence et al, USP 3390030 (1968) & CA 69, 45036g (1968) [Aqueous slurry blasting compon which contain a Si alloy and an aerating agent, have decreased susceptibility to dust explosions and excellent underwater shock & thrust energies. The compon is comprised of AN 30–75, NaNO₃ 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 NaNO₃–NaHCO₃ aerating mixt 0.04–0.4%. As an example of prep, a soln AN 52, NaNO₃ 15.6 & water 16.5 parts was adjusted to pH 5.4–6.0 with NH₂OH or HOAc. A mixt contg NaHCO₃ 0.046, NaNO₃ 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 mix 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 deten rate 5250m/sec]

422) A.A. Albert, USP 3390031 (1968) & CA 69, 53324q (1968) [Gelled aq slurry expl compn contg an inorganic nitrate 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 NaNO₃ was added to a mix comprised of 41.7% AN & 13.0 NaNO₃, slurried with water 17 at 100°F & ground smokeless propellant 25 ps. After adjusting the pH to 4.5–5.0 with H₂O₂, 0.7 part guar gum in 2.1 ps ethylene glycol was added and mixed in the soln, followed by 0.08 part cross-linking agent (guar gum) in 0.4 ps 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]

426) J.P. Merryweather et al., USP 3397095 (1968) & CA 69, 78899q (1968) [Gelled aq AN expl compns which were freeze-resistant and nonhardening were obt by incorporation of a small amt of H2CN, and espec of H2CN 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 H2CN sln was then added with mixing for 30 sec, after which a premix 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 NaN3 was added and mixing contd until the mix 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 booster characteristics, 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 prep by incorporating gas bubbles and a crystal-habit modifier. For example, an expl was prep by placing in a rotary mixer AN "neutral liquor" (normally 80%) at ≥150°C and, after starting the agitation, adding a crystal-habit modifier (Na methylnaphthalenesulfonate), followed by a soln (granulated sugar), NaN3, 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 explsnsitizers suitable for incorporation in various formulations, as for example in blasting expls, such as contg AN 54.20, NS 22.00, DNT 4.15, NaN3 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]
340) 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 ~3%), proteinaceous material (preferably foamyable) and having mol wts of 5 to 200 thousand (preferably ≤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 NaNO₃ and 1 lb guar gum was added, followed by flake Al 2.0, DNT (mp 26°C) 11.0, granulated sugar 5.0, and formamide 2.0 lb. After 4–5 min of mixing, 5% aq Na₂Cr₂O₇ 100 ml and 5% aq KSB tartrate 200 ml were stirred in. The resulting blasting agent had d 1.24, detont rate unaffected in 4-inch column at 60°F 4600m/sec and 3820 in 2-in column at 72°F]

341) T.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 NaNO₃), 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, NaNO₃ 15.0, ethanoamine nitrate 18.3, guar gum 1.5, Al 5.0, and water 16% by wt. It detont rate was 5000m/sec.]

342) Louis McDonald, USP 3403061 (1968) & CA 69, 98129w (1968) [Conditioning particulate materials for use in org expls, is described. Solid precipitated 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 ≥1 liq or solid org Si compd (such as methylchlorosilane of the general formula R₃SiR₃SiR₃SiR₃Si), in which the R's are alkyl, aryl, alkoxy, or aryloxy groups, resp, but ≥1 of them is a halogen atom or an NH₂, OH, alkoxy, or aryloxy group). Thus, wet RDX was dried
and, after being treated with 30 parts Me₃SiCl₂ in 70 ps dimethylsiloxane was incorporated into melted TNT for formulation of Comp B or Cyclolol. Expls made in this manner had a more const compn throughout 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]

343) Ph.W. Fearnow, USP 3406051 (1968) & CA 69, 108263b (1968) [Aqueous expl compns free of self-expl sensitzers (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 & NaNO₃ 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 (Na₂Cr₂O₇ in soln) was added while the slurry was pumped into borehole. These compns detonated at ca 5000m/sec at 60°F]

344) 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, gelsonite 2, guar gum 0.6 & NaNO₃ 12.7 to a soln of AN 38, NaNO₃ 15, stabilizer 0.1 and a soln of guar gum 0.2 in ethyleneglycol 0.4 in water 17%]

344a) W.C.G. Baldwin, BritP 1109256 (1968) & CA 69, 4119r (1968) (Exp 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, NH₄Cl 7.0, NaNO₃ 15.1 &
NaCl 5.6%. For prep 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 estets of polyhydric alcs (such as mixts of NG with NGc) for use in Dynamites, was achieved by incorporating small amts of acetylacetone, benzoylaraceton, or phloroglucinol. Mixt of NG 60 & NGc 40% stabilized with 1% acetylaceton withstood Abel stability test for >300 mins]

436) V. Rank et al, GerP 1274479 (1968) & CA 69, 78894p (1968) [Mining expl comps are prep'd 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 5–6% 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 mix was sprayed with spindle oil (110g oil on 2kg AN) and cartridge. Its deton rate was 2900 m/sec at 20cm from the Gelatine-Donar 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-briassence and increased storability. Example of permiss expl: explosive oil 34.0, NC 1.25, rosin 0.80, AN 11.0, sawdust 1.2, and limestone powder 51.75%. Example of nonpermiss 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, Brit P 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 comps contg a soluble polyflavonoid sensitizer, are described. For example, a slurry expl compn contg AN 51.5, NaN_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 Pentoite. A similar expl which contd 54% AN but no polyflavonoid failed to detonate when initiated by 100g of Pentoite]

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 ag oxidizing salt mixt or soln comprising 10–20% water, 0.25–2% gau gum, and one or more salts (NH_4K, Na, Ca nitrate and/or K_2CO_3 or K_2Cr_2O_7). These slurry comps remained viable 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 NaN_3 or (KNO_3), and/or NH_4Cl showed increased heat and energy of expln by addn of an equim amnt of NH_4 oxalate. Addal 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, IzvVysshUcheb-Zaved, GornyZh 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 Kirvii Bog Basin using Dinonitol and Ammonit-62HzV. After removal of the blasted material the concn of CO & NO2 exceeded the standards. To reduce adsorption of CO and NO2 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 fire and dust. Three types of Ammonites were tested. The protective charge (sheath) consisted of a double-walled polyethylene capsule (I.D. 84 & O.D. 150mm), filled with water below the walls. The expl (1.63kg) 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 core perpendicular to this was placed a vibrigraph to record the results of expliniti by an elec detonator. A Cu tube with expls was also used to detect the compressive wave of expl. By this method, safe, min distances were established for expls of different power.]

445) J.J. Minnick, USP 3409484 (1968) & CA 70, 21467v (1969) (Aqueous alkylamine expl slurries contg an inorg nitrate oxidizing salt, a lower alkylamine nitrate, water, a non-explosive, finely divided, air-entrappping 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-entrappping 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 16600ft/sec] Note: Prepns 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, NaNO3 10.0, granular Al (94-100% <8-mesh, ≤5% <100-mesh US) 15.0, Al (paint-grade flakes) 1.0, hexamethyleneetetramine 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 comps, having a high deton rate, low frp, viscosity, and impact sensitivity are provided by mixts of 70-92% of an oxidizer (such as N2H4NO2 & its mixt with N2O4ClO4 in a 4:1 ratio for increased sensitivity), and including up to 25% of metal nitrates (such as Na, K, Ca or Al nitrates and perchlorates for reducing foaming during prepn). Also are incorporated 6-15% N2H4, and 2-15% NH3 (as a frp 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 Guar-Tec 503) and 0.5-5% wetting agents (such as Me2SO4, HCONH2, 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 comps sensitized with an air-entrappping 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 MeNO3 17.0 with NC 1.0 until a gel was obtgd and then stirring in AN 60.0, guar gum (thicker) 0.6, urea-HCHO resin microballoons (air-entrappping material) 2.0 & water 19.4% until homogeneity was obtgd]
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 NaNO₃ & 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 1.66g/cc. They were stable in moist atm for up to 6 days] (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% CaCO₃, and a maximum of 6% SiO₂]

454) V. Rank et al, SAfrican P 67 00348 (1968) & CA 70, 69760k (1969) (Blasting expls of high detonation 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 0 balance of 5.5-6%. AN of desired porosity was prepd by grinding corn 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 0 balance]

455) G.M. Lukaszewski, Australian Inst Mining-MerProc 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 matrices. 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 SO₂]

456) G.A. Teichmann, SAfrican P 67 00518 (1968) & CA 70, 69758r (1969) [Flexible explosive tape for seismic prospecting is prepd from a train of individual flat chgs (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 deton 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, SAfrican P 67 00519 (1968) & CA 70, 69759g (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, AfricamP 68 01174(1968) & CA 70, 69766s (1969) (Explosion charges of low brisance for smooth blasting, contg NG and/or NGc 33–66, kieselgur 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) (Crybrs high-brisance expls (such as Penterythritol Trinitrate) 60 parts was homogenized with 40 ps of an aq dispersion contg 45% poly(vinyl acetate), 9% DBU-PH & 46% water and the resulting paste dried at 70° to give a solid of d 0.92. Its detone vel was 5200m/sec at a chge diam of 28mm. Similar chges were prepd using RDX or Trimethylene-trinitramine in lieu of PE Trinitrate).

460) H. Kinura & 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 obtained by mixing AN 55, TNT 25, 3.2% aq sohn 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. Sarch syrup was also used)

461) Heinz Dehn, BritP 1129777(1968) & CA 70, 49164r (1969) [Expl compons 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 Me2SO)]

462) W.C. Thomison & Th.E. Slykhouse, AfricamP 68 01234(1968) & CA 70, 49165s (1969) (Slurry expl compons capable of complete propagation when detonated under pressure (as high as 70kg/cm²) 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, NaN3 0–60, water 5–25, gum arabic 0.2–2.5, Al (30–150 mesh particulate) 5–40, and a carbonaceous non-explosion fuel (such as C pow or org liqs) ≤20%]


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, Na2SiF6 12.70 and wood dust 0.28%)

465) E.M. Aradan & Ch.H. Noren, AfricamP 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 prep, or by the use of a closed-cell rigid foam or cellular low-d fuel. This method permits formulation of compons 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. Fearnan, AfricamP 68 02829 (1968) & CA 70, 116786g (1969) (Water-contg expl compons 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 mix suitable as a mining expl or a flare proplnt consists of an intimate mix 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 prep'd by adding of finely ground flame-coolants (such as NaCl, BaSO4, NaHCO3 or metal salts of oxalic or fumaric acids) to AN-fuel oil mixts in which AN had a sp surface of >70 (preferably ca 2000)cm²/g]

469) Yu.S. Mets & I.T. Kolensichenko, 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 & NO2 formed depended upon the compns of expls and conditions of loading]

470) L. Moelng & W. Hofmeister, KaliSteinsalz 1968(3), 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 crit 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, gelatious expls, known as "gommess". It is called "gum A" in CA which is misleading because it is not a gum, but a hard, gelatious expl contg NG 92–94 & collodion cotton 8–6%. It corresponds to Ger "Sprenggelatine" or Amer "Blasting Gelatin". A list of numerous "gommess" 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, 131113 (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, 9 pp (1968) & CA 70, 13126r (1969) [In this rept expls 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 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 & KH2PO4 in 1:29 ratio) was added to hold the pH at 4.5–5.5]

474) L.V. Dubnov & I.T. Kolensichenko, Gornyizh 1968, 143(5), 56–9 (Russ) & CA 70, 13128r (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. Demiduk & B.D. Rossi, VzryvnoyeDelo 1968, (65/22), 5–15 (Russ) & CA 70, 116755x(1969) [Development of Russ industrial expls, such as Dinamony (Dynamons) (approved by the Govt in 1934) and Igdonity (Igdonites), developed in 1959, and included in the official list of nonpermissible expls in 1966] (See also in this Vol under Dinamony and under Igdonity)

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 Igdonit)


478) A.B. Plekhanova et al, Gornyizh 1968, 144(9), 40–2 (Russ) & CA 70, 30538u(1969) (Testing of Igdonit at a northern Ural bauxite mine, showed that on its expln the formation of toxic gases CO & NO₂ was lower than from expln of Ammonit 6ZhV. Compsns 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, 31927x(1969) [Effect of degreé of dispersion and specific surface of AN on the critical diameter of Igdonit (qv)]


481) M.F. Kosharnov, 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 Igdonit)


483) A.V. Brichkin et al, Ibid, 1968, (65/22), 75–92 & CA 71, 31940b(1969) (Aerosol process for charging loose expls, such as Igdonit, into boreholes is claimed to be more convenient, safe and rapid than pneumatic charging)


485) N.F. Adrianov et al, Ibid, 1968, (65/22), 109–27 & CA 71, 31926b(1969) (Testing of Igdonit 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 Igdonit was similar to those of Ammonit 6ZhV, Detonit 10A, Detonit 6A and Dinafalit, 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) (Igdonit 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 Igdonit, 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 Igdonit 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)


492) L.A. Mamsurov et al, ibid 1968, (65/22), 182-87 & CA 71, 31924z (1969) (Effectiveness of Igdonit 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 Igdonit in flint, limestone and granite showed that it was more economical to use than Detonit, Ammonit No 6 and Skol’nyi (Rock) Ammonit)

494) G.A. Berishvili & V.D. Gabidzashvili, VzryvnoyeDelo 1968, (65/22), 201-05 & CA 71, 31937f (1969) (Effectiveness of Igdonit 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 Igdonit in cleaning faces of the Chordskii barite deposit showed that it was more economical to use than Ammonit No 6)


498) V.P. Tambellini, ibid 1968, (65/22), 228-35 & CA 71, 31936e (1969) (Testing of Igdonit for use in external charges showed that it can be used effectively for blasting in limestone, rubblestone layer (max 70cm thickness), and reinforced concrete (300-1000cm² 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 (exp1 contg porous granulated AN & Diesel oil) is superior to BA-62 (exp2 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 consituents are described. The surfactant proposed was a Na, NH₄, 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. Goffatt (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 60-77°C and 30ps TNT (in 3.35-2.38mm 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 polycrystalline, and 20 parts NaNO₃ based on the exp1 compn. Then 907g HCONH₂ was added and after stirring for 3.5 mins, 25g K₂Cr₂O₇ (in 5% soln) added over 15 secs and 100g of a 5% soln of K antimony tetratrate 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°C or 110°C)

503) P.R. Goffatt (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 NGe 9.5, KNO₃ 55.25, NH₄Cl 28, NH₄ oxalate 5 & water-proofing additives 2.25 parts. Other formulations are listed)

504) Hirtenberger Patronen-, Zundhutchen und Metallwarenfabrik AG, FrP 1540861 (1968) & CA 71, 83183b (1969) (Expl compns contg microcryst AN are described. Such AN was obted 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
naphthenalene). For example, a mix contg
94.3% prilled AN (1.2mm diam; 0.14% water;
oil retentiveness 4%, 2.8% gas oil, and 2.9%
naphthenalene (0.1mm) was more powerful
than the mix contg 94.4% AN & 5.6% oil)

506) DuPont & Co, FrP 1544937 (1968) &
CA 71, 103745s (1969) [AN expls are de-
scribed which comprise a C-contg combust-
able (such as sugar), a thickening agent
(such as guar gum), a crosslinking agent
for the gum (such as K₂Cr₂O₇ soln), a gas-
encapsulating material (such as marrow of
sugar cane), and an anionic surfactant as
crystal-modifying agent (such as Na methyl-
naphthalenesulfonate). For example, an
expl was prepd by mixing at 65° in a rotating
blender the following ingredients: AN 56,
water 14, NaN₃ 15, sugar 8, sugar cane narro
3, Na methyl-naphthalenesulfonate 1, steeriac
acid 3, and guar gum 1% by wt. The gum
crosslinking agent (5% K₂Cr₂O₇ 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 con-
temporary caps) was 4500m/sec at 32°C
and 4150m/sec at 4°C

507) Schweiz Sprengstoff-Fabrik AG, FrP
1549958 (1968) & CA 72, 68851v (1970)
(Polystyrene foam beads were used as fil-
lers for regulating the charge density of
expls used for blasting purposes)

508) Commercials Solvents Corp, FrP 1550911
(1968) & CA 71, 93220a (1969) [Exp slurries
containing AN, methanol, water, urea nitrate,
guar gum or a resin UCAR C-149 or WSR-131
are described. For example, a mix 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 ex-
cellent shattering effect on rock were obtd]

509) R.G. Hall, S AfricanP 68 03385 (1968)
& CA 71, 409298g (1969) [Nitrate-fuel-binder
expl compds are described which were prepd
from mixts of K, Ba, or org (aliphatic or
guanidine) nitrate, a fuel (C, S, sugar, a com-
bustible metal powder, or mixts), and a re-
sinous binder. The binder consisted of a
lq polymerizable resin dissolved in an org
solvent. A polymerization catalyst or ac-
ccelerator for the resin was also included]

510) K. Shiino & S. Oinuma, KogyoKagaku-
Kyokaiishi 1968(5), 347–52 (Japan) & CA
71, 103734n (1969) [A comp is described,
contg sym-Dinitrodimethylurea 45, NC 5
& AN 50%, as an excellent expl having the
the following props: impact sensitivity
(max nonexpl height of 5kg falling hammer)
58cm, heat of expln 1185cal/kg, gas vol 901
l/kg and ballistic mortar value 146 (TNT 100)
Also are given props of sym-Dinitrodimethyl-
urea]

511) Ph.W. Fearnow, S AfricanP 68 03478
(1968) & CA 71, 23401m (1969) [Aqueous
expl compds of d 1.0–1.5 contg no self-
explosive or metallic sensitizers, yet capa-
bile of reliable detonability at low temps,
comprise 40–75% inorg oxidizer (such as a
nitrate or perchlorate of NH₄, Na, K, Mg,
etc or their mixts), 2–40% partially nitrat-
eg aromatc sensitizers (such as MNT, DNT,
PhNO₂, DNB, MNX, DNX, or their blends),
1–30% of a C₃₋₄ lower aliphatic monoamide
sensitizer dispersant (such as HCONMe₂,
AcNMe₂, propionamide, or acrylamide),
0.2–5% thickener (such as guar gum, quinse
seed, gums, gelatin, casein, agar, etc) with
a crosslinking agent (such as KSB₄H₄O₇·
½H₂O), 5–30% water, 2–20% nonexpl car-
bonaceous fuel (such as carbon, coal, sugar,
wood & paper pulp, etc), and ≥ 0.01% of a
sol proteinaceous material (such as egg al-
bumin). A detailed description is given of
prepn of a mixture which had deton rate of
5100m/sec at 56°C, in 5-inch diam unconfi-
ded polyethylene container]

512) M. Novotny et al, SbVedPr, VyskChem-
kotechnol, Pardubice 1968, (17)
(Pt 1), 115–22 (Czech) & CA 71, 31944f
(1969) [Safety measures were made during
work in coal mines with charges of expl
STV-40 (Semitinit 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 charge was wrapped in an inner, thin-walled polyethylene tube and an outer wrapping of vitreous fabric. In use, the desired length of charge was cut from the roll. Since standard safety practice has not permitted cutting of charges in operating mines, the expl 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, 37080g (1970) [Factors affecting the amt and compn of noxious gases during blasting were detd. Accdg to results obtb in Donbas (Russia), the amts of CO involved in blasting with Ammonit and Dinoflotit varied within the limits 10-40, 40-100, and 100-150 dm3/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 mix 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 prep'd 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 prep'd by dissolving a poly(vinylbutyral) resin in castor oil 43 (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 "carboxyacrylic" fuel (such as acetic, propionic, tri-chloroacetic, nitropropionic, benzoic or aze-latic acid; or acetaldehyde) are described. On the basis of 100 parts by wt of anhyd HNO3, the compn contd: carboxyacrylic 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 [SnCl2 or Cr(NO3)3] 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 prep'd by mixing anhyd HNO3 13.8, AcOH 10.9, DNT 7.3, AN 64.1, Gantrez AN-169 resin 1.0, SnCl2 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 HNO3 or "carboxyacrylic" fuel (AcOH) component before bringing the gelling agent in contact with the crosslinking agent (Cr chromate). E.g: HNO3 11.8-14.0, AN 70.0-71.5, Gantrez AN-169 10.0, Cr(NO3)3 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) (Porousable slurry expl comps are described, composed of an inorg O-supplying salt, water, thickener & fuel. E.g: granular AN 79.7, Diesel oil 2.4, CCl₄ 8.0, water 7.0, guar gum thickener 0.5, ethylene glycol dispersant 1.0 & NH₄₂-lignosulfonate stabilizer 1.4%)

518) C. Dunlinson & W.M. Lyerly, USP 3431155 (1969) & CA 70, 116788n (1969) [High-performance, water-contg expl comps having densities ca 1.4 were obted by incorporating in conventional oxidizer fuel-water mixts a sensitizer contg a salt of an inorg oxidizing acid (such as HNO₃, HClO₃ or HClO₄) and a base (such as H₂N₂NH₃, MeNH₂, Me₂NH, Me₃N, urea, guanidine, aniline, etc). The preferred salts being MeNH₂•HNO₃, Me₃N•HNO₃ and HNO₃, H₂NC₃H₅•NH₂•H₂O. Thus, a mixt of AN 35, water 15 & MeNH₂•HNO₃ 30 parts was heated with stirring to 54–60°, and a premix of NaNO₂ 15 & guar gum 0.8 part stirred in until thickening was observed (ca 3.5 mins). Then sulfur 2, PhOH•HCHO resin micro-balloons 1, and carbon powd 2 parts were incorporated and finally ca 1% K₂Cr₂O₇ 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 comps 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 (1963) [Dry expl comps 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% non-detonable 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 comps contg Al, KClO₄ 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-naphthoxyacetic acid lauryl amide, 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 KNO₃ and/or NH₄Cl 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 anticaeking props was obted 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 steaic 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 acryl amide crosslinked with a C₄–₅ aliphatic aldehyde as a polymeric gelling agent, are described. E.g: AN 41.77, water 22.50, acrylamide 2.06, (NH₄)₂S₂O₈ 0.98, HNO₃ (75%) 0.32, NaNO₂ 12.86, TNT 19.28, and HCHO (37% concn) 0.37 part. The deton rate was 2890m/sec]

comprising \( \geq 1 \) inorg oxidizing salt (selected from nitrates, chlorates, perchlorates or their mixts), \( \geq 1 \) liq fuel (water-sol or insol), \( \geq 1 \) insol combustible material in particulate form and water]

526) Th. P. Rudy & T. W. Nakagawa, USP 3442726 (1969) & CA 71, 31963-p (1969) (Particulate nitronium perchlorate coated with a complex pyridine salt, suitable as a solid compatible oxidizer in various formulations, is described)

527) J. R. Thorntn, USP 3442727 (1969) & CA 71, 31967-r (1969) (The storage stability and reliability of blasting compns can be improved by the inclusion of a mono- or diester of \( \text{H}_3\text{PO}_4 \) or its salts as the emulsifier for the \( \text{HNO}_3 \). This eliminates the need to add an acid-resistant gelatin stabilizer. Typical emulsifiers are: lauryl \( \text{H} \) phosphate, terradecyl \( \text{H} \) phosphate, etc. The blasting compn can be prepd either by mixing all of the ingredients, or by mixing a preblend of \( \text{HNO}_3 \) and oxidizer salt with the fuel & emulsifier)

528) R. A. Simpson, USP 3442728 (1969) & CA 71, 31969-r (1969) (Gelled nitric acid blasting agent, such as compn contg 70% aq \( \text{HNO}_3 \) 17, \( \text{PhNO}_2 \) 10, sorbitan monopalmitate 1.5, AN 70, Gantrez AN-169 1.0, and 30% aq \( \text{Cr(NO}_3)_3 \) 0.5 by \( \text{w/w} \), 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, 31973-q (1969) (Galactomannan water-bearing expls were improved by increasing the rate at which the galactomannan can be crosslinked by \( \text{Cr}^3+ \). This acceleration could be effected by a sol reducing agent such as \( \text{K} \) antimonyl tartrate. This process is particularly suitable for use in situ formulation and mixing)

530) H. G. Knight, Jr, USP 3442729 (1969) & CA 71, 147404-u (1969) (Aqueous slurries of inorg oxidizer salts thickened with mixts of partially hydrolyzed acrylamide polymers & copolymers and \( \text{NH}_4 \) or alkali metal acrylates are described. Thus, a compn was prepd from water 17.9, propellant 34.8, AN 21.9, \( \text{NaNO}_3 \) 12.9, ethylene glycol 10.9, pine oil 0.1, 22% hydrolyzed polyacrylamide (viscosity 4000cP) 0.7, \( \text{Na} \) acrylate copolymer 0.3, \( \text{Al(OAc)}_2\)\( \text{OH} \) 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, 51829-a (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°C)

532) E. F. Stykhouse et al, USP 3446681 (1969) & CA 71, 31972-p (1969) (Gelled slurry expl compns contg 0.1–5% of a ter-polymer gelling agent (comprising 1–60% acrylonitrile and/or methacyronitrile, 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 \( \text{Cr(NO}_3)_3 \cdot 9\text{H}_2\text{O} \) 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 sensitiizers, and water) solvents for inorg oxidizers, and/or liq fuels, kerosene, glycerol, glycol, etc. Thus, a suspension of Guartec UF-725 in 0.3 propylene glycol was added to a soln of \( \text{HCNO}_2 \) (Fe-free) 10 in \( \text{H}_2\text{O} \) 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 \( \text{NaNO}_3 \) 10, prilled AN 53.45 and particulate Al 10 parts, and finally 0.9ml 1M aq \( \text{Cr(NO}_3)_3 \)/\( \text{lb mixt} \) stirred in]

agent, usually free of self-expl ingredients is described, which includes AN, water, NH₄ClO₄ (or NH₄NO₃), 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 Artexol 34) 5.6, oleic acid phosphate emulsifier 3, AN 100, NaNO₃ 16, H₂O 29 and microballoons 2 parts are mixed with heating to above the emulsion temp (114°F), the mix cooled to 114°F, where gas occlusion occurs, and the mixing is discontinued at 110°F. The product is a blasting emulsion of 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 0.5400 ft/sec.]

[Gelled HNO₃—AN blasting compns were described which included aq HNO₃ solns of an inorg nitrate (NaNO₃), 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 HNO₃ (60%) 60, NaNO₃ 10, MeOCH₂CH₂—maleic anhydride copolymer hydrolyzate 6, and Fe₂(SO₄)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°.]

[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 prep by this method had good oil absorbency and the resulting AN-FO expl satisfactory blasting performance.]

[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 NaNO₂ in combination with H₂N—SO₃NH₄. Thus, an inorg oxidizer salt expl compn was prepd by mixing water 16.1, a propellant 32.1, AN prills 37.6, NaNO₃ 9.7, H₂O 3.1, pine oil 0.1, guar gum 0.8, cross-linking agent 0.08, NaNO₂ 0.055, fumaric acid 0.10 and H₂N—SO₃NH₄ 0.11 part. The compd had d 1.17 and deton rate 5400m/sec at 6° in a 5-inch diam pipe when initiated with 140g Pentolite.]

[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 solid 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.]

(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 HNO₃ (42%BÉ) 14.0, AN 69.5, MeNO₃ 13.0, sorbitan monopalmitate 1.5, water-sol MeOCH₂CH₂—maleic anhydride copolymer Gantrez AN-169 1.0, and
Ct 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 inorganic 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 prep from 89% (+20-mesh) AN pills, 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. Urnsebach, USP 3457127 (1969) & CA 71, 72519q (1969) [Urea-nitric acid addition product expls were obt by controlling the reaction temp during prep in such a manner that self-thickening addn products, rather than nitro cmpds, were formed. The thickened slurry, capable of supporting granular or particulate fuels (such as Al, Mg, TNT, coal, or gilsonite) could be pumped or poured into boreholes. For example, a blasting compn was prep from concd HNO₃ 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, 72517t (1969) [Particulate self-expl nitratated 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 ≥50% unhydrolyzed amide groups and a mol wt of one to 25 million. Such slurries are exp useful for transporting expl sensitizers used in aq slurry blasting agents. Thus, a mixt was prep by stirring together 66% dry NS, 39.8% water and 0.2% Polychlor 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, NaClO₄ 19.50, NaN₃ (≤0.2mm) 0.59, Ca(NO₃)₂+4H₂O 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 3436838 (1969) & CA 71, 114823s (1969); Division of USP 3409708 (CA 70, 21466s) (A solid propellant used as substitute for Dynamite was prep by mixing 7–20% starch with 4.05–16% vinyl chloride copolymer, heating to 80°, and mixing with 60–90% AN, and a perchorlate, chlorate or an alkali nitrate]

545) Heinz Dehn, USP 3436848 (1969) & CA 71, 126729d (1969) [An economical and controllable expl was obt by treating a hydrated salt of a metal (such as Al, Cr, Fe, or Zn) and an oxidizer (such as HNO₃ or HClO₄) with a dialkyl sulfide to drive off all or part of the water of hydration to produce a corresponding cysl dialkyl sulfide solvate. Thus, a soln of Al(ClO₄)₃·6H₂O in Me₆SO was distd at 80° and 10mm (to remove water and excess Me₆SO) until appearance of crysts. On cooling the hexa solvate Al(ClO₄)₃·6Me₆SO pptd and was recovered by filtration and purified by washing with Me₆SO and C₂H₆. When mixed with an oxidizer, it decmpl on denation as follows: Al(ClO₄)₃·6Me₆SO + 12NaNO₃ → AlCl₃ + 12CO + 42H₂O + 6S + 12H₂O. Use of smaller amts of Me₆SO permitted the formation of partial hydrates, such as tetrasolvate dihydrate, Al(ClO₄)₃·4Me₆SO·2H₂O]

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 amount 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 2960 m/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. Pozdnjakov et al, GomyiZh 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 Ammonite 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 Salotrol and its application in open-pit mining. Salotrol 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 3500–4000 m/sec)

550) W.E. Wales & D.J. Nagy, USP 3456574 (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 between 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 before 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_3NaCl_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 3700 m/sec]

553) W.M. Lyerly, USP 3471346 (1969) & CA 72, 4843m (1970) [Water-bearing expl comps, which contd inorg. oxidizers. water & N bases were improved by including 0.2–2% by wt of salt, distilled water. This was mixed with the hot soln of the blasting compn, as an auq soln or as a paste-like dispersion before addition of gelling agent. It modified the crystal of AN resulting in smaller porous crstls. The N-base salts were derived from amines and strong oxidizing acids. A typical expl consisted of AN 36.8, water 15.0, MeNH_3NO_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_3NO_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_3NO_3...
30.0, NaNO₃ 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 prep'd from aromatic nitrocomps and nitramines and/or nitric esters and/or powd metals were desensitized by sol poly-chloronaphthalenes. The use of these comps produced better desensitization to rapid heating, and comparable desensitization to impact than obt'd with emulsified insol waxes)

556) Ph.G. Butts, USP 3473981 (1969) & CA 72, 4846q (1970) (Gas-generating AN-based comps containing melamine 4–8%, which acted as a coolant, greatly reducing the flame temp. For example, compn containing AN 68, Guanidinium nitrate 7.5, C-black 2.0, Na barbiturate 2.0 & melamine 6.5 parts had the calc'd flame temp of 1785°F, while similar comps containing 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 containing sulfur and Na nitrate. It has been suggested (USP’s 3249477 & 3282752) that the sensitivities of aqueous slurries containing NaNO₃ 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, NaNO₃ 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°C to completely dissolve the salts. A mix of S 7.0, powd. gisperite 6.0 and guar gum 1.0 part was added to 60.45 ps of the soln previously described. Then 25.3 ps comm. particulate NaNO₃ was added. This slurry contained 39% NaNO₃, 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 exphin and reduces the amount 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 chge filled with the same chge, but with no tube of the Al–AN mixt]

559) G.H. Walker & C.E. Bailleux, USP 3475236 (1969) & CA 72, 33911n (1970) [A non-aq cap-sensitive expl comprised of MeNO₂ (gelled by 2–8% NC) 10–30 and AN with NaNO₃ or LiNO₃ 90–70%. The explo had a long shelf life]

560) Ch.H. Grant, USP 3475238 (1969) & CA 72, 45635c (1970) [Gelled slurry expl comprising an intimate mix of liq and solid phases are described. The liq phase containing a natural gum (such as guar gum) and was maintained at pH 3.0–6.5 by the addn of an acid (such as HNO₃) 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 NH₃ satd with AN) to initiate crosslinking. Detonation of expl due to usal crosslinking was avoided]

561) H.A. Jessup & L.L. Udy, USP 3485686 (1969) & CA 72, 68552w (1970) [Aqueous expl slurries containing a Rodex crosslinking agent. For example, a slurry containing AN 31.5, NaNO₃ 13.5, water 15.0, guar 0.19, ethylene glycol 0.3, gisperite 6.0, S 3.0, paint-grade Al powder 0.30, coarser Al 1.7 and NH₄ClO₄ 28.5% lost its crosslinking after 6 hrs. Addn of a small amount of a 50:50 soln of Na₂Cr₂O₇ and K₂S₂O₈ resulted in improved crosslinking. A further improvement was achieved by addn of ca 0.25% of borax or Na ligninsulfonate]

562) Y. Takagi & T. Komiyama, Japan P 69 28376 (1969) & CA 72, 33860d (1970) [Prepn of porous granular AN suitable for use in expls]

563) John McKee, Brit P 1171536 (1969) & CA 72, 57360h (1970) [AN-FO expls were
563) 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 NH₂, CO & oxides of N (thus 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(O₃CH)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 initiaton – No 1 cap (80:20–Hg fulminate–KClO₃) ]

566) U.D. Matzke, Erzmetall 1969, 22(B), 365–70 (Ger) & CA 72, 23038u(1970) (Review of use of mobile mixers in prep of AN-carbon and slurry expl comps )

567) Paul Lingens, FrP 1578383(1969) & CA 72, 113993k(1970) (Powd, gelled, or plastic, antifiredamp expls comprising a sensitizer and a ternary mixt of NH₄Cl, 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 accord to the equation: 5NH₄Cl + 3KNO₃ + MgO —> 3KCl + MgCl₂ + 4NH₃ + 10H₂O. For example, a granular expl contg a mixt of NG 5.4, NGc 3.6, KNO₃ 45, NH₄Cl 40 & MgO 6% had O bal +0.2%]

568) L.G. Pavlovskii et al, Gornyī Zh 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 detn 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 NaN₃ 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, NaNO₃ (acrating agent) 11 & water 35 parts ]

570) V.I. Chikunov et al, Gornyī Zh 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 obtb 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: Acqd by 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 insufusable and insoluble in all solvents

571) E. Demelenne, Ann Mines Belg 1969(6), 606–23 (Fr/Flemish); CA 72, 23035t(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 expls occurring on Aug 2, 1968, in 3 boreholes made in the chalcopyrite deposit of Gaisk combine. The ore in which boreholes were drilled contd FeS2, CuS, CuFeS2, CuSO4, SH2O, 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 H2SO4). 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 expls (out of 55) exploded spontaneously at different intervals of time. The reason for such premature expls 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, were 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 Sangrouat-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 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-PrNO3 or AmNO3. 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, 48446 (1970) (A liq expl prep from AN 69:1, 1:1-DNT/DNIX mixt 10, a nonionic surfactant (commp 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, 48411 (1970) (Industrial expls with low shock sensitivity were obtd as for example, by kneading TEGDN (Triethylene glycol Dinitrate) mixed with some NG+NGe and gelled with 0.7%
Collodin 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 den was 1.12, denon 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 denon 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 hetero- geneous 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-val 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, Vzryvnoye-Delo 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\textsubscript{2})\textsubscript{4} and oil. The degree of disintegration and the distribution of particles after the expln were compared with the results of an expr carried out in a manganese deposit quartry 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 Igdonit for breaking down agate-cong rocks is discussed. Although Igdonit 98/2 (AN 98 & Diesel oil 2%) was more suitable, because it caused less disintegration of rock surrounding agate, Igdonit 95/5 was recommended because of superior penetrability of the fuel into AN]

583 B.D. Buclutskii, Ibid 1969, (67/24), pp 186-201 & CA 73, 16940z (1970) [Industrial efficiency of the expl breakdown of mica-cong rocks in open pit mines of the Aldan mica combine was deter. 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. Satyendr & 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 deter 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\textsubscript{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 prep by replacing NG & NGc with Pen- taerythritol Triturate. It quickly gels the NC forming a stable and nonhygroscopic gel which easily mixes with the usual fillers]

586 V. Pesata & S. Vitke, 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/cm\textsuperscript{2}
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 (CanadInsMiningMet) Bull 1969, 62(692), 1317–25 & CA 73, 57619v(1970) (Water-gel expls. A review with 28 refs. New values, expl or calcld, 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, FrAdd 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 4.29g 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 prep by nitrating with mixed nitric-sulfuric acid winding wastes and spun wastes from cotton mills, as described in the patent)


592) B.P. Ekonsson, GerOffen 1808922(1969) & CA 73, 16968q(1970) (Safety expls prep by adding to NG-NGc mixt a desensitizer such as an org ester, or an ether, as for example, diethyleneeglycol monoethyl ether)

593) H.E. Todd, SAvfricanP 69 03621(1969) & CA 73, 16969r(1970) (AN resistant to sublimation and fuming was prep 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 NH₃)

594) G.L. Griffith et al, USP 3489623(1970) & CA 72, 57361j(1970) (Gelling polyol polynitrate–NC expls prep by using nitroparaffin solvents. Polynitrate (I) were not good solvents for NC (II) and, as such, were not ca-

pable 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 thckned 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 MeNO₂ (10g) and allowed to stand in an open cont-
tainer until MeNO₂ evapd. A very firm gel of d 1.43 was obtd. As III, PrNO₂ & EtNO₂ could be used)

595) Laszlo Nagy, BányászKohasZapok, Bányász 1970, 103(1), 47–52(Hung) & CA 72, 68817p(1970) (Expl characteristics of Pexit (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, GomyiZh 1970, 145(2), 56–9(Russ) & CA 73, 27143x(1970) [Investigations of deton failures during blasting by Granulit AS and Trotyl (TNT)]

597) J.H. Paasch, SAvfricanP 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, SAvfricanP 69 04373(1970) & CA 73, 47184k(1970) (Expl slurries comprising an aq sln of an oxidizer (such as of AN) in which dry particles were sus-
pended together with a thickener or gelling agent, and were subject to control over aera-
tion, foaming and other gas inclusions)

599) G.H. Walker & C.E. Ballard, SAvfricanP 69 05414(1970) & CA 73, 57648d(1970) (Expl mixts consisting of powd AN, NaNO₃ & LiNO₃, sensitized with nitroparaffins and gelled with NC or Me vinyl ether, are de-
scribed. They have a long shelf life and are powerfu)

minute mixing of components under controlled conditions is described. An aq slurry contg AN-\(\text{NaNO}_3\) 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, 681692z (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 methylphosphale 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, Gorny\(\text{Zh} 1970, 145(3), 36\) (Russ); CA 73, 37076(1970) (Investigations of processes for initiating Russ expls Ildenit, 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, K\(_2\)(OH)\(_6\) 0.05, ZnCrO\(_4\) 0.5, Zn(NO\(_3\))\(_2\) (buffer) 0.2, C\(_2\)H\(_5\)Cl 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) (Explos 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 \(\text{NaNO}_3\) 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. Feamow, 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 of 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 alkyamine, 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) (Decomp rates of Amm perchlorate expls contg 25 wt % AN were increased and the induction times decreased by including >2 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\(_4\)ClO\(_4\) and 10ps AN with added 0.0135% K\(_2\)Cr\(_2\)O\(_7\), and 0.16% NaCl)
610) SERADEX, GerOffen 1940061 (1970) & CA 73, 57646b (1970) (A permissible expl with low defgmr tendency was prepd by addn of 2–10% thermally stable polyethylene, swollen polystyrene, or urea and 1–10% Na bicarbonate to dmnd expl mixts)

611) K. Ehnhardt, 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 cont An, Na nitrate, a fuel and a thickening agent. Thus, 8.0 parts granulated sugar and 0.6 ps gua gum wereadded at 660 to 87.4 ps sown contg NaN03 22.3, AN 45.0 & water 20.1 parts. Addn of 4.0 ps bagasse mare (or expanded grain flakes) and 5% ag K2 CO3 (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) [Gelatinized 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 tributylphosphon 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. Mottensen & 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, NaN03 10–40, water 10–20, sulfur 0–10, fuel (gylonite or bituminous coal) 1–10, Al 0.1–8 and gua gum (contg small amt of borax) 0.2–2%; and to reduce the d to 1.25, 0.36% of 35% H2O2 in contact with 0.1–0.2% KI or Mn02 catalyst was mixed with the slurry to be pumped into the bore]

615) L.N. Roberts, GerOffen 1940711 (1970) & CA 73, 79087x (1970) (Liquid TNT expls for the opening of geological formaons having cracks in the vicinity of well-bores are described. Expl mixts contg TNT 25.4–47.5, MnO2 25.4–47.5, RDX (Hexogen) 2.5–5.0, HMX (Ooctogen) 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, 56066c (1970) (Thickened aqueous nitrate slurry expl with good water-resistance, nonstickness, and improved flow props without sepn tendencies, such as contg AN 27.63, NaN03 21.0, Plantago gum 0.9, guar gum 0.16, fumaric acid 0.01, HCONH2 1.6, HOCH2CH2OH 0.30, K3Sb2O7 0.003, NH4 lignosulphonate 0.10, TNT pellets 30.0, water 18.3 and Me2CH2CH2OH 0.30 parts. The slurry had a d 1.45 and deton rate 4500m/sec)

617) Wasag-Chemie AG, FrDemande 2000533 (1970) & CA 73, 47186n (1970) [ANFO expls with low content of poisonous gas were obtd by incorporating 3–6% KNO3 or alk-earth nitrate. For example, expl mixt of AN prills 88.7, Diesel oil 6.3 & KNO3 5% (OB –1.4%) produced 3.36 volumes N oxides and 3.6 vols CO, while a similar mixt without KNO3 gave 8 and 9 volts, respectively]

618) N. Tomoishi, JapanP 70 09280 (1970) & CA 73, 79062a (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–polyethylene glycol monostearate. The setting point was <30° and thus the mixing proceeded easily]

619) M.W. Arct, 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 (NH4)2HPO4, chalk and (NH4)2SO4. TNT & m-DNBz were used as comparative stds]

620) J. Chatel de Ragué de Brancion, GerOffen 2003930 (1970) & CA 73, 89728w (1970) [Sensitized AN, plastic or slurred, expls contg AN 31–85.5 and sensitzers (such as NG gel, DNT, powd Al, celulose glycol and/or Pentoil) 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-PolezLisop 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. Incpmt of Al increased the heat of expln. The expl recommded contd Al 17–21, water 15, TNT 8–9%, the rest being AN)

623) A.N. Dubyrev et al., GornyZh 1970, 145(5), 66–8 & CA 73, 89711k (1970) [Gases formed during expls 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 Skol’nyi (Rock); Ammonit 6ZhV and Detonit VA-8. Comps of these expls were not given in CA. They are, however, listed later in this Vol.]

624) F.G. Gagauz & A.V. Drehnitsa, Vzryv.-nayeDelo 1970, (68/25), 19–23(1970) & CA 73, 100623f (1970) [Comps of toxic gases CO & NO₂ and their amounts released during blasting in mines of various ores, by mixts of AN & DNN (Dinitroanaphthalene) was detd. The ore dust after the expln contd 1–7x10⁻⁴% adsorbed CO and 1–2x10⁻⁴% adsorbed NO₂. 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 amounts 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 expls employed in sinking of mine shafts in Krivoi Rog Basin were detd for AN–DNN-based expls. Max amounts 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 connc. The concn of CO₂ formed in dry shafts was higher than permitted. The concn of NO₂ 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 amnt 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 amnt of gaseous expln products was found, but an increase in rock strength caused an increase in CO formacl from 29 to 39 liters per kg expln. Cooln of expln products by water flowing into the shaft caused an increase in the NO₂ concn]


627) B.D. Rossi & V.A. Usachev, Ibid, 1970 (68/25), 64–7 & CA 73, 89709t (1970) (Detn of the amnt 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 initiacl 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) (Derm 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 expls 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 detn for various expls were in better agreement with the calc'd values of relative heats of expls 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., VzryvnayeDelo 1970 (68/25), pp116-22 & CA 73, 89710 (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 these 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= (V1-V2)/V3, where V's are volumes of gaseous expln products (V1, obtd on expln in a steel tube and a sand wad in the Dolgov Bomb; V2, obtd on expln in a free space in the Dolgov Bomb; and V3, 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 segms 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)


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 of 2-4% nitro ethers (VP-1 or VP-3) to Ammonit PZhV-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 PZhV-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 prep 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 prep of Gheksamon no 5 and Gheksamon no 6 is simpler and less dangerous than that of Gheksamon no 1. No comps 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 NH₄ salts (oxalate, citrate, tartarate, carbonate, and fluoride), Na fluoride and urethane as combbstn inhibitors for AN permissible expls was studied. The combbstn rates of expls with addn of inhibitors were measured as a function of pressure (up to 1000kg/cm²). In addn to Ammonit PZhV-20, the following comps were tested: ASA-1, ASA-2, ASA-3 and AF-1. It was concluded that permissible Ammonit-type expls with decreasing combbstn tendency can be obt by using org NH₄ salts (such as oxalate, or fluoride), urethane or NaF as inhibitors. Comps 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(NO₃)₂·4H₂O].

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 NOxides) during deton, was studied with Ammonit 6, Ammonit 6·ZHv and Ammonit 6 with 2 parts Na naphthylmethanedisulfonate (1) 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] (Comps of Ammonit 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 assoc 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 expl 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 comps based on TNT, TNB, Tritonal, etc contained 0.1–15% spray-dried Ca₃PO₄ 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 15% Tetryl would not propagate the deton, while similar sticks contg 1% Ca phosphate (of specific surface area 15m²/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 ag expl slurryes contg small proportions of fuel, high props of water, and no expl sensiti-izers 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 aments of HMeTeA were tested using 39g Pentalite 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 decomp. The catalyst was prep'd by substituting Cr₂O₃ for NO₃⁻ 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 mix of sugar beet pulp 8.0, gisonite 2.6, sulfur 4.0, and gua gum 0.3 part was sad with a soln of AN 42, water 15, NaN₃ 15, guar gum 0.25, and HOCH₂CH₂OH 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 prep'd 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% cross-linking agent (HOCH₂NH₂)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 mix of: fine AN 69, the condensation product of octylphenol with 13 moles ethylene oxide 1.3, NaNO₃ 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 propipt, are described. The sensitizer was prep'd by adding sufficient NC solvent (MeCOBu-lso) 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 isotope to give the residual hard porous 100-2000µ NC particles of d 1.2–1.5 contg some noncolloidal NC. Blasting expl was prep'd by mixing the above densified NC 10–35 with inorg oxidizer salts (such as AN, NaN₃, and/or NaClO₄) 39–56, water 18–31; Hexamethyleneetramonium 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 ferro silicon 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 prep'd as claimed in USP 3442729]

654) Y. Wakaizono & 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 propipt, or fuels; water, a thickening agent guar gum 0.1–2.0%, borate or borates ≤ 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% hexamethyleneetramine (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 hexamethyleneetramine]

655) Bureau of Mines Information Circular IC 8371, April 1968, US Dept of Interior, Pittsburgh, Pa, 15213, "Active List of Pemissible 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. "Pemissible Explosive" is one that conforms to the basic specs and tolerance limits prescribed by current Bureau of Mines schedules: Sched H1 for explosives and Sched 26A for blasting devices
LIST OF SPECIAL NAME DYNAMITES AND THEIR SUBSTITUTES
(Arranged in Alphabetical Order)

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 comps are in CA of the Ref
Ref: S. Tat & E. Hindor, RevMinerol (Bucharest, Romania), 17(11), 490-92(1966) & CA 67, 55801a(1967)


Abelite (Brit). See Vol 1, p A2-R & Gua, Trattato VI(11)(1959), 342

Absorbing 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 cntg 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


Aerolit (Danish). See Vol 1, p A108-L

AF-T (Russ). See under ASA and AF-1 (Also in AddnlRef 643)

Afor 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) Naoum, NG(1928), 411 2) PATR 2510(1958), p Ger 3-R

Agesid A (Ger). See Wetter-Agesid A in PATR 2510(1958), p 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 borcholes 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'matrite in Russ). See Vol 1, p A140-L and PATR 2145(1945), p Rus 1

Alsilitte (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)

Pentritit (Aluminized) (p A147, Table)

LeRoux No. 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 I 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

Amidoène (Swiss). The mixture (b) was a Dynamite which was known for its use in blasting in the Donaube. 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, Sp & Russ). Exps used for commerical 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 Yaremko & Svetlov (1957), p160, where the following types are listed including their properties: Ammonal No 1, Skal'nyi (Rock), Pressovanovy (pressed)–AN (waterproofed), TNT, RDX, Al (powder) & Ca stearate Ammonal No 2, Zby, Skal'nyi, Pressovanovy–AN, TNT, Al & Ca stearate

Ammonal No 1, Zby, Skal'nyi, Patronirovanovy Poroshok (Cartridge Powder)–AN, TNT, Al & Ca stearate Ammonal No 2, Zby, Skal'nyi–AN, TNT & RDX Ammonal VA–2–AN, TNT, Al & Ca stearate or asphalite with paraffin Ammonal VA–4–AN, TNT & Al Ammonal VA–8–AN, TNT, Al & Ca stearate or asphalite with paraffin

Note: The amt of noxious gases formed on exp of Ammonal VA–8 and liberated by exploded rock masses was detd by A.N. Dubyrev et al and described in Gomjyzh 1970, 145(5) & CA 73, 8970lg(1970) (Listed as Addn Ref 623 under DYNAMITE)

Ammon-almatrit No 98 (Russ). See item (c), under Almatrites in Vol 1, p A140-L
Ammonocabasit (Ger). See Wetter-Ammonocabasit A, B, D & E described in PATR 2510 (1958), p Ger 260, Table

Ammoncarbonit or Ammonkarbonit (Ger); Ammoncarbonit (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

Ammondynit (Ger). See Vol 1 of Encycl, p A293-R

Ammonex (Amer). See Vol 1, pp A293-R to A295-R

Ammonitro (Ger). See Vol 1, p A295-R

Ammonignite (Amer). 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 Encyclopaedia, 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

Ammonitro (Ger). See Vol 1, p A295-R
Table 1

<table>
<thead>
<tr>
<th>Components and some properties</th>
<th>No ?</th>
<th>43A</th>
<th>43B</th>
<th>43C ***</th>
<th>No ?</th>
<th>HI</th>
<th>H5</th>
<th>No ?</th>
<th>No ?</th>
<th>No ?</th>
</tr>
</thead>
<tbody>
<tr>
<td>Am nitrate</td>
<td>42.0</td>
<td>46.0</td>
<td>56.0</td>
<td>45.0</td>
<td>46.0</td>
<td>50.0</td>
<td>50.0</td>
<td>55.0</td>
<td>52.0</td>
<td>50.0</td>
</tr>
<tr>
<td>Na nitrate</td>
<td>9.8</td>
<td>8.0</td>
<td>8.0</td>
<td>-</td>
<td>-</td>
<td>5.0</td>
<td>5.0</td>
<td>8.0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Ca nitrate, 4H₂O</td>
<td>-</td>
<td>8.0</td>
<td>6.0</td>
<td>10.0</td>
<td>8.0</td>
<td>15.0</td>
<td>15.0</td>
<td>10.0</td>
<td>10.0</td>
<td>15.0</td>
</tr>
<tr>
<td>Mg nitrate, 6H₂O</td>
<td>8.4</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>10.0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Guanidine nitrate</td>
<td>-</td>
<td>8.0</td>
<td>10.0</td>
<td>15.0</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PETN</td>
<td>9.8</td>
<td>2.0</td>
<td>-</td>
<td>1.0</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PI1-Salz</td>
<td>-</td>
<td>-</td>
<td>5.0</td>
<td>6.0</td>
<td>-</td>
<td>20.0</td>
<td>20.0</td>
<td>20.0</td>
<td>25.0</td>
<td>25.0</td>
</tr>
<tr>
<td>RDX</td>
<td>-</td>
<td>-</td>
<td>8.0</td>
<td>-</td>
<td>10.0</td>
<td>10.0</td>
<td>10.0</td>
<td>10.0</td>
<td>10.0</td>
<td>10.0</td>
</tr>
<tr>
<td>Terra-Salz</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>30.0</td>
<td>-</td>
<td>25.0</td>
<td>25.0</td>
<td>20.0</td>
<td>20.0</td>
<td>20.0</td>
</tr>
<tr>
<td>TNT</td>
<td>-</td>
<td>0.3</td>
<td>0.3</td>
<td>0.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>8.0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>&quot;Vulkanol&quot;(emulsifier)</td>
<td>-</td>
<td>0.3</td>
<td>0.3</td>
<td>0.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Density (cast)</td>
<td>-</td>
<td>1.58</td>
<td>1.61</td>
<td>-</td>
<td>-</td>
<td>1.53</td>
<td>1.53</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Casting-Temperature</td>
<td>-</td>
<td>105</td>
<td>105</td>
<td>-</td>
<td>-</td>
<td>108</td>
<td>112</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Density of Fragments</td>
<td>41m</td>
<td>-</td>
<td>38m</td>
<td>39m</td>
<td>-</td>
<td>39m</td>
<td>-</td>
<td>40m</td>
<td>40m</td>
<td>-</td>
</tr>
<tr>
<td>Mining Effect</td>
<td>21m³</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>References</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>4</td>
<td>5</td>
<td>4</td>
<td>5</td>
<td>5</td>
<td>4</td>
<td>-</td>
</tr>
</tbody>
</table>

The composition given by Römer (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. Naoûm (Ref 1) lists seven formulations, while Beyling & Dtrieph (Ref 2) list four. A typical comp is: Ammon 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 WW2 for military and civilians uses. Table 1 from p Ger 6 of Ref 5 lists most of these expls
Refs: 1) Naoûm, Expls(1927), 119-121
2) Beyling & Dtrieph(1936), 94-95 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 (Amniachnaya Selita Vzryvchatyiye Veshchestva) (Ammonia Salpeter Explosive Substances) together with Amnonal, Ammontol, Ammotol, Dinamon). All these expls are now practically replaced by Igdonit (qv)

Comps 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 comps:

- Ammonit No 8, Ammonit No 8-PV, Ammonit AP-1, Ammonit AP-2, Ammonit AP-2PV, Ammonit AP-5Zbh, Sernyi 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 6Zbh, Ammonit No 7 Zbh, Ammonit, Skal'nyi (Rock) No 1, Ammonit, Skal'nyi No 1 Zbh, Ammonit No 6-PKV, Ammonit No 7-PKV, Ammonit No 9, Ammonit No 10 and Ammonit PZbh-20

In Table XII are given comps 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, 7, 9 & 10 as "modern" Russ permissible explosives of 1962.

Gorkovenko (Ref 6) lists NaNO₃, TNT & NaCl as components of Ammonit PZhV-20, but it seems that AN must also be included in its compns.

Dubyrev (Ref 7) lists among expls investigated, Ammonit No 1, Ammonit No 1, Skol'nyi (Rock) and Ammonit 6ZhV, without giving their compns.

Azarkevich (Ref 8) lists Ammonit 6ZhV without giving its compn.

Notes:
1) Waterproof-coated AN
2) In Ref 3 it is called "Predokhranitel'nii" (Permissible) without assigning it No 8
3) The values are averages from Refs 3 & 4
4) TNT may be replaced by other aromatic Nitrocompds, such as TNX (Trinitroxylene) or TNN (Trinitrophenanthrene)
5) 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:

<table>
<thead>
<tr>
<th>Components</th>
<th>No 6 (Powdered)</th>
<th>No 6 (Pressed)</th>
<th>No 6 (I) ZhV</th>
<th>No 7</th>
<th>No 8 (II) Permissible</th>
<th>No 9</th>
<th>No 10</th>
<th>V-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amm Nitrate</td>
<td>79</td>
<td>79</td>
<td>79</td>
<td>81.5</td>
<td>68</td>
<td>87</td>
<td>86</td>
<td>82</td>
</tr>
<tr>
<td>TNT</td>
<td>21</td>
<td>21</td>
<td>21</td>
<td>14</td>
<td>10</td>
<td>5</td>
<td>8</td>
<td>16.5</td>
</tr>
<tr>
<td>Woodmeal</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>4.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Pine Bark</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>2</td>
<td>8</td>
<td>6</td>
<td>-</td>
</tr>
<tr>
<td>Asphaltit</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1.0</td>
</tr>
<tr>
<td>Paraffin</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.5</td>
</tr>
<tr>
<td>NaCl</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>20</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Density</td>
<td>1.0-1.15</td>
<td>1.25-1.35</td>
<td>1.0-1.15</td>
<td>1.0-1.1</td>
<td>1.0-1.15</td>
<td>0.9</td>
<td>0.9</td>
<td>0.95-1.1</td>
</tr>
<tr>
<td>O-Balance</td>
<td>+0.4%</td>
<td>+0.4%</td>
<td>-0.64%</td>
<td>+0.55%</td>
<td>+3.5%</td>
<td>+2.9%</td>
<td>+1.5%</td>
<td>-0.64%</td>
</tr>
<tr>
<td>Trauzl Test</td>
<td>370</td>
<td>370</td>
<td>370</td>
<td>350</td>
<td>250</td>
<td>310</td>
<td>320</td>
<td>370</td>
</tr>
<tr>
<td>Brisance</td>
<td>15</td>
<td>19</td>
<td>15</td>
<td>14</td>
<td>12</td>
<td>11</td>
<td>12</td>
<td>15</td>
</tr>
<tr>
<td>Detonation Velocity</td>
<td>3800</td>
<td>-</td>
<td>4070</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>References</td>
<td>3 &amp; 4</td>
<td>3 &amp; 4</td>
<td>4</td>
<td>3 &amp; 4</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>4</td>
</tr>
</tbody>
</table>

Ammonite (British). The following Ammonites which passed the Buxton Test are listed in Marshall, Vol 3 (1932), p 119: 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 GB/Britain. 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
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 CO₂ 20.0%; Trauizl Test 215cc b) AN 78.0, K nitrate 5.0, alkali chloride 8.0, meal 5.0 & NG 4.0%; OB to CO₂ +11.8%; Trauizl Test 200cc

Refs: 1) Naoum, NG(1928), 434-35 2) PATR 2510(1958), Ger 5-R

Ammonpek (Russ). See Vol 1, p A382-L

Ammonpentrinitis (Swiss & Ger). See Vol 1, p A382

Ammonpulver (Austrian). Mixture of AN 85 with charcoal 15%, proposed in 1890 as a propellant, 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 propellant. Its modification used during WWII, which may be considered as a sort of Dynamite, consisted of AN 50, NC(12%N) 22, DEGcDN (Diethylene glycol Dinitratite) 22, hydrocellulose 5 & Centralite 1%


Ammons (Brit). See Vol 1, p A286-R

Ammonsalpetersprengstofe. Ger for Ammonium Nitrate Explosives

Ammonsenigelatines (Brit). Se Vol 1, pp A382-L & A383-L

Ammonsuperggelatine (Ger). See Vol 1, p A383-L

Ammutal or Russian Mixture. See Vol 1, p A383-L

Ammonxyl (Russ). Same as Ammoksil

Ammonyaku (Japan). See Vol 1, p A383-L

Ammon. Spanish for Amnonal. 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 conng large amounts of firedamp and coal dust. Its compn and props, which were camed by Bandurin & Rukin, are given in Vol 3 of Encycl, p C447 & C454-R

Ammotol 80/20. Russ industrial and military expl conng 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
Andersen's (Danish). See Vol 1, p A401-R

André (Explosif). See Vol 1, p A402-L.

Anfex. A Russian mining expl consisting of porous, gran slaked AN and Diesel oil. It is more effective in blasting than BA-62(yv), which contains nonporous AN & Diesel oil. Ref: B.N. Kukib, VzryvnoyeDelo 1968 (63/22), 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, p 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 Penrybn (Brit). See Vol 1, p A466-L

Antifume Blasting Gelatin (SoAfrican). See Vol 1, p A466-L

Antigel de Sureté (Belg). See Vol 1, p A466-R

Antigrisuon d'Arendonck (Belg). See Vol 1, p A466-R

Antigrisuon (Explosifs) (Fr). See Vol 1, p A466-R with Table which includes Antigrisuon No 1, No 2, No 3 and II. Also Grisounite roche & Grisounite couche. Table on p A467-L lists Antigrisuon roche, couche and salpetree

Antigrisuoreux (Explosifs) (Fr). See Vol 1, p A367-L, Table, which lists Explosifs anti-grisourex Nno 7, Nno 9 and Nno 69

Antigrisoutines (Fr). Same as Explosifs anti-grisourex [Giuia, Trattato VI (1959), 342]

Antonite caus (Ital). See Vol 1, p A473-L

Antonite gallery, 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 manufct between W1 and W2 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 Addnl Ref 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 Fabrica Naval de Explosivos, Azul, Provincia Buenos Aires are manufct Dynamites in addn to other expls. Dynamites are also manufct at 2 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


Arit. Accdg to info obtd from one of the Ger reptes available in the Library of Aberdeen
Proving Ground, Maryland, it is a mining expl manufd by the VEB (Volkseigener Betrieb) Sprengstoffwerke at Gnashitz, E. Germany. It contains: NG, NC, TNT, sawdust and inorganic nitrates

Arktie (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

Arnoud’s Explosive (Guatemala). See Vol 1, p A486-R

Arocio 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. Pozdnjakov 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, SiO₂ 10, & Amm oxalate 10%); AS-2 (AN 60, TNT 10, NaCl 10, SiO₂ 10, & Amm oxalate 10%); AS-3 (AN 65, TNT 15, NaCl 10, SiO₂ 5 & Amm oxalate 5%); and AF-1 (AN 70, TNT 10, NaCl 12, SiO₂ 5 & Amm fluoride 8%) were investigated by Glazkova & Popova from the point of the effectiveness of their ingredients: Amm oxalate and fluoride as combustn 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 combust 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)


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>
<thead>
<tr>
<th>Composition (%) and some properties</th>
<th>Astralit 2</th>
<th>Astralit 3</th>
<th>Astralit 4</th>
<th>Astralit ON</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonium nitrate</td>
<td>80.0</td>
<td>79.0</td>
<td>68.3</td>
<td>80.0</td>
</tr>
<tr>
<td>TNT + DNT</td>
<td>12.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Vegetable meal</td>
<td>3.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>TNT + DNT + meal</td>
<td></td>
<td>17.0</td>
<td>27.7</td>
<td>20.0</td>
</tr>
<tr>
<td>Charcoal</td>
<td>1.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Paraffin oil</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nitroglycerin</td>
<td>4.0</td>
<td>4.0</td>
<td>4.0</td>
<td></td>
</tr>
<tr>
<td>Oxygen Balance,%</td>
<td>-</td>
<td>+2.5</td>
<td>-</td>
<td>+0.3</td>
</tr>
<tr>
<td>Trauzl Test, cc</td>
<td>-</td>
<td>390</td>
<td>-</td>
<td>375</td>
</tr>
<tr>
<td>Pb Block Crushing, mm</td>
<td>-</td>
<td>16.2</td>
<td>-</td>
<td>16.0</td>
</tr>
<tr>
<td>Sensitivity to Initiation (requires)</td>
<td>No 1 Cap</td>
<td>-</td>
<td>No 3 Cap</td>
<td></td>
</tr>
<tr>
<td>Velocity of Detonation m/sec</td>
<td>-</td>
<td>5400</td>
<td>-</td>
<td>4900</td>
</tr>
<tr>
<td>Density of Cartridge</td>
<td>-</td>
<td>1.09</td>
<td>-</td>
<td>1.03</td>
</tr>
<tr>
<td>Heat of Explosion, kcal/kg</td>
<td>-</td>
<td>957</td>
<td>-</td>
<td>1006</td>
</tr>
<tr>
<td>Temperature of Explosion, °C</td>
<td>-</td>
<td>2170</td>
<td>-</td>
<td>2220</td>
</tr>
</tbody>
</table>
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, VzrymovyeDelo 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. Defret 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.

*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


*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


*Bergenström Explosive or Salite* (Swed). See Vol 2, p B102-L

*Bergmann Explosives* (Ger). See Vol 2, p B102-L

*Berg-Roburite Explosives*. See Vol 2, p B103-R


*Bicarb or Bikarb* (Ger). A type of permissible expl contg large amounts of Na bicarbonate & of Na chloride and small amts of NG. They were patented before WW II 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 162 kcal/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°C 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

*Björkman, (C.G.) Explosives* (Swed). Accdg to Daniel (Ref 1, pp 72-3), C.G. Björkman 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 *glukodine*. Daniel (Ref 1, p 712) also stated that the same scientist patented in 1867 a Dynamite known as *Seranin* (spelled Séfranie in Fr). Its compn is given in this Vol under DYNAMITE, Historical. Daniel also stated on p 396 that C.G.B. invented the so-called Kraft (Poudre), but does not say when. Its compn is: NG 55.36, K chloride
16.96, K nitrate 15.18 & pulverized cork 12.50%

Gody (Ref 2) listed on p398, under Gu- 
kodine, two similar comps of Dynamites in- 
vented 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 

Black Carlit (Japan). See Kuro Carlit

Black Dynamite. See Vol 2, p B165-L

Black Powder (BkPdr) or Gunpowder. A de- 
tailed 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 Paulilres (Fr). See Vol 
2, p B179-R

Blanketed or Covered Explosives. See En- 
veloped Explosives, known as Sheathed Ex- 
plodives

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 Aktie-
bolaget Bofors at Karlskoga, province of 
Vämland, Sweden. Activities of Bofors 
Industries are described in Vol 2 of Encycl, 
pp B218-L to B220-L, but we have no in-
formation 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 manufc Dynamites and other industrial expls are listed in Ref 2: a) Fábrica Presidente Vargas at Piquere, São Paulo State; b) Fábrica de Estrela at Vila Inhomérin, State of Rio de Janeiro c) Companhia Dinamites do Brasil at Rio de Janeiro; and d) DuPont do Brasil Sá Industrias Químicas (Duperial)


Brigg's Explosive (Amer). See Vol 2, p B265-L

Britanite (Brit). See Vol 2, p B300-R

British Ammonials. 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

Brittenites (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


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


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

Cabuecine (Brit & Fr). See under Carboazotine in Vol 2, p C51-R

Cahüsit (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 Wetterammoncabüsit

Calcinit (Calcinithe). A type of mining explosive containing large amounts of technical calcium nitrate \([\text{Ca(NO}_2\text{)\_2 \cdot 4\text{H}_2\text{O}}]\), 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 Calcinit: NG 20, Ca nitrate 66 and charcoal 14%. Stickland (Ref 3) gives for Calcinit 1 manufd at the Krummel Fabrik of D A -G the following composition:

- NGc (Nitroglycol) 6.0, DNT 4.8, TNT 7.2, Ca nitrate (tech) 38.0, Am Niträte 35.5, wood meal 8.0, caput mortuum dye (Fe\(_2\)O\(_3\)) 0.5%


**Callenborg’s Dynamite.** See Vol 2, p C8-R

**Cambrítes (Brit).** See Vol 2, p C13-L

**Camphorated Blasting Gelatin (Austroian).** See Vol 2, p C23-R

**Canadian Dynamites.** See Canadian Patents listed as Addn1 Refs under DYNAMITE

**Camel 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

**Carbidè (Fr).** See Vol 2, p C51-R

**Carbzoazotiné (Brit, Fr & Ger).** See Vol 2, p C51-R (Known also as Cabuesite or Cabiüsït)

**Carbocarbonitê (Brit).** See Carbon Carbonite in Vol 2, p C60-L. Its Ger name is Kohlen-carbonitê

**Carbodynamites (Brit).** See Vol 2, p C52-L and Giua, Trattato VI(1)(1959), 343

**Carbogelatiné (Brit).** See Vol 2, p C53-R and Giua, Trattato VI(1)(1959), 343

**Carbon Carbonite.** Same as Carbocarbonite or Kohlencarbonitê

**Carbonit (Ger).** A permissible Dynamite invented in 1885 by Bichel & Schmidt and modified many times since then. Naoum (Ref 1), Davis (Ref 2) and PATR 2510 (Ref 3) list the following Ger Carbonites (See Table 4)

---

**Table 4**

<table>
<thead>
<tr>
<th>Composition (%) and properties</th>
<th>Carbonit</th>
<th>Carbonit I</th>
<th>Carbonit II</th>
<th>Carbonit Extra</th>
</tr>
</thead>
<tbody>
<tr>
<td>NG</td>
<td>25.0</td>
<td>25.0</td>
<td>30.0</td>
<td>35.0</td>
</tr>
<tr>
<td>Collodion cotton</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>K nitrate</td>
<td>30.5</td>
<td>-</td>
<td>-</td>
<td>25.5</td>
</tr>
<tr>
<td>Na nitrate</td>
<td>-</td>
<td>30.5</td>
<td>24.5</td>
<td>-</td>
</tr>
<tr>
<td>Ba nitrate</td>
<td>4.0</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Spent tan bark</td>
<td>40.0</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Meal</td>
<td>-</td>
<td>39.5</td>
<td>40.5</td>
<td>34.7</td>
</tr>
<tr>
<td>K bichromate</td>
<td>-</td>
<td>5.0</td>
<td>5.0</td>
<td>-</td>
</tr>
<tr>
<td>Na carbonate</td>
<td>0.5</td>
<td>-</td>
<td>-</td>
<td>0.5</td>
</tr>
<tr>
<td>Density</td>
<td>-</td>
<td>-</td>
<td>1.10</td>
<td>1.26</td>
</tr>
<tr>
<td>Heat of Explosion, kcal/kg</td>
<td>576</td>
<td>536</td>
<td>602</td>
<td>-</td>
</tr>
<tr>
<td>Temperature of Explosion, PC</td>
<td>1874</td>
<td>1666</td>
<td>1639</td>
<td>-</td>
</tr>
<tr>
<td>Velocity of Detonation, m/sec</td>
<td>2443</td>
<td>3042</td>
<td>3850</td>
<td>4070</td>
</tr>
<tr>
<td>Truwl Test (10g sample)</td>
<td>235 cc</td>
<td>240 cc</td>
<td>258 cc</td>
<td>-</td>
</tr>
</tbody>
</table>

(See also Kohlencarbonit under Kohlensprengstoffe)

Refs: 1) Naoum, 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 AddnRef 400, under DYNAMITE)

**Carbonit (Brit).** See under Carbonites in Vol 2, p 61-Rand 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 Naoum (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: Naoum, NG(1928), 402

Carbonites, French Modifications. Accdg to Naoum (Ref 2), Carbonites were not allowed in French coal mines on acct of their rather high temp of explosion, which ran betw 2400 & 3000°C. On suggestion of Comm Francsie 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)

Carlit or Koritto. A Japanese military expl suitable for demolition purposes. See Vol 2, p C69-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

Cépék'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

Celitite or Zeltit (Get). See Vol 2, p C125-R

Centralite R11 (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

Chorbrite 418 (Belgian). A permissible expl described by Naquin & Demelenne (Ref) and listed in this Vol under Additional References on Dynamites as No 330

Cheddites. See Vol 2, pp C155 to C161

Chemische Fabrik's Dynamites (Ger). See Vol 2, p C178-R

Chbidite (Canad). See Vol 2, p C179-L

Chlorate Explosives. See Vol 2, pp C202 to C209

Chlorotit (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
Ref:s: 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-Ammomites (Ger). See Vol 3, p C298-L
### Table 5

<table>
<thead>
<tr>
<th>Properties</th>
<th>Chloratit 1</th>
<th>Chloratit 2</th>
<th>Chloratit 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na chlorate and/or K chlorate</td>
<td>70-72</td>
<td>73-75</td>
<td>85-91</td>
</tr>
<tr>
<td>Vegetable meal</td>
<td>1-2</td>
<td>1-2</td>
<td>0-4</td>
</tr>
<tr>
<td>TNT and DNT</td>
<td>18-20</td>
<td>18-20</td>
<td>-</td>
</tr>
<tr>
<td>Paraffin</td>
<td>3-4</td>
<td>3-4</td>
<td>-</td>
</tr>
<tr>
<td>Nitroglycerin</td>
<td>3-4</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Liquid hydrocarbons (flash point not less than 30°)</td>
<td>-</td>
<td>-</td>
<td>5-12</td>
</tr>
<tr>
<td>Oxygen Balance</td>
<td>+3.0%</td>
<td>+1.9%</td>
<td>-</td>
</tr>
<tr>
<td>Lead Block Expansion</td>
<td>290 cc</td>
<td>280 cc</td>
<td>-</td>
</tr>
<tr>
<td>Lead Block Crushing</td>
<td>20 mm</td>
<td>19.5 mm</td>
<td>-</td>
</tr>
<tr>
<td>Sensitivity to Initiation (requires at least)</td>
<td>No 3 Cap</td>
<td>No 1 Cap</td>
<td>-</td>
</tr>
<tr>
<td>Gap Test (using 25 mm cartridges)</td>
<td>8 cm</td>
<td>8 cm</td>
<td>-</td>
</tr>
<tr>
<td>Velocity of Detonation</td>
<td>5000 m/sec</td>
<td>4300 m/sec</td>
<td>-</td>
</tr>
<tr>
<td>Density of Charge</td>
<td>1.57</td>
<td>1.46</td>
<td>-</td>
</tr>
<tr>
<td>Heat of Explosion</td>
<td>1250 cal/g</td>
<td>1280 cal/g</td>
<td>-</td>
</tr>
<tr>
<td>Temperature of Explosion</td>
<td>3645°</td>
<td>3700°</td>
<td>-</td>
</tr>
</tbody>
</table>

*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


*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


*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, 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

Comaro Explosives (Brit & Amer). See Vol 3, p C542-L

Comcob Cellulose Dynamite (Amer). See Vol 3, p C542-L

Comet 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

Comstock 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

Gotter 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

Crattiti (Ital). See Vol 3, p C555-L
Cremonita (Ital). See Vol 3, p C556-L

Cremonita (listed by Pérez Arta). 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

Cugniete (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

Cyclotrimethylenetetramine 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 Cezkoslovenska Sbrojovka Acriova 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 in Prudy (Prague) 14 (3), 72–4 (1966) & CA 65, 5292 (1966). It is listed as Addnl Ref No 264 for DYNAMITE


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 & Karstair's Powders (Brit). See Vol 3, p D18-R

Dean Explosive (Brit). See Vol 3, p D21-L

De Castro (Explosive) (Fr). See Vol 3, p D37-L, where it is erroneously spelled "DeCastro 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 comps are not given in CA.


Delatite (Explosif) (Belg). See Vol 3, p D49-R.

Delborbe (Explosif) (Fr). See Vol 3, p D54-R.

Deligne (Explosif) (Belg). See Vol 3, p D55-R.

Demacar (Explosives) or Macarites (Belg).

Sec Vol 3, p D55-R.

Demétroide, 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 nitro-comps 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.


Dispan Explosive. See in this Vol, p D1153-L.

Dispan Gelatin. See in this Vol, p D1153-L.

Dinaftalit (Dinaphthalite). Russian mining expls described in this Vol. One of such expls is Zeményi Dinaftalit (Granular Dinaphthalite). There are also: Dinaftalit No 1, which contains noncoated AN 88 & DNN 12%, and Dinaftalit No 1 ZbV, which contains waterproofed AN 88 & DNN 12%.


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 grisé" are on p C455-L.

Dinamite. Italian for Dynamite.

Dinamon. Russian for Dynammon. See under DYNAMMONS in this Section on DYNAMITE.

Diorrexina (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

Donaritis. 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

Duplixite. 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) Naoud, NG (1928), 334 2) Beyling & Drekopf (1936), 90—91

Dynamit 2 (Ger). NG 56—61, NC (from surplus smokeless proplnt) 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: Naoud, 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, nitrocompounds 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 Naoud, 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: Naoud, 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, nitrocompounds 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/cm 2. 
It is suitable for use in demolition of re-
inforced concrete and steel constructions
Refs: 1) A. Streitbacher, private communi-
cation, 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 DYNAM-
ITES 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 Guhr-
dynamite 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 other 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 Dyna-
mite 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: Dynamon Sprengstoffe (Aus-
trian & German), Dinonomi (Italian) and 
Dimonony (Russian). They are mechanical 
mixtures of AN (Ammonium Nitrate) with 
various combustible, but nonexplosive sub-
stances, such as sawdust, lignite, bark 
flour, peat, coal dust, charcoal, etc. Dyna-
mons 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, how-
ever, for reinforced concrete, steel construc-
tion and hard rocks

Following are examples:
A) Dynamon of E. von Geldern consisted 
of AN mixed with naturally carbonized cel-
luloses, such as peat, fallen leaves, rotten 
trees, etc (Ref 1)
B) Austrian Dynamon: AN 87-88 & char-
coal 13-12% (Refs 2a & 6)
C) German Dynamon: AN 90 & red char-
coal 10%; its density was 1.01 and detona-
tion velocity 4100-4210m/sec (Refs 2b & 12)
D) German Dynamon: AN 95,5 & charcoal 
4.5%; its density was 0.865 and detonation 
velocity 3380m/sec (Refs 2b & 12)
E) Dynamon I: AN 88 & lignite 12% (Ref 5)
F) Italian Dinamon, described by Belgano
Ref 9a, p 163) and in Vol 3 of Encycl, p C440-L has a quite different compn. No Dinamon are listed in Giua, Trattato VI(1) (1959), Alphabetical Index, p 845
G) Russian Dinamon, 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 crystals 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. Dinamon were introduced in Russia beginning in 1934, but now they are practically replaced by Igdonit (qv)

The following formulations are described in Vol 3, p C441-R: Dinamon “D”, Dinamon “K”, Dinamon “Sh” 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 Trauski 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 “Sh” (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
Other Refs on Dinamos are given in the following Addn 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 15p of water. After cooling, the mass was thoroughly blended with 70p of K chlorate, 30p of K carbonate, 10p of starch in 5p 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

Refr: Daniel, Dict (1902), 232

Dynamosites (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 hot nitrooluenes

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, Colloed 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 Gelnigite) (Ref 1, p 385)

The newer types include: a) Dynamobel No 3 NG 15.0, NC 0.5, Di- & Tri- nitro-toluene 1.5, AN 53.5, woodmeal 5.0 & Na chloride 25.0%. Power 76.4% of Gelnigite (Ref 2, p 120); b) Polar Dynamobel NG (with NGc) 15.0, NC 0.5, Di- & Tri- nitro-toluene 1.0, AN 53.0, woodmeal 5.5 & Na chloride 25.0%. Power 77.3% (Ref 2, p 120); c) Polar Dynamobel No 2 NG with NGc 15.0, NC 0.5, Di- & Tri- nitro-toluene 1.0, AN 62.5, woodmeal 5.0 & Na chloride 16.0%. Power 83.4% (Ref 2, p 120)


Substitute Dynamites

Listed in Additional References Without Their Compositions

Gheksonmy (Hexammons). Russian permissible expls based on 8–10% normally dispersed or finely dispersed RDX (Gheksgon in Rus) and AN (Ammianchnaya Selitra, in Russ). In CA 73 of Ref are listed Gheksonmy no 1, no 5 and no 6 (spelled by the abstractor as hexamon no 1, hexazon no 5 and hexammon no 6, without giving their comps)

Ref: V.F. Starkozheev & V.F. Vasil'eva, VzryvnoyeDelo 1970, 68/25, pp 250–55 & CA 73, 79055n (1970) (Stable-detonating permissible expls, including Gheksonmony no 1, no 2 & no 3 and Ammonit PZhV-20) (Listed as AddnRef 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, AddnRef 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 nonpermisssible 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. Rejs: 1) B.Ya. Svetlov, VzryvnoyeDelo, Sbornik 49/6(1962); RefZhKhim 1963, Abstr No 20N/390; CA 61, 6849(1964) (Modern Russ nonpermisssible 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, 311453e (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 AddnRefs 114, 568, 596 and 602 for DYNAMITE

IGDANITY (Igdanites). Accdg to Khablov (Ref 1), they are nonpermisssible, 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 equi-
valent: "Institute of Mining, Academy of Sciences, USSR, is given.

Svetlov (Ref 2) described modern Russ nonpermiscible 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 ensure full utilization of the expl props of Igdanit, the Diesel oil must be uniformly distributed on granules or crystals of AN. Since there is no chem bonding between 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 dodecylbenzenesulphonate (II), Na dinaphthyl methanedisulfonate (III) and OP-7, which is a mixt of alkylated phenol derivatives of poly(oxyethylene) (IV). The 0.5–1.0 mm 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 100 g nitrate in a burer 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, the 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 Urals bauxite mine showed that on its expln the formation of toxic gases CO & NO2 was lower than from expln of Ammomit 62bV 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 Ammomit 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 bote 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 & NO2) 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.

Iponit. A new Polish safety expl consisting of granulated porous AN 94 & fuel oil 6%. Iponit with min water content made of fuchsin-coated AN with apparent d 1.01–1.1 gm/cc and 1.4 mm 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 mix 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.


Kuro Carlit (Black Carlit). A Japanese industrial expl described in the following Refs:

Ref: 1) K. Ito, KogyoKayakuKyokaishi 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

Monsnit. An expl mixture consisting of AN 72, Amm pircate 23 & petroleum asphalt 5%

Ref: 1) Blinov, v2 (1949) 2) PATR 2145 (1955), p Russ 11–L

Metanit and Miporit. Russian permissible expls prepd by enclosig 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 4900 m/sec. Miporits had a higher resistance to dynamic compression and lower detonation rates than Metanits. Their core consisted of an Ammonit and microporous rubber. For example, Miporit No 1 contg 4.5% rubber and its deton rate was 3570 m/sec

Ref: N.S. Bakharevich, VzryvnyeDelo 1966 (160/17), 143–56 & CA 67, 13424k (1967) (Listed as AddnlRef 310, under DYNAMITE)

Miporit. See above under Metanit and Miporit

Nitrol. A new Yugoslavian mining expl described in Ref:


Nobelit B. One of the Gdr 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 0.1 mm), sensitized with TNT. Exact compn is not given in CA of Ref


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)


Pobedit (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 & 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. Compounds of these expls are not given in Ref 1, pp 25–6 (See also Ref 2)

Bakharevich (Ref 3) studied Ammonits and Pobedit 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 Pobedit to obtain expls, known as Metonits, which did not ignite methane-air mixts and had deton rates of 4620–4900 m/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


5) F.M. Gel’fand et al, Ibid, (63/20), 177–78 & CA 69, 11862h (1969) (Results of testing water-resistant Pobedit VP-4)

Russkoye Smes’ (Russian Mixture). Blinov (Ref 1) gives its compn as AN 50, TNT 38 & TNX (Trinitroxyylene, called in Russian Ksilli) 12% (See also Ref 2). Gorst (Ref 3) gives a different compn: PA (Picric Acid) 51.5 & DNN (Dinitronaphthalene) 48.5%  


Note: Shilling (1946), p 90, calls the compn given by Gorst Russkii Splot (Russian Alloy or Fusion)

Russkii Koktel’ (Russian Cocktail). An incendiary liquid used during WWII against tanks. It consisted of K chloride 80 & Nitrobenzene (or Nitrotoluene, or Nitroxyylene) 20%

Refs: 1) A. Stettbacher, Protar 10, 160 (1944) 2) PATR 2145 (1955), p Russ 20–R

Soletral. 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 Gelpignite, and its rate of deton 3500–4000 m/sec


Securite or Sekurita. A gelatinized O-balanced safety explosive patented recently in Sweden by Wetterholm. It is described in this Vol of Encycl, p D1255-L

Sementinit 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 NaNO₃) 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, NaNO₃ 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 comps were not given in CA of Ref
Ref: F.M. Gel’fand & V.S. Alipchenko, VzryvnayeDelo 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 $\frac{1}{2}$–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 (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 propellant 
Ref: Glossary of Ord (1959), p104-L

"E" (Explosive)(Japan). Blend of AN (Ammonium Nitrate) 40 and TNAnS (Trinitroanisole) 60% 

E-4 Great Enjion. 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 Ersatzprengstoffe (German Substitute Explosives) in PATR 2510 (1958), p Ger 44 (Table)

E-7. A solvent-extruded composite propellant 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 (PBL 5828)(1945) and OSRD4748a (PBL 12095)(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 a core mixture of BrFdr 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 sub-assemblies: 1) Anchor tube – a steel tube,
Soil cross-section portraying EAW-20 Earth Anchor principle. Note cutaway showing tension take-up capability.
Earth Crustal Studies. In recent years, there has been a vast increase in seismic studies devoted to: 1) the earth’s compon and origin, and 2) the detection of nuclear expls. 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


Note 1: This test was developed in France under the name Essai dans la terre by the Commission des Substances Explosives and described in MP15, 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. Stockland, 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 2200 lb bomb (11 tons). Both bombs resembled a torpedo in appearance.


Earthquake Powder (Brit). Expl mixture patented in 1898–1899 by Curtis & Darnford (Engl P 17878) and manufd by Curtis & Harvey, Ltd. Its comp is KNO₃ 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
Ref: 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

**Éborage 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 Smoke-producing 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 Atomic-Energy Commission Rept CF-58-10-43 (1958) & CA 54, 2028 (1960) an explosion which occurred while dissolving the alloy in boiling conc 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 Kostевич (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
Ref: 1) N.W. Berger, SS 1, 150 & 169(1906) 2) M.M. Kostевич, 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
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, glycercin, glycol, etc., acids for nitrations (such as nitric & sulfuric), alcalies (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, cartridgeing, 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.


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 unpolshed cotton by the Schönbein process, but purified more thoroughly. The dried material was loaded uncompressed 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 propnt, even in unrifled weapons (Ref 2, p41).

A slower-burning, nearly smokeless proplnt, was prepd in 1864 by Captain and later Major E. Schultz of Russian 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, Schultz's product contd ca 20% of unnnitratd lignin. Schultz'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 Schultz'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 Schultz's invention and in 1869 a factory was established at Eyworth, 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 Schultz started a factory in partnership with Voltz and Lichtenberger at Herzbach in

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.
Hesse-Darmstadt, Germany (Ref 2, p48)

The next successful smokeless propellant 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 with the nitrates of K and Ba with the addition of coloring-matter and small quantities of some organic comps. The NC, which consisted of soluble and insoluble types, was gelatinized to the extent of 50-80% by ether-alcohol to which a small amount 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 manuf. 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, untnitrated 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, woodcellulose 3.8 and volatile 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 manuf. 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 Bk Pr (Ref 5, p 289). A detailed discussion on this subject is given in Vol 2 of Encycl, p 8322.

In the USA, the EC Powders began to be manuf. 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 on “Bulk Powders” (Ref 5, p 289) lists the following powders with comps similar to EC Powders:

| Components | Percentage
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>No 1</td>
</tr>
<tr>
<td>NC</td>
<td>84.0</td>
</tr>
<tr>
<td>(%N in NC)</td>
<td>(13.15)</td>
</tr>
<tr>
<td>K nitrate</td>
<td>7.5</td>
</tr>
<tr>
<td>Ba nitrate</td>
<td>7.5</td>
</tr>
<tr>
<td>Starch</td>
<td>–</td>
</tr>
<tr>
<td>Paraffin oil</td>
<td>–</td>
</tr>
<tr>
<td>Diphenylamine</td>
<td>1.0</td>
</tr>
</tbody>
</table>

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 propellants for blank fire cartridges. Later, when dense colloidal propellants were invented and used in artillery projectiles, EC Powders were used occasionally in the igniter charges which set fire to the slower burning colloidal propellants. Accdg to Marshall (Ref 2, p 334), some fast-burning bulk powders were suitable for use as propellants 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 manuf. 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 screen.
US Std Sieve and is retained on a No 50 Sieve Properties of EC Blank Fire Powder:
Oxygen Balance to CO₂ +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)
Expiration 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, ButMineApparatus, 2kg wt 19cm (comparable to PETN)
Initiation Sensitivity — minimum detonating charge 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

As was already mentioned, EC Powders and other quick-burning formulations were unsuitable for use as propellants in rifled weapons. In order to obtain propellants suitable for such purpose it was required to prepare propellants considerably slower burning than EC or Schultz’s Powders. This was achieved by using formulations with NC in which the cellulosic structure was completely destroyed by treating pulped NC with colloiding (gelling) agents. Historical development of such propellants 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

EC Pulver. One of the older German propellants: Collodion Cotton 28.0, Gun cotton 26.0, Ba & K nitrate 38.0, camphor 2.0, wood-pulp 4.0, gelatinizer 0.5 & moisture 1.5%

ECRASITE (Ekaosit). 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, per- tards and demolition charges. It was manufd by the Kubin and Sietsch, 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 Lehalleure (Ref 7), Ecrasite consisted of Ammonium Trinitrocreosylate mixed with K nitrate in such a proportion that complete combustion could be obtd. It melted below 100° and was less powerful than PA (Picric Acid). Pepin Lefhalleure also stated that mixt of Ammonium Trinitrocreosylate with Ammonium Nitrate was used as the French Mining Explosive known as L’explosif de mine français, type C, n° 1 lb

Accdg to Chalon (Ref 2), as quoted by Marshall (Ref 3) and Davis (Ref 8), Ecrasite was Ammonium Trinitrocreosylate, prepd by treating 2,4,6-Trinitro-m-cresol with ammonia

Venin, Butlou & Lécorché (Ref 5, p 454) were of the opinion that Ecrasite was Ammonium Trinitro-m-cresylate, which they called Le créezylate d’ammoniaque or simply Crézylate. The same authors called on p 457, the compn consisting of Crézylate 60 & Mélinite (PA) 40%, as Crézylate 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 NH₄ salt.

Accdg to Seitzbacher (Refs 6 & 8) Austrian expl Ekaosit did not contain any Ammonium Trinitro-m-cresylate but was either straight
PA or PA mixed with ca 10% of Dinitro- naphthalene 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 Ektrasit melted at ca 86°.

A great expln of a magazine contg a large quantity of Ektrasit took place at Kiev, Russia during its Austro-Hungarian occupation in 1918.

Hallest (Ref 9) proposed using Ammonium Trinitrocresylate (which he called Ecrapine) in admixture with NG and other ingrdnts, as a substitute for Dynamites, claiming that such mixts are less susceptible to extremes of temperature than are Dynamites.

Giua (Ref 11) stated that Ecrapine is Ammonium Trinitrocresylate and that in addn to using it straight in Austria for loading shells, it was used in various mixts with AN.


**EDCJet** (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 50 mph.


**EDAP.** Abbrev for Ethylene Dioxymonoperchlorate, described in Vol 6 of Encycl.

**EDD.** Ger abbr for Ethylenediamine Dinitrate, known also as DIAMIN.

**EDD** (or EDDN). Amer abbr 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 (MCOM), 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 geared for the production of a chemical defoliants for use in Vietnam.

As of 1968, Edgewood Arsenal employed 3800 civilian employees, and over 1600 military personnel.

Among the items manufactured 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, Ord 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. Pavlenkov, "Encyclopedic Dictionary" (in Russ) St Petersburg (1918), p 726 (Edinorog) and p 2959 (Shuvaloff, P.I.) 2) M.E. Serebrjakoff, "Interior Ballistics"; listed in Vol 3 of Encycl, p XV. Engi 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: KNO₃ 37.50, PA (Picric Acid) 9.38, KClO₃ 18.75, KCl 12.50, sulfur 3.12, Prussian Blue 3.12 & sawdust 15.64%, plus a caoutchouc solution as a binder; No 3: KNO₃ 24.25, PA 15.10, KClO₃ 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 Halcite, 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 Tetramethylcyclopanetane, also known as Nitropentane. 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 prep and properties are described in Vol 6, under ETHYLENEDIAMINE AND DERIVATIVES

**EDTA.** Abbr for (Ethylenedinitrilo) tetracetic Acid described in Vol 6
EDX. Hercules Powder Co's experimental composite solvent-extruded proplnr, developed during WWI. 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 expls 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 expls 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).

Acacc to Du Pont 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
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 dedd in a calorific bomb, which is usually higer, 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")


Effective Fragment. In terminalballistics, 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 relativ") (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 dedd 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)

\[ Q_u = Q - \Sigma(a + \beta x^2) \]

\[ Q_u = \text{Quantity of heat transformed into work by detonation} \]
\[ Q = \text{Potential of an explosive} \]
\[ t = \text{Final temp of the gases of expln after expansion (at atmospheric pressure)} \]
\[ a \text{ and } \beta = \text{constants calcld for some expls in Ref 1, p 464} \]

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")

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 D255 & 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
2) Expls & Pyrots 3(10)(1970)

Effects of Inert Components on Detonation (or Explosion). Described in the paper of Copp & Ubbeholde, 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: NH₃ and N₂H₄ were X-irradiated at 100kr to det the effects of space environment on the storability of propints. At pressures P > 100mm and dosages > 45kr, 0.1% of NH₃ decompd to give N, H and an IR spectrum different than for NH₃. At 1295mm and 104kr, 0.3% of NH₃ decompd to a gas contg N and H. IR spectra on 3 fractions of the product disst in vacuo showed NH₃ in the 1st two fractions and N₂H₄ in the 3rd. Liq NH₃ at 115kr gave 0.5% of condensible gas of N₂H₄ 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% N₂H₄ was converted to 1% NH₃, a noncondensible 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 N₂H₄ at -35°, 75% of the product at -90°, and NH₃ at -196°. P-time curves indicated formation of mols larger than N₂H₄. The possibility of larger N chains is remote, but HN₃ and azides are possible

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 Plastics 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 SympCombust (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 & Bobolov, 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), 92 pp & CA 43, 6326-27 (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°C 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°C, 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, 0.25 g sufficed

Effetbrisant (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, p C345 ff

Effet Neumann (Fr). See Manroe-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 propiant, only from 15 to 40% is actually utilized as kinetic energy to the projectile. Of the remainder the greater part remains in the

<table>
<thead>
<tr>
<th>Explosive</th>
<th>Density</th>
<th>Grams of MF Required</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Room Temp</td>
</tr>
<tr>
<td>MF (Mercuric Fulminate)</td>
<td>1.20</td>
<td>&lt;0.25</td>
</tr>
<tr>
<td>Blasting Gelatin</td>
<td>-</td>
<td>&lt;0.25</td>
</tr>
<tr>
<td>Cheddite No 2</td>
<td>1.0</td>
<td>0.25</td>
</tr>
<tr>
<td>PA (Picric Acid)</td>
<td>0.90</td>
<td>0.25</td>
</tr>
<tr>
<td>Guncotton</td>
<td>0.40</td>
<td>0.25</td>
</tr>
</tbody>
</table>
propellant 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 calc'd by Hardcastle (Ref 3) and by others (Refs 4 & 5). It is presented here in Table E3.

<table>
<thead>
<tr>
<th></th>
<th>Fr-lbs</th>
<th>Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carried away by bullet as</td>
<td>2300</td>
<td>31.4</td>
</tr>
<tr>
<td>muzzle energy</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carried away as gas</td>
<td>254</td>
<td>3.5</td>
</tr>
<tr>
<td>energy to muzzle</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carried away as heat</td>
<td>260</td>
<td>3.5</td>
</tr>
<tr>
<td>Carried away as energy of</td>
<td>35</td>
<td>0.5</td>
</tr>
<tr>
<td>rotation</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Impacted to barrel by</td>
<td>260</td>
<td>29.2</td>
</tr>
<tr>
<td>friction</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Impacted to barrel by hot</td>
<td>1880</td>
<td>26.0</td>
</tr>
<tr>
<td>gases</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Recoil</td>
<td>11</td>
<td>0.1</td>
</tr>
<tr>
<td>Heating cartridge case</td>
<td>5</td>
<td>0.1</td>
</tr>
<tr>
<td>Ejected at muzzle</td>
<td>2315</td>
<td>31.7</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td>7320</td>
<td>100.0</td>
</tr>
</tbody>
</table>

Similar calc's regarding the Mark VI cartridge were done by Kelly (Ref 4).


**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 cusher 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, TransAmInstMining-MetEngrs 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.


**EFR.** Hercules castable rocket propellant 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 chloride 90 & petroleum 10%


**Egg for Manipulation of Acids.** It was mentioned on p A88-L of Encyc, Vol 1, where two references are given. A more comprehensive description is given in Marshall 1 (1917), pp 129-30

**Égoutage des poudres** [French for Drainage (or Dripping) of Propellants]. When the trempage ("water-drying procedure") for removing volatile solvent from propellant 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 KCIO₃, KNO₃, charcoal, tannic acid and rosin
Ref: Daniel (1902), 238

El. 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 ref: was C, now declassified. Eimite is a stoichiometric mix of atomized Mg, recrystallized KNO₃ & 20–40% NC (13.15% N). It is manufactured by using conventional solvent propellant processing equipment. The extruded strands are cut to fit throughout 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)

Eindrohtzünder. Ger for One Wire Igniter or Primer
Ref: Beyling & Drekopf (1936), 220

Einfachezündor. 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 BlkPdr)

Einheitspulver (EP). A German standard propellant of later period of WWII, called "Unit Powder" by Pike (Ref 1). It was one of the "G" Pulvers (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" propnt for all ammunition in order to minimize the differences in ballistics previously obtd when propnls 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

Einheitspulver R61. One of the German WWII rocket proplnts: NC (12.5% N) 59.8, EGDN (Ethylene glycol Dinitrate) 35.3, hydrocellulose 1.5, EthylUrethane 1.1, DPhUrethane 0.8, Irg W.4 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
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 (1950), 177

**Eismine 42 oder Flascheneismine** (Iceme or Flask Iceme). 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 adapted for use as an A/P (Antipersonnel) Land Mine, by encasing it in a concrete sheath contg shrapnel slugs.


**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 M. 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 (KClO₄ + C black) was reduced from 65% to 30%, making its compn KClO₄ 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 casable 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 casable.

**EJE.** A propellant consisting of NC (13.1% N) 26.0%, NG 22.0, EC 2.0, carbon black 7.0, KClO₄ 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-
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 Kümmler Fabrik of Dynamit AG but did not come to the manufacturing stage. These proj contained 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 expls could take place.
Ref: 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 breech 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.

Ekrosit. 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% Dicylglycerin Ternaminate 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.


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:

\[ M = \text{Stress/Strain} = mg2/\pi^2 s, \]

where \( M = \text{Young's Modulus}; s = \text{elongation produced by weight, } m, \text{ in a wire (or bar) of length, } \lambda, \text{ and cross section of radius, } r \).

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).


Elloy. 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

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 charge which disappears accdg to Ohmic conductivity having no relation to the decay of polarization


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) E1ZtZ 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

Electra. See Elektra
delay functioned. Fuze EIZvZ(59A) was of identical construction but twice as large (Ref 1, p 172 & Ref 2, p Ger 2)
b) EIZvZ(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) EIZvZ 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
pyrotechnic delay mixture. On expiration of burning of the delay, the flash compn and the BkPt 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 photo-flash 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 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).

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. Lecastey, 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. Cran, "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 Wheatstone, verbessert von Hipp) for measuring time intervals in solving ballistic problems is described in Cran, Vol 3(1927), 129–32

Electrical Conductivity. See Conductivity, Electrical; Conductometry and Conductometric Titinations 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

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, thermo-electricity, 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
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 exp. 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.


Electrical Firing Properties of Some Lead Compounds of Nitroresorcinol. Lead Mononitroresorcinol (LMNR), PbO₂C₆H₄(NO₂) (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, ppm was conducted under different conditions. The resulting compds showed no variations of buming characteristics and hence in transfer time. In this respect LMNR seemed to behave similarly to Lead 4,6-Dinitroresorcinol (4,6-LDNR), PbO₂C₆H₄(NO₂)₂ (See p D1275-R in this Vol).

Ref: 1) K. Rhujanga Rao et al, R&D Department of Indian Detonators Ltd, Hyderbad, India (No date is given in Ref 2) 2) Gunther Cohn, Ed., 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 Custom 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.
**References:**

1. Custom Materials Inc, 279 Billerica Rd, Chelmsford, Mass, 01824 (No date)
2. Gunther Cohn, Edit, Expls & Pyros 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 comps. For example, in prep 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 prep of expls contg chlorates mixed with aromatic nitrocmpds and animal or vegetable oil, the nitro cmpds 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 sensivities 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 (1963)

**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')

\[ M = \mu \sqrt{F/\mu} \]

where \( \mu \) 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 \( F = mm'/\mu^2 \)

ESU are based on the strength of electrical charges \( (q \text{ and } q') \): \( q = r \sqrt{k_F} \), 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**

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Unit of:</th>
<th>Practical CGS</th>
<th>CGS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>emu</td>
<td></td>
</tr>
<tr>
<td>O</td>
<td>Resistance</td>
<td>1 ohm</td>
<td>1.1124 x 10^{-12}</td>
</tr>
<tr>
<td>A</td>
<td>Current (Strength)</td>
<td>1 ampere</td>
<td>2.998 x 10^{6}</td>
</tr>
<tr>
<td>V</td>
<td>Electromotive</td>
<td>1 volt</td>
<td>0.0033349</td>
</tr>
<tr>
<td>F</td>
<td>Force(Potential)</td>
<td>1 farad</td>
<td>8.0992 x 10^{11}</td>
</tr>
<tr>
<td>C</td>
<td>Capacity</td>
<td>1 coulomb</td>
<td>2.998 x 10^{9}</td>
</tr>
<tr>
<td>H</td>
<td>Quantity</td>
<td>1 henry</td>
<td>1.1124 x 10^{-12}</td>
</tr>
<tr>
<td>J</td>
<td>Inductance</td>
<td>1 joule</td>
<td>10^{7}</td>
</tr>
</tbody>
</table>

CGS is an abbreviation for centimeter-gram-second units adopted since 1874

**Ref:** Hackl'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 manufactured in the US, and not 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 distance that should be kept between the source of RF energy and blasting cap circuits.

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. Ruhlemann. 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 electric fuzes. All of these fuzes were cylindrical in shape and, with the exception of Type 5, used Al for the case.

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 contained 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 electric impact fuze illustrated in Fig E8.

As the bomb was placed in the plane, a
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 condensors, 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 (ElZtZ 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.


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.

**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 Mk 35 Mod 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**

<table>
<thead>
<tr>
<th>Nomenclature</th>
<th>Physical Characteristics</th>
<th>Electrical Characteristics</th>
<th>Explosive Train Milligrams (Mean)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Lead</td>
<td>Max in</td>
<td>Body Length</td>
</tr>
<tr>
<td>XM60</td>
<td>B2</td>
<td>.194</td>
<td>.535</td>
</tr>
<tr>
<td>T65</td>
<td>B1</td>
<td>.194</td>
<td>.695</td>
</tr>
<tr>
<td>T68</td>
<td>B1</td>
<td>.194</td>
<td>.600</td>
</tr>
</tbody>
</table>

*Symbols:*
- Lead — B — Button with length B1 0.065 and B2 0.167 inch  
- Bridge — C — Carbon (1K to 10K ohms)  
- Expl Train — LMNR — Leadmononitroresorcinol  
- MLst — Milled Normal Lead Styphnate mixed with NC lacquer  
- DLA — Dextrinated Lead Azide  
- l — 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, Mk 115 and Experimental Delay Fuze Primer are described in Vol 4, pp D858 & 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. Du Pont'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
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 Mk46 Mod 0 (p D846, Fig 1–27)
b) Instantaneous Navy Electric Detonator Mk5 Mod 0 (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 (p D848-L, no Fig)
e) Instantaneous Army Electric Detonator M48 (p D848; Fig 1–30)

1) Instantaneous Army Electric Detonator M51 (p D848; Fig 1–31)
2) Instantaneous Army Electric Detonator T20E1 (p D848, no Fig)
3) Instantaneous Button-Type Electric Detonator T44 (p D849-L, no Fig)
4) Instantaneous Stab-Electric Detonator T29 (p D849-R, no Fig)
5) Instantaneous Electric Detonator T39 (p D849-R, no Fig)
6) 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 (Leadmononitrosorocinate), LSt (Lead Styrphate with gum binder), MLA (Milled Lead Azide), MLS (Milled Normal Lead Styrphate mixed with NC lacquer), PETN and RDX

**Addtl Refs:**
B) NipponChemDrugCo, JapanP 12093 (1960) & CA 54, 25829 (1960) (A semiconducting SiO or powdered mixture of Si & Zr is placed between 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", Wohntechnische Monatshefter", 1966(11), 488–97, with 20 refs

Electric Detonators, Primers and Blasting Cops. Analytical Procedures. See Std-MethodsChemAnalysis 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—Primmers, 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 Carbonbundum
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 358568 (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:

Electric Exploders or Blasting Machines are discussed in Vol 2 of Encycl, p 212 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 BiKPt, 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 igniting 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)

Electric (or Electronic) Fuze is one which depends for its arming and functioning upon events of an electronic nature. Such a fuse 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, P1BDF 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 containing 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.

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 boursrelet (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 (Eisenkerne) (4), suspended against the compression spring (Federschiene) (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 crossrod (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 pp 137 are not included here because
they do not seem to be any better than the
above described fuze and, besides, their de-
scription 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 ElzJ ZS30 (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",
pD734-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 Flaschen-
mine, described in Ref 2, p Ger 107-R, with
Fig on p D106.
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 con-
sisting of KClO₄ 46.0, Paris Green 20.3,
powd shellac 5.8 & dextin 27.9%. This
presented a green spot for viewing. The
wafer is held in place by a polyethylene
sleeve .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
Refs: Barkley & Dexter Laboratories, Boston,
Mass, Rept 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) “Acuder” 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 8 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 manufactures 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 combustion 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 based on 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 Corp 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.

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 Wattson (which was the first practical improvement of Volta's pile invented in 1800 and modified in 1801 by Dr. Clinckshank) 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, 9/4 tons of BkPdr, loaded in three separate charges, were fired simultaneously by means of a very large Wattson 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 Veyns (Ardèche). For this, a charge 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.
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 manufa 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 259-52 of Ref 1.

The description of current methods of application of electricity for blasting operation is given in DuPont's Blasters' Hdb (Ref 2)

individually, 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 con-
ductor a spark discharge can occur. Then
materials in the path of the discharge may be
ignited (or even exploded) if they are suf-
ciently sensitive and the energy in the dis-
charge is sufficiently high (Ref 27, p 169
& Ref 4, pp 1–2)(See also item VIII. Ele-
ctrostatic Discharges and Sensitivities of
Explosives to Initiation by)
Types of Extrinsic Electricity:
I. Lightning. If it strikes an explosive or
a blasting circuit, detonation is very pro-
able, regardless of any precautions known
at present time. Even a near miss will
probably cause a detonation. Electric charges,
large enough to detonate electric caps, have
been induced in blasting circuits by strokes
several miles away. The danger from light-
ning is considerably increased if there is a
transmission line, fence or stream to conduct
electricity between 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 re-
moved from lightning) and then stored on any
conductive body, such as man or equipment,
isolated from ground. When potential of
the charge 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 po-
tentials.
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
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 pneu-
atomically 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 initia-
tion, 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 Con-
tral 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 between source
and 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 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 explosions 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 premature detonations. This shows that no conductive devices should be allowed to enter a borehole containing 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).


In addn to a brief definition of static electricity and its discharge given under Introduction to this section in item I, 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 photoelectric ionization from the cathode. Protons are produced throughout 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 uses of expls was conducted beginning in 1942 at the US BurMines. 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 demd by Fleischer & Burde (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) prep'd a literature survey on expl initiators in which conductive mixtures are described and some interpretation of results is given. Accord 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 mix should fire with a spread of 0.10 microsecond or a std deviation of 0.30 μsec. 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 résumé 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 vanDaloh 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 (Résumé 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 microseconds or a standard deviation of 0.030 microsecond. 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 sensitivity of expls to electrostatic discharges. 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
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:

\[
E = 5 \times 10^{-9}CV^2
\]

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, contains 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
with the calibrated spark gap (SG). During a test, the condenser is left connected to the power source thru a high resistance (R₁ and R₂) 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 SW₃; L—electrode cabinet safety and illuminating lamp; SW₁—remote-control switch to operate solenoid; SW₂—safety light switch; SW₃—HV mercury cup switch; SW₄—condenser cabinet door interlock switch; R₃—protective water resistor; R₄—tap resistor = 1 meg to 500 meg; F—1/32amp HV fuse; MA—0-25 milliamperes; V—866-A rectifier; T₁—0-135 volt variotran; T₂—plate transformer, 7500 volts rms, each side, 30 ma; T₃—filament transformer, 2.5 volts/5 amp/10000 volts insulation.

FIG E12 Electrode systems

The resistor (R₄) 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 (R₂) 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 (SW₃), 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 (SW₄) before the door to the condenser cabinet can be opened. The light in the electrode cabinet is automatically turned off by the switch (SW₄).
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 (SW3) 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.
### Table E6

**Highest Electrostatic Discharge Energy at 5000 Volts**

for Zero Ignition Probability for Representative Materials

<table>
<thead>
<tr>
<th>Material Description</th>
<th>Highest Energy (joules) for Zero Ignition Probability</th>
<th>Type of Ignition</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Unconfined</td>
<td>Confined</td>
</tr>
<tr>
<td>Amm Picrate (as received)</td>
<td>&gt;12.5</td>
<td>6.0</td>
</tr>
<tr>
<td>Amm Picrate (thru 100 mesh)</td>
<td>0.025</td>
<td>0.8</td>
</tr>
<tr>
<td>Black Powder (KNO₃ type) (thru 100 mesh)</td>
<td>&gt;12.5</td>
<td>1.6</td>
</tr>
<tr>
<td>Black Powder (NaNO₃ type) (thru 100 mesh)</td>
<td>&gt;12.5</td>
<td>1.6</td>
</tr>
<tr>
<td>Diazidodinitrophenol</td>
<td>0.012</td>
<td>0.012</td>
</tr>
<tr>
<td>Fusee Mixture</td>
<td>0.62</td>
<td>0.62</td>
</tr>
<tr>
<td>Igniter Composition</td>
<td>0.21</td>
<td>0.62</td>
</tr>
<tr>
<td>KClO₃/Pb(SCN)₂</td>
<td>&lt;0.00075</td>
<td>&lt;0.00075</td>
</tr>
<tr>
<td>Lead Azide</td>
<td>0.0070</td>
<td>0.0070</td>
</tr>
<tr>
<td>Lead Stynphate</td>
<td>0.0009</td>
<td>0.0009</td>
</tr>
<tr>
<td>Magnesium Powder, grade B</td>
<td>0.007 to &gt;12.5</td>
<td>Deton</td>
</tr>
<tr>
<td>Mercuric Fulminate</td>
<td>0.025</td>
<td>0.025</td>
</tr>
<tr>
<td>80/20-MF/KClO₃</td>
<td>0.054</td>
<td>0.054</td>
</tr>
<tr>
<td>Nitrocellulose (13.4% N)</td>
<td>0.061</td>
<td>3.1</td>
</tr>
<tr>
<td>Nitroglycerin (25°C)</td>
<td>&gt;12.5</td>
<td>0.90</td>
</tr>
<tr>
<td>Nitroglycerin (60°C)</td>
<td>&gt;0.056</td>
<td>1.0</td>
</tr>
<tr>
<td>Nitrostarch</td>
<td>0.007</td>
<td>0.21</td>
</tr>
<tr>
<td>PETN (as received)</td>
<td>&gt;11.0</td>
<td>0.21</td>
</tr>
<tr>
<td>PETN (100 mesh)</td>
<td>&gt;0.021</td>
<td>None</td>
</tr>
<tr>
<td>Smokeless Propellant (graphited, thru 100 mesh)</td>
<td>&gt;12.5</td>
<td>&gt;12.5</td>
</tr>
<tr>
<td>Smokeless Propellant (ungraphited, thru 100 mesh)</td>
<td>0.012</td>
<td>4.7</td>
</tr>
<tr>
<td>Tetryl granular (as received)</td>
<td>&gt;11.0</td>
<td>4.68</td>
</tr>
<tr>
<td>Tetryl granular (thru 100 mesh)</td>
<td>&gt;0.007</td>
<td>4.38</td>
</tr>
<tr>
<td>TNT granular (as received)</td>
<td>&gt;11.0</td>
<td>4.68</td>
</tr>
<tr>
<td>TNT granular (thru 100 mesh)</td>
<td>&gt;0.062</td>
<td>4.38</td>
</tr>
<tr>
<td>Tracer Composition</td>
<td>&gt;12.5</td>
<td>6.2</td>
</tr>
</tbody>
</table>

**Note:** The ignition of secondary HE’s unconfined is apparently due to an explosion 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 compiled 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 electrode-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 BlkPdr (up to 7% moisture) is more sensitive than dry BlkPdr 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 - 0.00052 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 x 0.0003 x 10^-7 x (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, propants 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) with the powdered material itself using a suitable adhesive. Occurrence of expls 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 "kitstick", 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 kitstick is even better.

It was found that an operator sitting on a wooden stool could generate (by frictional contact with the wood) sufficient electrostatic charge (such as 17000–20000 volts) to detonate sensitive expls if he approached them in such a manner that the charge becomes grounded thru it. A charge 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")

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>B–HMX</td>
<td>40000</td>
<td>0.400</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Composition B</td>
<td>12000</td>
<td>0.036</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cu Chlorotetrazole</td>
<td>60000</td>
<td>0.009</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Explosive D</td>
<td>40000</td>
<td>0.400</td>
<td>0.10</td>
<td>0.025</td>
<td>0.16</td>
<td></td>
</tr>
<tr>
<td>HEX M-24.1</td>
<td>20000</td>
<td>0.100</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>HEX-48</td>
<td>60000</td>
<td>0.900</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MOX-2B</td>
<td>36000</td>
<td>0.290</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PETN (2.6 microns)</td>
<td>12000</td>
<td>0.036</td>
<td>0.31</td>
<td>0.062</td>
<td></td>
<td>0.085</td>
</tr>
<tr>
<td>PETN/TNT</td>
<td>8000</td>
<td>0.025</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>RDX</td>
<td>20000</td>
<td>0.100</td>
<td>0.60</td>
<td></td>
<td></td>
<td>0.80</td>
</tr>
<tr>
<td>RDX/Acetylene</td>
<td>24000</td>
<td>0.144</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Black 90/10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ditto 75/25</td>
<td>32000</td>
<td>0.256</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tetryl (on 100 sieve)</td>
<td>8000</td>
<td>0.025</td>
<td>0.020</td>
<td>0.007</td>
<td>0.0005</td>
<td>1.25</td>
</tr>
<tr>
<td>TNT (thru 100 mesh)</td>
<td>12000</td>
<td>0.036</td>
<td>0.077</td>
<td>0.062</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* 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 exps caused by electrostatic discharges were to make the operator's body normally non-conductive, to have manipulating tools resistant to short-circuit 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 microswitch which allowed the high voltage to be on only when the door was 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 amount of energy (0.015 J) is the most commonly accepted energy value that an individual may have with a charge 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 ButMines 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 1/4 to 1/2 inch diam. The materials and medium were placed in a rubber container and rolled on a rolling mill for 2 or 3 hours. For blending the cooled 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 & 0.058, respect-
The evaluation of 60 mg of conductive LA/graphite-95/5 and 200 mg conductive PETN/graphite-25/5, 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 1 microfarad 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 Genter in Ref 33).
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 Gemmer. 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 E25 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 Gommer with those reported by PicArsn or by NOL. Consequently, there is an evident need to determine whether the present sensitivity tests accurately reflect the true sensitivities of expls and the purpose of rept listed here as Ref 33 is to review these methods and to outline a research program for developing improved testing procedures (Ref 35, pp 2-3).

Moore, Smarr & 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 in probability of ignition above the low-energy peak is due to energy loss from the arc to spark formation. In the high energy range, the peak is evidently due to spark discharge alone. LS t (Lead Sphynate) does not exhibit as large a difference between the contact and gaseous discharge regions (Ref 35, p 5)

MSW investigated the effects of a wide range of parameters. Two techniques were used: a capacitor discharge between electrodes with a fixed gap, and a capacitor discharge between moving or approaching electrodes. In the latter technique, actual contact between 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 between 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, pp 5-6).

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, p 6).

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 hPtMs 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
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 addn 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 exprs of smaller particle sizes were more sensitive. Colloidal LA was the most sensitive of all the exprs 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 Ganzner (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 $10^{-5}$ 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 exprs 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 design of minimum energy, or for the estimation of acceptable energy levels in hazards analysis (Ref 35, p 12).

Geissner (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 amount 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 117, 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 obt, 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
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 obtained. Actually one should obtain a minimum energy for every given configuration and test setting. Wyatt (Ref 20) obtained 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 reproducibility 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 Electrostastic 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, USNatl Bureau 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 $3 \times 10^{-4} \mu F$; detd that surface resistance of an insulator, such as wool may decrease by a factor of 16 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]
5. J. Fleischer & J. B. Buttle, USP 2421778 (1947), p 5 & CA41; 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]
7. R. F. Wilkerson "Research and Development in the Field
Table E8

<table>
<thead>
<tr>
<th>Explosive</th>
<th>Before Tumbling (Joules)</th>
<th>After Tumbling (Joules)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure LA</td>
<td>1.8</td>
<td>5.8</td>
</tr>
<tr>
<td>DLA</td>
<td>2.0</td>
<td>9.6</td>
</tr>
<tr>
<td>PVA-LA</td>
<td>0.2</td>
<td>0.7</td>
</tr>
</tbody>
</table>

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", Pic-ArsnTech Rept 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", USBuMines-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 Exps & Pyrots 2(6) (June 1969)] 30b) C.T. Davey, The Franklin Inst, "Why Ground", in Exps & 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", ProcSixthSymp 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. Genter, "An Electrostatic Sensitivity Test of Composition B", PicArsnTechRept 4119 (Dec 1970) 34) P.V. Satyavatlan & Kurti (Indian Detonators Limited, Heyderabad 500018, India), CurrentScience 41(19) (Sept 10, 1971), pp 663–65. Abstracted in Exps & 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]

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 (Anti aircraft) guns larger than 8.8 cm. One of such primers EIZ C/22 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 dinitroresorinate 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 138–1)

### Filling Composition

<table>
<thead>
<tr>
<th>Lead/Tin Foil Disc</th>
<th>Pressed at 1200 kg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Varnished with NC</td>
<td>Dead load or higher</td>
</tr>
</tbody>
</table>

### ZINC PLATED

- Steel Shell
- Steel Support Ring
- Steel Contact Ring
- Insulating Cup (Plastic Material)

### Lead Tin Foil Washer

0.05–0.1 mm

**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:

- Bridge wire ("fuse") (such as of 80/20–Ni/Cr alloy) was soldered to two leads consisting of two metal foli strips separated by a millboard, serving as an insulator; b) Fusehead;
- c) Molded Plastic insulator; d) Brass contact
This produced a low-tension fusehead (Ref 2, p Ger 53-R)

Note: The "comb" shown in Fig E19 was used at Trisdorf 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" (mixture 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 compos, the back of the comb was sheared off (Ref 2, p 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. W. O. S. "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

<table>
<thead>
<tr>
<th>Item No</th>
<th>Nomenclature</th>
<th>Physical Characteristics</th>
<th>Elec Characteristics</th>
<th>Firing Time</th>
<th>Explosive Train</th>
<th>Priming</th>
<th>DLA</th>
<th>Base</th>
<th>Other</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>XM85 B1</td>
<td>.194</td>
<td>.300</td>
<td>W</td>
<td>16 2.5 500 K</td>
<td>MLSt</td>
<td>40</td>
<td>-</td>
<td>II &amp; D1</td>
</tr>
<tr>
<td>2</td>
<td>XM87 B [see Note (a)]</td>
<td>.194</td>
<td>See Note (a)</td>
<td>C</td>
<td>.004</td>
<td>100 200</td>
<td>5K See Note (a)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>XM88 B2</td>
<td>.194</td>
<td>.315</td>
<td>C</td>
<td>.004</td>
<td>100 200</td>
<td>50K MLSt</td>
<td>40</td>
<td>- II &amp; D1</td>
</tr>
<tr>
<td>4</td>
<td>XM89 B [see Note (a)]</td>
<td>.147</td>
<td>See Note (a)</td>
<td>SG</td>
<td>.0016</td>
<td>500 2000</td>
<td>5 CLA</td>
<td>See Note (a)</td>
<td>-</td>
</tr>
</tbody>
</table>

**Symbols:**
- Lead — 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 Stypnate mixed with Nitrocellulose lacquer
  - II 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

### Electric Proximity Fuses (Ger).

The type of fuse, described in Ref 1 and designated as E1AZ(26) was cylindrical in shape and coolant (see Fig E20) the charging plungers AB (surrounded by insulating material) a charging condenser C1, a firing condenser C2, resistances R1 & R2, 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 fuse case and was aligned with the air tube lead from the nose of the bomb C250Flam. The base of the fuse was threaded to receive the gaine, which housed the electric primer (which contd match compn and BkPdr), the detonator (which contd LA/LSt mixture over 37.2, Ti 11.8, Acetylene black 1.0%, or FA composition 874: N-Pb stypnate 40.0, Ba(NO3)2 44.2, Ca Si2 13.0, Tri-nitroresorcinol (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 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 of the 20mm ammunition. A typical composition for these primers is as follows:
Pb stypnate 40.0, TNT 10.0, Ba(NO3)2
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 R2 to C2 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 tremble 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/cm².

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-R1 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 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 fuze, 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 Du Pont Co.
All-purpose Mk 1 Electric Squib

There are also electric squibs which are used for military purpose. One such device is the "US Navy All-Purpose Mk 1 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 bezel (5), which may be LS(Lead Styphnate), LA(Lead Azide) or LMNR(Lead Mononitroresorcinare) + KClO₃ (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 McCormick 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 24 thread

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
<table>
<thead>
<tr>
<th>Squib No</th>
<th>Diameter (In.)</th>
<th>Length (In.)</th>
<th>Type Bridge</th>
<th>Type Lead</th>
<th>Charge Weight</th>
<th>All Fire Current</th>
</tr>
</thead>
<tbody>
<tr>
<td>M1A1</td>
<td>0.270</td>
<td>0.370</td>
<td>Wire</td>
<td>Wire</td>
<td>65 Mgs</td>
<td>0.545 Amp</td>
</tr>
<tr>
<td>M2</td>
<td>0.270</td>
<td>0.370</td>
<td>Wire</td>
<td>Wire</td>
<td>65 Mgs</td>
<td>0.545 Amp</td>
</tr>
<tr>
<td>XM3</td>
<td>0.287</td>
<td>0.365</td>
<td>Wire</td>
<td>Wire</td>
<td>90 Mgs</td>
<td>5.0 Amps</td>
</tr>
</tbody>
</table>

**NOTES:**

1) 20% Diazodinitrophenol (DAxDNPh) 60% Potassium Chlorate, Grade I, Class C 15% Charcoal, Class D 3% Nitrosochrom (NS)

2) 32% Lead Thiocyanate 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

**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 tell-tale 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 Mark 20 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.

**Electric Time Fuze (ETF).** Its definition is given in Vol 4, p D881-L.
Electroanalysis. See under Electrochemistry

Electroanalytical Analysis. See under Electrochemistry

Electrochemical Nitrations. A method developed in 1936 in Sweden by Öhman for prep 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.


Electrochemistry, Electrochemical Engineering, Electroanalysis and Electrodesorption

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 electrodissolution(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, as 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 base 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 semi- micro 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 electrogrographic 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

Electrodecentration 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)

4) F. Fichter, "Organisch Elektrochemie", Steinkopf, Dresden (1942) (From the book "Die Chemische Reaktionen"., vol 6) (Reproduced by Edwards Bros)
5) S. Glassstone, "An Introduction to Electrochemistry", VanNostrand, NY (1942)
9) J.J. Lingane, IEC (AnalEd) 16, 147-52 (1944); 17, 332-33 and 640-42 (1945) (1946)
10) V. Tamburini, "Analisi quantitativa per eletrolisi", Tipogr G. Fraioli, Alpino, Italy
12) H. Diehl, "Electrochemical Analysis with Graded Cathode Potential Control", C.F. Smith Chemical Co, Columbus, Ohio (1948)
13) F.T. Rabbitts, Anal. Chem. 20, 181-82 (1948) (Mercury cathode cell for rapid electrolysis)
23) B.E. Conway, "Electrochemical Data", Elsevier Press, Houston, Texas (1952)
26) K & O, "Electroanalytical Methods", D.J. Gross & R.W. Murray); 784-841 (Electrochemistry by G.C. Akerlof; 841-46 (Electrodeposition 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's Dic (1944), 298-L.

Electrodynamometer. An instrument for measuring the intensity of faradic and alternating currents  
Ref: Hackh's Dic (1944), 298-L.

Electroendosmosis. The production of endosmosis(qv) by an electric current (Compare with Electro-osmosis)  

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).  
Morphy & Menichelli of Jet Propulision 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 0.51mm (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/m² (10000 psi) against the bridgewire. The unit is completed by hermetically welding a metallic end seal onto the body.

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 charge. 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. Morphy, Jr & V.J. Menichelli, Ordn 57, 236-37, Nov-Dec 1972

C) Pelme Division of Quantec 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)  
E) Anon, "Electroexplosives Handbook", published by Hollex, Inc, 2751 San Juan Rd, Hollister, Calif, 95023, listed in Expls & Pyrots 2(8)(1959) (A pocket size, 33pp, booklet congl 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. Wamer & 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, 3357031, 3546623, 3646471 & Brief 1159144) J) 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 telemetrying 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 58/8(1958) (Conf) (Not used by us) 2) Anon, "Safety Principles for Operations Involving Electroexplosive Devices", NavOrdRept OD 10775(1959) 3) R.E. Grove, Naval Weapons Laboratory (NAVWPNLab) TM
No W-20/60 (1960) HERO Testing; Techniques and Procedures, Dahlgen, 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 Electromagnetic Energy", NOTS TP 2629 (1961), China Lake, Calif
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, FILLTS, Model 13
7) HERO Newsletter, "A Quarterly Review on Hazards of Electromagnetic Radiation to Ordnance", US Naval Weapons Laboratory (Nov 1967), Dahlgren, Virginia

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 in the explosive 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

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 podes 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 Re/s: 1) J.L. Austing & Richard Gortowski, "Circuit for Studying the Electrostatic Sensitivity of Electroexplosive Devices", Explosives 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 D1088-L

A number of non-destructive tests have been proposed, but they are not standardized nor in general use as yet. See following article
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 manuf acture. 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 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 (i.e. condition of bridgewire weld and intimacy of contact between the bridgewire and expl or pyrotech charge) 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 mix in contact with them. The fundamental equation used in these measurements is:

$$C_p \frac{d\theta}{dt} + \gamma \theta = I^2 R_p (1 + \alpha \theta)$$

where:
- $C_p$ = Heat capacity of the system, watt sec/°C
- $\theta$ = Temperature rise above ambient, °C
- $t$ = Time, secs
- $\gamma$ = Heat loss factor, watts/°C
- $I$ = Current in the form of step function, amperes
- $\alpha$ = Temperature coefficient of resistance, ohm/ohm/°C

In the paper of Harwood & Stewart (Ref 3) are discussed methods for arriving at values for $\gamma$ and for determining the thermal time constant. It is concluded that establishment of proper ranges of $\gamma$ 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 work at PicArs, Naval Ordnance Lab, and The Franklin Institute, and he lists seven documents of years 1954 to 1963, which are available from the Defense Documentation
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-milliseec pulses to the bridgewire with 50-milliseec cooling periods between 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 milliseconds required for the wire to reach approx 63% of its steady-state average temp. Instrumentation techniques allow for dem 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 between these qualitative signals and faults that exist in the EED design or in manuf have been successfully detd. At the present time, most of the critical failure mechanisms within the EED bridgewire systems have been correlated to heating-curve irregularities.

Essentially, each EED design has to be calibrated for fault type. Poor bridgewire welds have been identified and classified. Cases of air gaps existing between 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).

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 Staphylate 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 μ capacitor charged to 1.5 volts. Produced in batches of over 1000 (See Fig E25)

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

Electroluminescence. The emission of light as a consequence of electrical discharge in gases. Typical examples are neon lights, mercury vapor lamps and lightning

Electrolysis. Decomposition of a compound (liquid, molten or in solution) by an electric current, which results in the local and spatial separation of the ions of an electrolyte (qv)
and the transfer of their respective charges
Ref: Hackh’s Dict (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 solutions of acids, alkalies,
or salts
Ref: Hackh’s Dict (1944), 298-R

Electrolytic Cell. A combination of a liquid
or semi-liquid electrolyte (solin 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 wound an insulated wire; so long as
an electric current passes thru the wire, the
iron has magnetic properties
Ref: Hackh’s Dict (1944), 298-R

Electromagnetic Cannon (Elektromagnetische
Kanone, in Ger). Prof C. Birkeland of Norway
patented in 1906 an invention consisting of a
cannon using as power for propelling a pro-
jectile an electromagnetic effect resulting
from electric current flowing thru wire coils
(solenoids) wound around the barrel.
Accdg to information published in Russian
military journal "Russkiy 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 propellant 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 propellant
gases. Absence of flash and smoke is a pro-
PERTY particularly desirable for weapons operating
in closed chambers, such as casemates in for-
tress defenses and in turrets of warships. The
disadvantage of such a cannon was the re-
quirement of tremendous amount of electric
current for its operation. For example, calen
by an Austrian Captain Spath 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 Ober-
leutnant Justrow published an article entitled
"Die elektrische Kanone", listed here 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 numer-
ous closely connected in series, ring-shaped
electromagnets, which, on account of their
excitation winding, can be called electromag-netic coils or spools (Spulen)

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
calen of his cannon, Rogge undertook this
work. He gave a detailed calen 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 calculated to be 1/32.5 second.

Rogge also calculated 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 "Parist Gun" (Pariser-geschütz, 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.


4) Dipl-Ing E. Rogge VDE, "Probleme um Elektromagnetische Kanone", SS 34, 132–35(1939)

**Electromagnetic Compatibility (EMC).** In-house and site testing services are available for EMC. The Spectrum Control, Inc is equipped for testing to MIL-STD-451, MIL-STD-618B and similar RFI (Radio Frequency Interference) specs and also performs testing in accordance with FCC (Federal Communications Commission) and HEW (Health, Education & Welfare) requirements. The personnel are familiar with RADHAZ (Radiation Hazard) filter design and measurement problems.

The company also manufactures a complete line of filters, feed-thru capacitors, variable ceramic capacitors, RFI shields, and knitted wire gaskets.

Ref/s: 1) Spectrum Control, Inc, 152 E Main St, Fairview, Pa, 16415 2) G. Cohn, Ed, Expl & Pyro, 4(4), April 1971

**Electromagnetic Field; Detonation Wave in.**
See Vol 4 of Encyl, 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.

Thus electronic measurement of the power in the test environment, the chamber provides a means of estimating the amount 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 propellant 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 = milliamp; KHz = kilohertz and MHz = megahertz

2) G. Cohn, Editor, Explosions & Pyroids 3(8), Aug 1970 (Review of Ref 1)

Electromechanical Fuze. Its definition is given in Vol 4, p 3882-L, under Mechanical Fuze.


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 4.77x10^-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, 1.67x10^-24 grams. While an atom is the smallest particle of an element that can be isolated by chemical 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 atom.

The heaviest atoms have outer electronic shells containing 18 and 32 electrons. The number of electrons that can be gained or lost by an outer shell to form an ion (containing 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, xenon and radon. The nucleus of the atom consists of positively charged

---

**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 100 000 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 (O₂), hydrogen (H₂), nitrogen (N₂), sulfur (S₈), phosphorus (P₄), 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 (H₂O), sodium chloride (NaCl), sulfuric acid (H₂SO₄), etc (See also "Electrons' and Neutrons' Action on Explosives")

**Refs:** 1) Hack’s Dictionary (1944), 299-L (Electron), 568-R (Neutron), 691-L (Proton).

**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 electrostatic charges. Production techniques including pulsed beam, beam deflection (for various weld shapes, e.g. round, square and elliptical) and multi-station fixtures have been applied to reduce the number of pump-down operations.


**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 arts 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 deter 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.


**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 multishot 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 Encyc., under CAMERAS, HIGH-SPEED, PHOTOGRAPHIC, on p CI4-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 pCI7-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 ballisitic 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".


**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.
Electron 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 Encyclopædia and references are listed


(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 existing 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 programing 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 & aiming 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 mix was very unstable, the mix 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>
<thead>
<tr>
<th>Components</th>
<th>No 2 (Ref 1)</th>
<th>No 2 (Ref 2)</th>
<th>No 3 (Ref 1)</th>
<th>No 3 (Modified)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AN</td>
<td>94–96</td>
<td>91–90</td>
<td>70–75</td>
<td>71–75</td>
</tr>
<tr>
<td>Ba(NO₃)₂</td>
<td>–</td>
<td>–</td>
<td>16–21</td>
<td>18–20</td>
</tr>
<tr>
<td>Woodfloc</td>
<td>6–4</td>
<td>9–10</td>
<td>6–9</td>
<td>7–10</td>
</tr>
<tr>
<td>(slightly roasted)</td>
<td>–</td>
<td>–</td>
<td>6–9</td>
<td>–</td>
</tr>
<tr>
<td>with starch</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pine resin (purified)</td>
<td>–</td>
<td>–</td>
<td>6–9</td>
<td>–</td>
</tr>
</tbody>
</table>

Ref: 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 eyepiece. 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 Borties, 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. Brücke 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. Bruchs. 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 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)

Electrons' and Neutrons' Action on Explosives (Irradiation with Electrons and Neutrons of Explosives). Kallmann & Schränkle (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.

Murauor (Ref 3) has also observed the explosion of Silver Acrylile when irradiated with high-speed electrons. On the other hand, Trillat & Murauor (Ref 2) reported that they could not initiate expls, such as TNT, PA, Amm Picrate, NC, Smokeless Propellants and even NI₃ (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 decomp of nitrogen iodide at low pressure and showed the reaction to be:

\[ 2\text{NI}_3 \rightarrow 2\text{NH}_3 + \text{N}_2 + 3\text{I}_2 \]

Its activation energy is 18–19 kcal and its heat of formation is -35 kcal/mole. The decomp reaction is retarded by ammonia & water and also by an increase in pressure over the solid.

Bowden & Singh (Ref 5) found that when crystals 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, crystals of K nitrate (mp 334°) and also some metallic wires were treated in the same manner as LA. During these experiments, the crystals 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 crystals of expls with slow neutrons (flux 10⁶ neutrons per cm² per second). Bombardments of Silver Acrylile and of Pb, Cd and Li Azides produced no explns, although some changes (such as darkening or changes in color) were observed in the crystals.

In a second series of experiments, the crystals 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 NI₃ did produce an expln, but this was not due to neutrons, but simply to the well known fact that NI₃ usually explodes at ordinary temp as soon as ammonia (which is usually mixed with NI₃) 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, JP4 Propellant, LA, DDNP, NG & MF. No correlation was found between radiation resistance & thermal stability, mech sensitivity or ignition temp.


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 (ekathenium) and 94 for ekaosmium.

**Refs:** 1) Hackh's Dict (1944), 299-L and 629-30 2) Lange's Handbook 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 currents.

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.


**Electron Tube Chronographs** (Elektronenröhrenchronographe, in German). They are devices for measuring time intervals which incorporate 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 CHRONOGRAPHES, 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).


**Addl Refs:** A) J.F. Roth, SS 28, 42-6 (1933) (Electronic Tube Chronographs for Laboratory Method of Determining Detonation Velocities)
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 sol is acidic or basic. Thus, the acidity of the sol 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 Encyclopedia (Ref 6). 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 Electrophoresis). (Ref 7) Kirk & Othmer 1965, 846-65 (Electrodialysis)


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 insu- lated 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. Electro- static pertains to static electricity or elec- tricity at rest. It can be produced by friction or atmospheric electricity. Electrostatics is the science of static electricity. Electro- static discharges are frequent causes of fires and explosions. This problem is dis- cussed 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 poten- tial

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 Currents 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, Edir, Explo & Pyrotes 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,

\[ F = \frac{1}{k}(qq/r^2) \]

where F is the force, k a constant depending on the constants used, and q & q 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^-8 EMU = 10^9 x 3ESU = (VoltSec)/Ohm = 1/96489 Faraday, where EMU is abbr for "electromagnetic unit" and ESU for "electrostatic unit".

(See also under Electrostatic Units)

Ref: Hackh's Dict (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
ticles of sulfur trioxide (SO₃) 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 entrainment 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 Hohlfield, who tried to apply it, apparently unsuccessfully, as early as 1824. Guitard (1850) and then Sir Oliver Lodge (1885) independently described methods essentially similar to that of Hohlfield. Shortly 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 World War II, 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 diameter and from 5 to 15 feet long. Its lowest 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).

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., is 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 7,500 to 10,000 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.
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, p 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 electric 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 Encycel, p C547-R)


**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 charges 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.
2) G. Cohn, Edut, 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 through 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)

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 fkd 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 Sehger & Eterling (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/hr (22 miles/hr).


Elektro (Electra). A WWII, German continuous wave navigation system using radio beacons providing multipe equispaced pattem.

Ref: Glossary of Ord (1959), 105-L.

Elektron (Electron). Accdg to All & En Expls (Ref 1) it is an alloy consisting of 8G/14Mg/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

Elektronbombe (Electron Bomb). An incendiary bomb invented in Germany in 1917, but not used until WWII. Its description is given in Vol 2 of Encyclopedia p B234 to B237 and on p B235 it 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

Elektronmetal Staub (Electronmetal Dust). An explosion of dust produced during machining operations with electron metal tool
place before WWII at one of the German plants. Bäck, who reported the accident (Ref), recommended applying, during machining, a constant stream of water to remove any Mg/Al dust

Ref: W. Bäck, Oberflächentechn 13, 55–6 (1936) & CA 30, 3238 (1936)

Element. See in this Vol under Electron, Atom, Atomic Number, Atomic Weight, etc


Eleostearic Acid (Eleomargaric Acid or Octadeca-9,11,13-trienoic Acid),

C₁₄H₂₃COOH; mw 280.44; colorless, rhombic scales; mp 48°C (α-form) & 71°C (β-form); bp 235°C at 12mm pressure (α-form); while β-form slowly decomposes. This unsaturated, monobasic, aliphatic acid occurs in oils of the seeds of Elaeococcus vernicia (Japanese wood oil) and tung oil. It was first investigated in 1875 by Cloëz (Refs 1 & 2) who named it oleostearic acid. The name was changed to oleostearic acid by Maquenne (Refs 1 & 3). Kamerata (Ref 4) obtained it by treating tung oil with NaOH in water & alcohol, and refluxing for one hr on a water bath. Morell (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 & sol in alc; its α-form is very sol in ether, while β-form is sol in CS₂.

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), 1515 (1918); 2) S. Cloëz, CR 81, 469 (1875); 3) L. Maquenne, CR 135, 696 (1902); 4) T. Kamerata, JCS 83, 1043–45 (1903); 5) R.S. Morell, JCS 101, 2083–89 (1912); 6) B.H. Nicolet, JACS 43, 938–40 (1921); 7) Kirk & Orthmer, 1 (1963), p 231 (Table): 8 (1963), p 815 (Table)

Elephant Brand Gunpowder (Brit). A type of BK/Dr manufd at the end of the 19th century by Curtis & Harvey, Ltd of London. It consisted of KNO₃ 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 NH₄ 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


Ellagittanic or Ellagotannic Acid, known also as Ellagotannin, C₁₄H₁₀O₁₀ 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 Punica granatum (Ref 2) and fruits of Myrobalanus cebula (Ref 1). Richmers (Ref 1) patented the use of organic substances as ellagotannic acid as ingredients of expl mixtures, together with NH₄ClO₄ alone or mixed with KNO₃ or NH₄NO₃. These mixtures may be called Richmers' Explosives.

Refs: 1) Richmers, Brit P 10510 & CA 3, 839 (1909); 2) Hackh's Dict (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, decanting 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

Elvonol. 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 Coopal Powder (See Vol 3 of Encycl, p
C512-R), manuf in England ca 1889. It
contd NC, Ba nitrate & resins and was colored
green by malachite (oxalate of tetramethyl-
diamidotriphenyl carbino1)
Ref: Daniel (1902), 255 (Émeraude)

Emery Paper Figure (Brit). See EPF

Emilite. An explosive patented by Audouin
(EngP 5899 of 1887), which was prepd by
nitrating the fraction of coal tar boiling at
185–200°C 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 Spectro-
chemical 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 spectro-
ometer. 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 elec-
trodes are used. In the spark method, the
"simple spark", Feussner spark, Hasler
spark and Wolfe spark are used.
Spectrochemical analytical methods re-
cieved a tremendous impetus during WWII,
because of the time and labor saving in-
volved
(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. Böttger, "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. Loubovoux, "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
   Sheiman, "Emission Spectroscopy (31
   references) 9) N.S. Sventitskii, "Visual
   Methods of Emission Spectroscopy", Davy,
   NY (1965), 344pp (Translated from Russian)
11) Kirk & Othmer, 2nd edit (Not found)

Emmens Acid. A yellow substance prepared 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 114-115°, called in the book of Daniel l'acide Emmens. Then the mother liquor was partly evaporated and cooled. This yielded a 2nd crop of crystals 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 prep'd 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 prep'd by melting together 5 parts of yellow crystals of Emmens Acid, melting at 114-115°, with 5p of AN and 6p of PA. This expl was tested by the US WarDept and found to be suitable as a burster charge 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 prep'd 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 prep'd by mixing together PA, AN and concd sulfuric acid. It was a spongy, yellow, odorless mass of density 1.47
Ref's: 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_{12}O_{5}OH_{2}C_{6}(CH_{3})(NO_{2})_{2}2OH;

mw 450.23, N 12.45%; orm-colored crts (glac ac et or benz + petr eth); mp - destabilates 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-bm in aq Na_{2}CO_{3}; and is unstable in aq (2N) NaOH.

It can be prep'd by treating Emadin with fuming nitric acid (d 1.5) & concd sulfuric acid (Refs 2 & 3)
Ref's: 1) Beil 8, 520, (743), [563] & 141541
2) Beil 8, (743) & 141581
3) O.A. Oestereic & W. Sykens-Toxopæus, Archiv der Pharmazie 249, 314 (1911) & CA 5, 2818 (1911)

Empfindlichkeit (Ger). Sensitivity or Sensitive, 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 propiants: Gun cotton 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 heterogeneous 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 manufacture 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 acides); 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 "Anti-foaming or Antifrothing Agents").

In many cases foam may be prevented by some mechanical means, such as by properly designed stirrers, while in other cases chemical methods must be used. The earliest anti-foaming agent was ether and it found use in laboratory practice. Following are some other anti-foaming agents used in industry: vegetable oils (such as pine oil), animal oils, mineral oils, ocyl alcohol, fatty acids and their esters, alkyl lactates, organophosphates, 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")


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, Cl, or Al stearetes; 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 propylene), 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), alginites, starch and starch derivatives, casein, glue, egg albumin, methyl cellulose, hydrated Mg and Al silicates, etc.

Refs: Same as in previous item.

Emulsified NC and NC Explosive. slurries of NC (CP, 3 Bal) in emulsified NG were prep'd 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 (80-95°)
rolling temp) after 5, 10 & 20 days. Best sheet was obt'd at 30° & 0.5% emulsifier; denitration was less for all samples than for usual propint (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 prep'n 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 ("émulsionant", 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 propint, 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) prep'd 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 propelnt described in conf Propellant Manual SP/4/M2 (1969), Unit No 1071

"en". Designation of ethylenediamine as used in formulas for coordination compds, such as cobalt complex Colen(NO₃)₉
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
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
Ref: 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 Encyel, p C327

Endecacellulose Nitrate or Endecanitrocellulose. C₂₄H₄₉O₉(ONO₂)₉₁₉. 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-cyclooctane and Derivatives. 1,5-Endoethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane.

\[
\text{H}_2\text{C} - \text{N} - \text{CH}_2
\]

\[
\text{HN} \quad \text{CH}_2 \quad \text{NH}
\]

\[
\text{H}_2\text{C} - \text{N} - \text{CH}_2; \text{ a hypothetical substance which may be considered as the parent compd of its dinitro derivative.}
\]

1,5-Endoethylidene-3,7-dinitro-1,3,5,7-tetraaza-2,4,6,8-cyclooctane 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,

\[
\text{H}_2\text{C} - \text{N} - \text{CH}_2
\]

\[
\text{O}_2\text{N} \quad \text{AN} \quad \text{HC}, \text{CN} \quad \text{N} \quad \text{NO}_2
\]

\[
\text{H}_2\text{C} - \text{N} - \text{CH}_2; \text{ mw 232.20, N } 36.20\% ; \text{ crystals (from warm ethyl acetate); mp 175}^\circ \text{ with a brown residue remaining up to 245}^\circ ; \text{ sl sol in warm acetone and methyl ethyl ketone; insol in hot benz, ethanol, AcOH, chl and dioxane. For its prep Chute et al (Ref 2) cooled a soln of 0.124g of dimethylnitrarnide in 2.75ml of 20\% acetaldehyde by an ice bath, while, 0.36ml 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 cryt from ethyl acetate raised it to 175\%. Its expl props were not determined.}

Chute et al (Ref 2, p 235) treated Di-nitroendoethylidene with 99\% nitric acid in order to obtain impure HMX (called by them 1,3,5,7-Tetranitro-1,3,5,7-tetraazacyclooctane) 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, Can J Res 27B, 225, 234 & 235 (1949)

Endomethylene-tetraaza-cyclooctane and Derivatives. 1,5-Endomethylene-1,3,5,7-tetraaza-2,4,6,8-cyclooctane.

\[
\text{H}_2\text{C} - \text{N} - \text{CH}_2
\]

\[
\text{HN} \quad \text{CH}_2 \quad \text{NH}
\]

\[
\text{H}_2\text{C} - \text{N} - \text{CH}_2; \text{ a hypothetical substance which may be considered as a parent compd of its dinitro derivative}
\]
tetramine (DuPont's code name Unicel-100). Another name is Perform and our abbrm, DNsoPMT.

\[
\begin{align*}
\text{H}_2\text{C} & \quad \text{N} \quad \text{CH}_2 \quad \text{NO} \\
\text{ON} & \quad \text{CH}_2 & \quad \text{N} \\
\text{H}_2\text{C} & \quad \text{N} \quad \text{CH}_2; \text{ mw } 186.18, \text{ N } 45.14\%
\end{align*}
\]

solid; mp = expln on heating (204°-205°C). 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) obtb DNsoPMT by nitrosation of a similar, but more concd, formaldehyde-ammonia soln. Richmond et al (Ref 4) repeated the prepns and confirmed the experiments of Duden & Scharf and of Henry.

Later they obtb 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 (0.36 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) prpd the dinitroso deriv by nitrosation of HMTA (hexamethylenetetramine) by adding sulfuric acid, drop by drop, to an aq soln of HMTA and NaNO₂ at <10°.

In ADL Rept (Ref 7a) is described the method of prepn of dinitroso deriv based on description given by P. Aubertin in MP 33, 227(1951): Reaction of Na nitrite & hexamethylenetetramine in aq soln at pH 2.0. The effect of acids on lowering the decom temp of DNsoPMT was studied by Ikura & 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).


1.5-Endomethylene-3,7-dinitro-1,3,5,7-tetraaza-2,4,6,8-cyclooctene; 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-tetrazobicyclic[3.3.1]nonane. Code named DPT or DNPT, which is abbrm of Dinitropentamethylene-tetramine,
\[
\begin{align*}
\text{H}_2\text{C} & \quad \text{N} \quad \text{CH}_2 \\
\text{O}_2\text{N} & \quad \text{H}_2\text{C} \quad \text{N} \quad \text{NO}_2 \\
\text{H}_2\text{C} & \quad \text{N} \quad \text{CH}_2 \\
\end{align*}
\]

H₂C — N — CH₂
O₂N — CH₂ — N — NO₂

H₂C — N — CH₂, mw 218.18, N 38.52%, OB to CO₂ minus 80.7%; wh crystals (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, C₆H₁₂N₄) 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), C₆H₁₂N₄·2HNO₃, in 4 moles acetic anhydride gave a 30.5% 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 dimethylene nitramide 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 dimethyl nitramide and methylenediamine sulfate conducted as follows: The dimethyl nitramide was prepd from 2 moles of CH₂O and 1 mole of NO₂-NH₂ as described on p 68 of Ref 4.

The methylenediamine sulfate was prepd by method of Knudsen (Ref 2) from methylene-diformamide 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 dimethylene nitramide 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°C 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% CH₂O basis) and melted at 206° to 208° with decomp.

The reaction proceeded as outlined in Ref 7, p 223:

\[
\begin{align*}
\text{CH}_2\text{OH} & \quad \text{H} \quad \text{N} \quad \text{H} \quad \text{HOH}_2\text{C} \\
\text{O}_2\text{N} - \text{N} & \quad + \quad \text{CH}_2 \quad + \quad \text{N} - \text{NO}_2 \\
\text{CH}_2\text{OH} & \quad \text{H} \quad \text{N} \quad \text{H} \quad \text{HOH}_2\text{C} \\
\text{H} & \quad \text{H} \quad \text{H} \quad \text{H} \\
\text{H} & \quad \text{N} \quad \text{CH} \\
\text{O}_2\text{N} - \text{N} & \quad \text{CH}_2 \quad \text{N} - \text{NO}_2 \\
\text{H} & \quad \text{H} \quad \text{H} \\
\end{align*}
\]

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, O₂N-CH₂-N(NO₂)₂-CH₂-ONO₂, 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 decomp. Yield called 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.NH₃₃

\[
\text{HN.CH}_3\text{NH.CH}_2\text{NH.CH}_2\text{NH.CH}_3\text{ or CH}_2\text{N(NH)}_2\text{, by addg to 37% HCHO at 0° nitramide; after dilution with water or with soln to be tested, the mixt was adjusted to pH 7.0 with NH}_4\text{OH or dI HCl. The yield was 21%.}
\]

Attempts to prepare DPT by oxidation of Dinitrosodimethylenetetrazacyclooctane did not succeed (Ref 7, p 220)

DPT is a rather unstable powerful explosive, less sensitive to impact than TNT (Brockton 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- tetrazacyclooctane, mp up to 280°, with decomp.
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°C 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°C 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 1:2.5:2.7:1, respectively).

HACS & IR (Ref 10) described the prepn of DPT of mp 205–06°C from hexamethylene-tetramine dinitrate with $\text{Ac}_2\text{O}$ at RT for 3 days (yield 31%) or with 90% sulfuric acid at 15°C for 45 min; also as a by-product of manuf RDX from hexamine.

HMX, which is called Homocyclonitride 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°C. After addn of water, the DPT was collected on a filter & washed with water. Product had mp 190°C (dec).


Addn Refs for 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, ActaChemScand 23, 1741, 1742 (1951) (UV & visible absorption spectra in ethyl alcohol)
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)
N) M. Witanowski et al, JACS 86, 2570 (1964) (N14 nuclear magnetic resonance spectrum of DPT)
P) J.A. Bell & I. Dusman, JChemSoc, C, Org 1966(9), 867 & CA 64, 19615 (1966) (Isolation of DPT from reaction of 1-acetoxy-methyl 3,5-dinitro-1,3,5-triazacyclopentane by treating with Na Azide in dimethylformamide)
1,5-Endomethylene-3,7-dinitro-1,3,5,7-tetra-oxa-2,4,6,8-cyclooctane (DPT) as impurity in crude RDX. Its detection and estimation by chromatographic method is described in Ref: E.W. Malmborg et al, Anal.Chem 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 Mecanique 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)

**Energiet** and **Tri-Westfalus**, known also as Nitroglycerin Pulvern, were German mining expls prep'd from surplus double-base propellants of WWI.

According to Naoum (Ref, p449), the plants of the Nobel Co ground the different kinds of NG propel 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 Energiet. WASAG prep'd a similar expl, under the name of Tri-Westfalus SN, using in 1919 the following method: the NG propel 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 into a granular paste. However, it was found later to be more economical and simpler to crush the NG propel in rolls or edge runners to thin plates and then reduce this brittle form further. The product obtd gave a lead block expansion of 350 to 350cc. Potash mining has consumed the largest quantities of these expls. When unconfined Energiet and Tri-Westfalus 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 edge of Energiet and igniting by a fuse which caused expln of Energiet.

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 naphthalene 0–5, paraffin and/or urethane/carbanid 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) Naoum, 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, Pr 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 mv²/2, 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)


Energy Content of Explosives. Since the consequence of detonation is the formation of gases: CO₂, CO, H₂O, H₂, N₂ and oxides of N₂, the energy release can be increased by choosing the ideal OB to CO₂ (oxygen balance to CO₂) 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 combustion of an org compd, one must subtract the heat of formation of the compd from the heat of formation of H₂O and CO₂. 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 noted for its good expl performance, with a heat of expln 1392cal/g. In considering a mixture of fuel and a source of oxygen, it is interesting to note that TeNMe (Tetranitromethane) (first introduced in expl mixs 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 Acetylde -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 substances 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 caled 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 Naoum (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. Benthelon expressed it in 1871 as the product of the specific gas volume and heat of explosion, V₀xQₑ (See "Benthelon's Characteristic Product" in Vol 2 of Encycl, p B105). W.H. Rinkenbach (Ref 3) using recent data for Qₑ & V₀, 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. Marshal (Ref 1, p 469) designated the expression 425xQₑ 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 Qₑ, can be dted as described in Vol 4, p D377

Expressions QₑV₀ and 425Qₑ 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 "brissance" 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 Benthelon Characteristic Product and the time consumed by the expln. This period of time was to be caled from the experimentally detd vel of deton. However, this method of drg 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 = brissance, f =
specific energy, \( d = \text{density} \) and \( V = \text{deton vel.} \)

A detailed discussion of Kast's calcs is given in Vol 2 of Encycl, p B265, under BRISANCE OR SHATTERING EFFECT. Kast's formula (which he himself called, accdg to Naoum, 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-L & 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 D632f 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 (pp 473f) 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 Naoum (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 (and Explosion), p D198; Energy of Detonation (and Explosion), p D246; 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 D470;

Work Capacity in Detonation (and Explosion), p D730

Refs: 1) Marshall 2(1917), 469-73
2) Naoum, 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)


Energy Content of Propellant Charge.

Accdg to Stickland et al (Refs) the energy content was considered in German calcs 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 820 cal/g were 4.3 kg, a propellant with a value of 570 cal/g (such as "cool" Nitroguanidine proplnt, known as Guadupa) required a charge of 6.2 kg (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:

\[
\frac{c_F}{y-1} = \frac{P(u_g - c_h)}{y-1} + K
\]

where:
- \(c\) = mass of propellant burned, equal to mass of gas
- \(y\) = ratio of specific heats of the gas (assumed value 1.30)
- \(P\) = space average pressure in lb/sq inch
- \(u_g\) = vol of propellant gas in cubic inches per lb
- \(\gamma\) = specific heat 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 propellant (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>
<thead>
<tr>
<th>Table E12</th>
<th>(W)</th>
<th>1.5pv</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blasting Gelatin</td>
<td>694</td>
<td>158</td>
</tr>
<tr>
<td>Dynamit No 1</td>
<td>497</td>
<td>108</td>
</tr>
<tr>
<td>Carbonit I</td>
<td>255</td>
<td>73</td>
</tr>
<tr>
<td>Black Powder</td>
<td>244</td>
<td>42</td>
</tr>
<tr>
<td>Ref: Marshall 2(1917), 469</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

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. Glosch, Zeitschrift für Elektrochemie, 57, 632-36 (1953), Pica-tinny 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):

\[
\frac{(W+C/2)^2 \cdot v^2}{2gR}
\]

where:
- \(W\) = wt of projectile
- \(C\) = wt of propnl chge
- \(V\) = vel of projectile in inch/sec
Energy Relationships in Detonation (and Explosion). See Vol 4 of Encycl, p D266

Energy (Relative) Release During Susan Test of 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 63829, 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 produce 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 x 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, ‘‘Elumens of Ammunition 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. Acc to Muraour & Aunis (Ref) the energy Q, in kcal liberate at constant volume by decomp of 1kg of prop- "nit (or explosive) is shown graphically to be linearly related to the temperature, in degrees K, developed on deflagration (o explosion) of propints between 2000 and 4000°K. The values of the velocity of deflagration can be obtd from the equation:

$$\log V = 1.47 + 0.845 (T/1000)$$

They agree with those calcd from equation

$$V = 1.37 + 0.27 (T/1000)$$

The propints 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, 282 (1941)

Addnl Refs: A) H. Muraour, Chimie et Industrie 50, 105-09; 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, 5180 (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", the weight 2 lbs 15 ozs, and the range of up to 2000 yds (Refs)


Engels patented at the end of the 19th. century the following expl compn: NG 60-70, “pyropapier” (See Note) 15-18, NH4 salts (nitrate, sulfate or chloride) 10-30, saltpeter 8-10, Pyroyline 5-10, Nitromannite 1.5, Nitrostarch 0.5, Nitrobenzene 0.5 & water-glass 0.5%

Ref: Daniel (1902), 268

Note: ‘‘Nitropapier’’ or Papier fulminant was prepared by plunging for 2 mins sheets of paper into a 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-propelled motor. It is also used for a rocket launcher, such as "Bazooka"

Engin criminal. 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 Höllenmaschinen

Engin à fumée chaude de Berger (Fr). A smoke-producing pyrotechnic device invented by Berger and used during WWI. It was operated by the combustion of mixtures such as CCl₄ 40, ZnO 20, powdered Zn 15, Ca silicide 15 & Na perchlorate 10%. Several models existed Ref: Pepin Lehauller (1935), p 470

Engin à fumée froide de Verdier (Fr). A smoke-producing device invented by Verdier and used during WWII. It operated by ejecting a mixture of stannic chloride and ammonia into a humid atmosphere. Several models existed Ref: Pepin Lehauller (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 turbo-jet engine


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-1r, 0.0013" in diam
with a DC resistance of 1 ohm. The initiating charge 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 vel 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 riffling
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 (Riffling); p 31 (Ban d.
rotating)

Enkeptic. See 2-Amino-5-nitrothiazole in
Vol I, p A263-R

ENIAC (Electronic Numerical Integrator
and Computer). The first electronic dig-

ital 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 chloride
80, MNT 15 & castor oil 5%. Used during
WWII as a bursting chge, probably for gren-
ades and mortar shells. (Compare with
Entoyaku)
Refs: 1) OpNav 30-3M (1945), p 28
2) R. P. Antonellii "Encyclopedia of
Explosives", Ordn Liaison Group-Durham
NC (1960), p 54

Enneanitrocellulose or Enneocellulose
Nitrate, C_{44}H_{80}O_{44} (ONO_2)_{a}. See Ennean-
i trate in Table under CELLULOSE NITRA-
TES OR NITROCELLULOSES on p C100-R
of Encycl, Vol 2

Enneaheptite, Anhydro-Pentanitrate, called
in CA 2,2,4,4-Tetakis (hydroxymethylnitrate)1-pyranol. See under Anhydroeneaheptitol
and Derivatives in Vol 1 of Encycl, p A404

Enols are weakly acid compds of the general
formula:
\[ R\overset{\text{C}}{\text{C}}=\text{C}-\overset{\text{O}}{\text{H}} \]

which are tautomeric forms of a substance of the
general type
\[ R\overset{\text{C}}{\text{CH}}-\overset{\text{O}}{\text{C}} = \text{O} \text{ (keto form)} \]

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 Dictionary", NY (1953), 265-L
2) Std Methods Chem Analysis 2A (1963), pp 455-59 & 461-62

**Entoc** (Engin téleguidé 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)

**Entoc.** (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

**Entflammungsprobe or Entflammungstemperatur 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) died at PicArsn "Enthalpy Change, Heat of Fusion and Specific Heat of Basic Explosives"
2) R. Velicky et al, PATR 2504, Jan 1959(U); Project TA3-5000B, Item C. Project 5A04-10-906

**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
Entoyaku. A Japanese explosive of light brown color used during WWII as a substitute demolition charge or as a bursting charge in hand grenades and mortar shells. Its composition was: KClO₃ 80, MNT 16 & castor oil 4% (Compare with Ennayaku)

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 calculations, but rather in the form of its increments or changes. Entropy is arrived at in thermodynamics in the form of the concept 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:

\[ \Delta S = \Sigma \frac{q}{T} \]

where \( \Delta S \) is the change in entropy and \( \Sigma \frac{q}{T} \) is the sum of the terms obtd by dividing the infinitesimal amounts 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

Entzündungsgemisch. Ger for Ignition (or Igniter) Mixture

Entzündungsprobe. Ger for Ignition Test

Entzündungs punkt or Verpuftungstemperatur. Ger for Ignition Point or Deflagration Temperature

Entzündungstemperatur. See Entzündungs punkt

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 gallery tests), by a mantle of an inert material, such as Ca fluoride (See Ref 1). This envelope is known in Belgium and France as goine and expls enclosed in it explosifs gainés to distinguish them from the explosifs non gainés (Vol 3 of Encyc, 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 Encyc, 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 Roundel, 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 (Ref s 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 inert sheaths have been Ca fluoride and Ca sulfate or Na bicarbonate. These expls found wide use in sulfur mines of Sicily.

Under the term *Vzryuchatiye veschestva v predokhranitel'ny 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 cortezza 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, NaN3, NaCl, Ca sulfate) in expl itself. Such expls are known as EqS (qv).


**Enzion 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 m, alt 9 vel 600 mph. Solid propellant.


**Enzion Rakete.** Ger Guided Rocket of WWII. See under Guided Missiles

**EODT** (US Army). Abbn 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**

*a-Epichlorohydrin (epi) Chloropropylene Oxide*, or 1-Chloro-2,3-epoxypropane, ClCH2-CH2-O, mw 92.51; liquid, less volatile than alc., eth., or acet., with chlf-like odor; fr p 528.15, bp 115.20, sp gr 1.1761 at 20°/20°, vapor pressure 12.5 mm at 20°.
viscosity 1.12 cps at 20°, n_D 1.4358 at 25°; miscible with alcohols, ethers and most other organic solvents; immiscible with water and petroleum hydrocarbons. It can be prepared 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 explosives. 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 nose, 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 (Rejs: 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-oxo-1,2-propanediol Trinitrate.

\[ \text{CH}_2-\text{CH(OONO)}_2-\text{CH}_2\text{ONO}_2 \]

mw 271.14, N 15.50%, by reacting epichlorohydrin & glycol and nitrating the product with mixed acid (HNO₃ 48.8, H₂SO₄ 48.6 & H₂O 2.6%) at 20° for 20 mins. The Trinitrate has d 1.4839 at 15°, n_D 1.4690 at 20°, heat of combustion 2405 cal/g at const vol & corrected to 18°(Ref 2), and its volatility at 80° is between Ethyleneimine 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)


Epichlorohydrin Perchlorate (called α-Überchlorsäureester des-y-chlor-propylenglykol in Ger), Cl₂CH₂CH(OH).CH₂ClO₄, mw 192.99; viscous sl fragrant oil, deflgd 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 prepared by reaction of α-epichlorohydrin in ether with 60% perchloric acid at 0−5° (Rejs: 1) Beil 1, 474 2) K.A. Hofmann et al, Ber 42, 4390 (1909) & CA 4, 584 (1910) 3) Urbanński 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'-isoproplydiphenol, (CH₃)₂C(C₆H₄OH)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 componds & solvent free coatings. Lenchitz et al (Ref 2) determined the heat of polymerization of Epon 828 and of its mix of ethylenediamine (EDA), diethylenetriamine (DTA) & triethylenetetramine (TETA)

Refs: 1) CondChemDict (1961), 156-6 & 444

Épuisottage (French for Dust Removal). The removal of dusty material from the grains of Bk,Al or smokeless propellants was effected in France by shaking the grains on a fine screen

Ref: Pepin Lehalleur (1935), 284

Epoxides are comnds incorporating a three-membered ring containing one oxygen atom and two carbon atoms, \( \text{O} \)

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; methanalysis 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


Epoxides. A name sometimes given to Epoxy Resins (qv)

Epoxyl. A prefix in organic nomenclature denoting an oxygen atom joined to each of two atoms which are already united in some other way, as \( \text{O} \)

\( \text{H}_2\text{C} - \text{CH}_2\text{epoxyethane and} \)

\( \text{CH}_2\text{CH}_2\text{epoxypropane} \)

Refs: 1) Hackh's Dict (1944), 310-L
2) CondChemDict (1961), 445-L

2,3-Epoxy-2-ethylhexanol,
\( \text{C}_3\text{H}_7\text{CHO(C}_2\text{H}_5\text{)CH}_2\text{OH}, \text{mw} 144.21, \text{liquid, sp gr} 0.9517 \text{at} 20/20^\circ, \text{fr p} -65^\circ, \text{bp} - \text{decomposes, sl sol in w. Its method of prep was not found in the literature}

Used as a stabilizer and as intermediate compd.

Ref: 1) Beil, not found 2) CondChemDict (1961), 445-L

2,3-Epoxy-1-propanol or Glycidol,
\( \text{CH}_3\text{OCHCH}_2\text{OH}, \text{mw} 74.08, \text{colorless liq, bp} 162^\circ, \text{sol in w, alc or eth. It can be obt by treatment of monochlorohydri} \text{n with bases and by other methods (Ref 1)}

Used as stabilizer for natural oils and vinyl polymers and also as demulsifier (Ref 2)

Ref: 1) Beil 17, 104, (50) & (104) 2) CondChemDict (1961), 542-R (Glycidol)

Epoxy Resins or Epoxides. The commercially available materials of this class are usually derived from epichlorohydrin and biphenol 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,
\[ H_2C=CHCH_2OC(CHOHCHOHCH_2)\_nROCH\_C=CHCHOH_2, \]
where R = C_6H_5C(CH_3)_3C_6H_5. 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 diphens or 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 \(-C\_O\_C\_O-\), or are derived from such compds. Thus "Carboxax" (which is trademark of Union Carbide Corp, New York 10017, NY for polyethylene glycols or methoxyethyl glycols, des-cribed 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 for 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).


Épreuve ou Essai. French for Test, Experiment, Assaying or Essay

Note: For reasons unknown to us, many tests, known generally as Épreuves 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 "Eihitzungsprüfung 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’utilisation 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 obd 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 150 cc 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 à l’inflammation. See Test 6 under Essais d’Explosifs

Épreuve a la dynamite de pilotes (Fr for the Test of Dynamite by Means of Piles). The test, first conducted at Budapest in 1861 by Lt Colonel Prodanovic, 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 exp1 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 resr 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 à 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 "Exsudation (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 totalisée (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 D399-R
c) Combustion en gouttière de 20 mm (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 Grotianni 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 Violette ou Épreuve dite a 110° described in Ref 1, pp 392–93, in Ref 4, p 107 and Ref 5, p 78
k) Épreuve Violette 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

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: Unirex, Unigex (Initial), Unigel, Unibel, Unifrax and Unikol are given in Refs
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:
B) P. Caldirona, 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 Caldirona'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 caled by various equations is presented for RDX, PETN, TNT, Tetrol, PA, AmmP, LA & MF and correlated with similar data obtd by other investigators)

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

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 equivalent power as measured by peak pressure or positive impulse. The EV is similarly defined.

Two independent means, graphical & relative pressure methods, are used to obt EW & EV from peak pressure measurements. Two comparable methods obt 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)

**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 Mt K.F. Kempt

**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/gravit acceleration in cm per sec per sec, which gives 0.00102g. One joule is equal to 10⁷ ergs.

Ref: Hack'hs Dict (1944), 291-R (Dyne); 312-L (Erg)

**Ehordt (Poudres).** Three formulations are known: 1) Artillery – KClO₃ 33.33, KNO₃ 33.33 & tannin or catechu 33.34% 2) Bombs – KNO₃ 50 & tannin or catechu 50% 3) Mining – KClO₃ 12.5, KNO₃ 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, Bruce ton, 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%) + Lechin (5%)] 12%. Its properties resembled the British PE No 2, but possessed improved low temperature behavior.


Ref: Hackh’s Dict (1944), 313-L

**EROSION OF GUN BARRELS** [Erosion (ou Usure) des bouches à feu, in Fr; Erosion der Schusswaffen; Bohrabnutzung, oder Bohraus- brennung der Gewehräufe, in Ger; Erosiya i Razgar Stvolya 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 propint, 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 propellants (which took place in the middle of 1860's) more rapid wear away of gun barrels became noticeable. The damage was the smallest with propellants based on NC (See Single Base Cannon Propellant in Vol 2, pp C31-R to C32-L), than with NG-contg propellants (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 expls of NGs (Nitroglycerine) 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 expls 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 expl 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 expl of NG was 2.3mm, Poudre B 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 Paqueline 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 Moraud of France (Refs 15 & 16), Lintés of Rumania (Ref 18), Schwinning of Germany (Ref 20), Podubayi of Russia (Ref 21), Hayes of the USA (Ref 22), Costier and Sutterlin of France (Refs 24 & 25), Corner of the USA (Refs 27 & 35), Alvaro-Alberto of Brazil (Ref 28), Gen Galliwitz of Germany (Refs 29 & 30), Daubrée of France and R. Austen of the USA (quoted from Pérez Ara, Ref 31), Fichard & Kovache of France (Ref 34), Green of the USA (Ref 36), Abram et al. of GB (Ref 37), Tavemier 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 propellt 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 propellant 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 between surface of the driving band and lands of
the bore becomes greater and greater, more
gases escape thru the muzzle without exert-
ing any pressure on the projectile. This
causes reduction of pressure in the chamber, as well as of muzzle velocity of the projec-
tile. 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. Chemov (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 re-
sulting 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 con-
ists 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 cong NG, in
addition to NC, cause more excessive damage
than those based only on NC, even if the
calorific values of propellants are the same.
Propellants cong colloidal NC cause less
damage than those cong the same amt of non-
colloidied NC (Ref 43, p 153).

Experimental procedure for det 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 Andriev & 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 re-
sults 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
is diminished by a device, such as
"cardboard obturator", preventing the es-
cape of gases between the rifling of the bore
and lands of projectile (Ref 43, p 154). One
of such devices was the so-called prissal'nik,
which can be defined as "greaser" or "lu-
bricator", 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
abbrev translation from the books of Blinov
(Ref 31b), prissal'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, cerasin & 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 prissal'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
appointably lower due to the presence of
carbonaceous products of decom 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 propblt 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 propellants of high ballistic potential, such as those contg NG. As these propellants were "hot" (calorific value about 950 kcal/kg) they caused excessive erosion, thus lowering considerably the life of the gun. For example, the life of AA guns using a 950 kcal/kg proplnt was only 1700 firings, while for a 820 kcal/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 propellant charge, this leads to a still brighter flash. In order to reduce the flash, more and more "flash reducing agent" (such as K₂SO₄) had to be incorporated and, as these agents are inert materials, they diminished the ballistic potential of propellants. 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 Udo Gallwitz and also the Laboratories of Dynamit AG started, as early as 1934, to investigate the problem of development of
proplints of low calorific value but of high ballistic potential comparable to NG proplints. As result of their work, a series of low calorific value proplints (known as "cool" proplints) were developed, such as "G" (Gallwitz) Pulver by Gen. Gallwitz. It contd DEGDN, NC, EtCentralite, K₂SO₄, MgO and graphite. It was called "K" Pulver by the Ger Air Force. This proplint was very suitable for high caliber guns, especially howitzers and mortars, but unfortunately, when fired from the same weapons as NG proplints, 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 proplints produce less erosion than NG proplints of the same ballistic potential was not valid.

"G" Pulver was, however, unsuitable for medium howitzers and mortars due to the low speed of combustion as compared to NG and straight NC proplints. In order to eliminate this drawback, the Dynamit AG suggested incorporation of NGu (Nitroguanidine). The resulting proplint, known as Gudolphulver, was very satisfactory in field howitzers from the point of view of ballistics, as well as because it produced only low muzzle flash without adding K₂SO₄ or other flash-suppressing agent and also low erosion and corrosion (Ref 29, 30 & 44).

Cool proplints developed in the USA during and after WWII contd large amounts of NG in addn to NC and "explosive oils" (NG, DEGDN, TEGDN, or Mettrol Trinitrate). A brief description of these proplints known as Triple-Bose Cannon Propellants is given in Vol 2 of Encycl, p C37-R, while compns of proplints M15, M17, T20, T29, T34 and T39 are given in Table V on p C34.

Several cool proplints were developed after WWII in Great Britain. They contd NCu (Pierite) in addn to NG (or DEGDN) and NC. Their compns are given in ARE Rept of 1953 (Ref 37). Erosive action of these proplints was tested in 17-pounder gun barrels made of Ni-Cr-Mo steel and results reported in Ref 37.

Several cool proplints were developed in Belgium after WWII. They were patented by the Recherches Chimiques SA, BelgP 565271 (1958), listed in CA 53, 10765 (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 phénomènes d’érosion)
2) P. Siwy, SS 3, 42 (1908) & CA 2, 2832 (1908) (Causes of erosion in guns)
3) E. Bravetta, SS 4, 361-65 (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. Leclercq, MAF 1, 1013-35 (1922) (Sur l’usure des bouches à feu)
7) P. Paquier, MAF 1, 1037-40 (1922) (L’usure, l’encairage, l’acier et le baguage)
8) I.A. Krylov, “Izucheniy Razgoraniya Kazalov Ogneupotrebnogo Oruzhiya” (Study of Barrel Erosion in Firearms), Gosizdat, Moscow (1927)
9) E. Piantanida, Rivista Marittima (October 1922) (Sulle alterazioni subite dall’acciaio di tubi animza 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, 3e fascicule (1929) (Usure des bouches à feu)
12) D. McNair, SS 25, 196 (1930) (Die Abnutzung von Geschützrohren)
13) G. Chekhov (G. Chekhov), MAF 10, 3e fascicule (1931); 11, 2e fasc (1932) and 12, 1er fasc (1933) (Usure des bouches à feu)
16) H. Muratour, MAF 12, 441-47 (1933) (Note sur le pouvoir érosif des poudres B additionnées de divers produits destinés à abaisser la température de combustion)
17) A. Rege, Rivista Marittima (1935) (Sull’usura dei cannoni)
18) J. Lintés, MAF 14, 175-223 (1935) (Étude critique sur l’usure des bouches à feu) [Translated from Rumanian Revista Artilleriei 1933, p 327 (May) and 394 (June)]
19a) Pepin Lefalleur (1935), 102-19 E.

Six 17-pounder gun barrels were used for comparative wear trials with propellants of different comps. 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" propellants 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 amr of erosion produced by propellants of FT = 2800°K. The results also showed that Picrite propellants (Picrite is Brit designation of Nitroglycerine, 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 "crack-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 propellants.

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 propellant gases and not to mechanical wear by the driving band (which was of copper). This bore enlargement in the three barrels which fired "cool" propellants (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 propellants (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 propellant 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 propellants showed no measurable increase in wear, whereas a small but definite amount of bore enlargement occurred in the three barrels which fired the 1950°K propellants.

In some guns which have fired std Service propellants and Cu-banded proj, 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 propellant 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 propellant 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 proj, the major factor responsible for wear of C of R region was the erosive action of the propellant 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 propellant gases overshadowed any wearing effect of the Cu band. Erosion by propellant 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 propellant 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 amount 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 propellant gases escaping past it during free-run travel.

As plastic bands would not suffer such drastic softening by escaping propellant gases
as Cu bands, it was reasonable to surmise that plastic-banded proj's 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 proj's in worn 6-pr, 6-cwt and QF 5.25 inch guns.

Crack-cracking was more prolific and persisted further along the bore in the three barrels which had fired Picrite proj's 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 bars than in the other barrels, a fact which would appear to be related to heating of the bore surface by propellant 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 propellants of FT = 1950°F were different in that one (F487/68) contd 13.4% 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); 38(a) J.E. Capell, PATR 2033 (1954) and private communication (1954)
38(b) B.T. Fedoroff et al, PATR 2145 (1955), p 409
38(c) 19 (Inoue's gasket for reduction of erosion in guns, known as "propalnik")
39) P. Tavenier, P. Prache & J. Berger, MP 37, 207-15 (1955) (Contribution à l'étude de l'érosion des poudres colloidales) (Comparison of erosion produced by Poudre SD (temp of expln at const vol 251°F), Amer prop pants with JPN (temp of expln 3886°F) and Ger froide (cool) propellant (temp of expln 1880°F) showed that poudre SD is particularly sensitive to the phenomenon of erosion. Unfortunately no compn of any of the above propents is given)
40) Anon, "Cannon Tube Erosion and Service Life Tests", OP Manual 70-50 (1956) (See Ref 32)
42) J. Boisson, MP 39, 381-96 (1957) & CA 53,

<table>
<thead>
<tr>
<th>Constituents</th>
<th>F488/312</th>
<th>F551/66</th>
<th>Cordite N</th>
<th>F487/68</th>
<th>F428/180</th>
<th>F487/46</th>
</tr>
</thead>
<tbody>
<tr>
<td>Picrite (NGu)</td>
<td>Nil</td>
<td>55.0</td>
<td>54.7</td>
<td>Nil</td>
<td>60.0</td>
<td></td>
</tr>
<tr>
<td>NC</td>
<td>56.5</td>
<td>21.0</td>
<td>19.0</td>
<td>70.0</td>
<td>65.0</td>
<td>20.0</td>
</tr>
<tr>
<td>NG</td>
<td>33.0</td>
<td>21.0</td>
<td>18.7</td>
<td>Nil</td>
<td>15.4</td>
<td>Nil</td>
</tr>
<tr>
<td>DEGDN</td>
<td>Nil</td>
<td>Nil</td>
<td>Nil</td>
<td>14.3</td>
<td>Nil</td>
<td>11.16</td>
</tr>
<tr>
<td>Carbamite (Ethylcentralite)</td>
<td>3.5</td>
<td>3.0</td>
<td>7.3</td>
<td>2.0</td>
<td>2.0</td>
<td>2.64</td>
</tr>
<tr>
<td>DBuPh</td>
<td>7.0</td>
<td>Nil</td>
<td>Nil</td>
<td>13.7</td>
<td>17.6</td>
<td>6.2</td>
</tr>
<tr>
<td>Na Cryolite</td>
<td>Nil</td>
<td>Nil</td>
<td>0.3</td>
<td>Nil</td>
<td>Nil</td>
<td>Nil</td>
</tr>
<tr>
<td>Adiabatic Flame Temperature, °K</td>
<td>2800</td>
<td>2800</td>
<td>2450</td>
<td>1950</td>
<td>1950</td>
<td>1950</td>
</tr>
<tr>
<td>Gas Composition (% by Volume) at T₀ °K</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CO₂</td>
<td>7.6</td>
<td>5.2</td>
<td>3.5</td>
<td>3.4</td>
<td>3.1</td>
<td>2.5</td>
</tr>
<tr>
<td>CO</td>
<td>45.4</td>
<td>27.7</td>
<td>31.3</td>
<td>51.4</td>
<td>52.3</td>
<td>32.3</td>
</tr>
<tr>
<td>H₂</td>
<td>16.8</td>
<td>16.6</td>
<td>22.6</td>
<td>28.6</td>
<td>28.9</td>
<td>29.2</td>
</tr>
<tr>
<td>H₂O</td>
<td>19.2</td>
<td>21.5</td>
<td>15.1</td>
<td>8.3</td>
<td>7.5</td>
<td>10.0</td>
</tr>
<tr>
<td>N₂</td>
<td>11.0</td>
<td>29.0</td>
<td>27.5</td>
<td>8.3</td>
<td>8.3</td>
<td>26.0</td>
</tr>
<tr>
<td>7-pr Barrel No.</td>
<td>Designation</td>
<td>Type</td>
<td>Flame Temperature</td>
<td>Charge Weight</td>
<td>Prior to Wear Trial</td>
<td>Total</td>
</tr>
<tr>
<td>----------------</td>
<td>-------------</td>
<td>-----------</td>
<td>-------------------</td>
<td>---------------</td>
<td>--------------------</td>
<td>-------</td>
</tr>
<tr>
<td>38130</td>
<td>F488/312/S</td>
<td>Non-picroite</td>
<td>2800°C</td>
<td>5 14 0</td>
<td>77</td>
<td>477</td>
</tr>
<tr>
<td>16113</td>
<td>F551/66/S</td>
<td>Picrite</td>
<td>2800</td>
<td>5 15 4</td>
<td>79</td>
<td>579</td>
</tr>
<tr>
<td>28013</td>
<td>Cordite N/S</td>
<td>Picrite</td>
<td>2450</td>
<td>6 5 0</td>
<td>75</td>
<td>575</td>
</tr>
<tr>
<td>10147</td>
<td>F487/68/T</td>
<td>Non-picroite (+DEGDN)</td>
<td>1950</td>
<td>7 4 12</td>
<td>78</td>
<td>578</td>
</tr>
<tr>
<td>16600</td>
<td>F428/180/T</td>
<td>Non-picroite (+NG)</td>
<td>1950</td>
<td>7 0 8</td>
<td>78</td>
<td>578</td>
</tr>
<tr>
<td>26809</td>
<td>F487/46/S</td>
<td>Picrite (+DEGDN)</td>
<td>1950</td>
<td>8 4 0</td>
<td>65</td>
<td>565</td>
</tr>
</tbody>
</table>

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, "Poroka i Vzvyvchatiye Veshchestva" (Propellants and Explosive Substances), GosizdatOboronProm, Moscow (1957), 152-55 (Razgar stvola oziydra i eroziyra) (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. Andreiev & A.F. Beljaev, "Teoriya Vzvyvchatiyk Veshchestvy" (Theory of Explosive Substances), Oborongiz, 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. Mochamukov, VoprosPoshchikovoiMet i ProchastiMaterialovAkadNaukUSSR 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 combat 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. Sirovryuk, AkustZh 12(1), 87-92 (1966) & CA 65, 417 (1966) (Effect of temp & gas content of a liq on cavitation processes) 50) Gorst(1972), 169-73

**Erosionless Priming (Erosionsfreie Zündung)**
Any priming composition contg MF (Mercuric Fulminate) with or without a chloride (usually KClO₄) 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(NO₃)₂. These compns were named "rostfreien 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 manud before and during WWII by the Rheinisch-Westfälische Sprengstoffe AG, Nürnberg (Refs 1, 2, 3 & 5). Ficherolle & Kovache (Ref 4) reported that erosion of sporting firearms caused by priming mixtures was smaller when using LSr (Lead Stephnate) Tetracene mixtures, than compositions contg MF
E 121


Erosion of Ground; Control by Explosives. See item h, p A113-R in Vol I 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) Naoum, 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 Amatol
b) Substitution of DNB for TNT in Amatols
c) Substitution for TNT, by nitraminas such as Dinitrodiphenylamine, Hexanitrodiphenylamine, Trinitroxyllene, Dinitronaphthalene, etc
d) The use of low melting hydrous inorganic nitrate compounds, such as Ca, K and NaNitrates, to permit 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, Pentaerythritol, Guanidine Nitrate, Ethylenediamine dinitrate, Methyldinitrate, 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 NH₄NO₃, 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/NaNO₃ (Sodatol) and 45/40/15 TNT/NaNO₃/Al had been used to a considerable extent.

Table E15 lists the principal "substitute explosives" used by the Germans during WWII.
<table>
<thead>
<tr>
<th>Components</th>
<th>Amatol</th>
<th>Ammonic</th>
<th>HEXO</th>
<th>HEXA</th>
<th>KMA</th>
<th>NaCl</th>
<th>TNX</th>
<th>Explosive</th>
</tr>
</thead>
<tbody>
<tr>
<td>TNT</td>
<td>50</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>40-50</td>
</tr>
<tr>
<td>Amm Nitrate</td>
<td>40-45</td>
<td>35</td>
<td>40</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td>45</td>
<td>55</td>
</tr>
<tr>
<td>Na Nitrate</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>9</td>
<td>9</td>
</tr>
<tr>
<td>K Nitrate</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>25</td>
<td>15</td>
</tr>
<tr>
<td>Ca(NO₃)₂.4H₂O</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>10</td>
<td>10</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>RDX</td>
<td>5-10</td>
<td>15</td>
<td>10</td>
<td>25</td>
<td>25</td>
<td>20</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td>PH-Salz</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>10</td>
<td>14</td>
</tr>
<tr>
<td>Urea</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Al Powder</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1.8</td>
<td>1.8</td>
<td>2</td>
</tr>
<tr>
<td>HNDPhA</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>DNN</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>30</td>
</tr>
<tr>
<td>DNB</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>10</td>
</tr>
<tr>
<td>PETN</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>GuN</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>10</td>
</tr>
<tr>
<td>DNDPhA</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>15</td>
</tr>
<tr>
<td>Na Chloride</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>TNX</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

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 Trinitroxyline.

To this table may be added the following:

a) An explosive composition prep by I.G. Farbenindustrie by nitrating a mixture of MNX, methylaniline and MNT. The nitrated product consisted of TNX 45%, Tetryl 50% and TNT 5%. 

b) An explosive mixture of the Krümmel plant of D A-G contained TNT 45%, Ammonium nitrate 40% and Al powder 15%. It was suitable for cast-loading bombs, grenades and land mines.

c) An explosive mixture of the Christianstad plant of D A-G was a slurry of 70% Ca(NO₃)₂.4H₂O 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, Tetranitrosticosterone (TeNMe explosives), Tetramethyl-nitraminetetramethylmethane, 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.

Eruptite. A chlorate based explosive manufld 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_2(NO)_2CH.CH_2O-CH_2CH.ONO_2, mw 194.10, N14.43%. Its props & methods of prodc 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 (F1 103%PA)
Refs: 1) Beil (not found) 2) AC-956/0rg EX 74(1941) 3) Blatt,OSRD 2014 (1944)
(under Nitrates) 4) Not found in CA

Erythene or Vinyl-thylene. See Butadiene in Vol 2 of Encyc. 1365-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): L-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 20/4º, mp 120º, bp 329-31º, sol in w(61% at RT), s1 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 obtb by us from Dr Jean P. Piccard (Ref 3) Er can be prepd from 1.4 butadiene, CH_2:CH.CH:CH_2 which, in turn, can be obtb in 70% yield, by modified Russian Process from alcohol and acetaldehyde:
C_2H_5OH+CH_3COH→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 Br_2 BrCH_2.CH:CH:CH_2Br
Na_2CO_3, KMnO_4 BrCH_2.CH(OH)CH(OH)CH_2Br
KOH /O /O 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 prep of erythritol (by Repppe), starting materials are acetylene and formaldehyde and reactions can proceed as follows:
CH:CH+CH_2O→HO.CH_2:CH.OH → Butene - 2-diol-1,4
HO.CH_2:CH.CH_2:OH → HO.CH_2:CH.CH_2:OH Cl Cl
HO.CH_2:CH.CH_2:OH NaOH

Mesoerythritol

Note: It has been claimed by the GerP 7340 25 of IG Farben-Industrie, CA 38, 981 (1944) that a soln of butene-2-diol-1,4 in an 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) & l23561 2) Daniel (1902), 480 (under Nitærhythrite)
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)

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 84.5-85º; bp explodes

Was prepd by Fichter (Ref 2) on stirring for 20 mins, 10g of erythritol with 50g of mixed nitric-sulfuric acid (1:2), previously
Erythritol Tetranitrate (ErTeN), Nitro-1,2,3,4-Butanetetrol Tetranitrate
(called by Stenhouse Salpetersauerer Erythronannit) (O₂NO)₂CH₂(CHOONO₂)₂CH₂(ONO₂)
mw 302.12, N18.55%, OB to CO +26.5%, OB to CO₂+ 5.3%; col plates (from alc), sp gr 1.59,
mp 61°C, 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 prep'd in 1849 by Stenhouse (Refs 1 & 2) by dissolving with cooling, erythritol in
fuming nitric acid and then precipitating the resulting ErTeN by the addn of concd sulphic
acid. Bergheim (Ref 7) patented a method of prep with better yield in which erythritol
(1 part) was dissolved (at 40-50°C) in 5.5 parts sulfuric acid and the resulting soln was added
to 7pts 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 (NH₄ or Na carbonate) and diluted with a large amt of cold water to reprecipi-
tate ErTeN

Accdg to Naoum (Ref 6, p 238), explosion of ErTeN proceeds as follows:
C₄H₆(NO₂)₄ = 4CO₂ + 3H₂O + 2N₂ + 0.50₂

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°C (205°F) vs 222°C for
NG in 5 sec
Friction sensitivity - detonates
Gas volume at NT 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 Cv, vs 1486
for NG
Heat of formation 376 cal/g at Cv, vs 400 for NG
Impact sensitivity, ButMines 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 10⁴ 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 medi-
cine against chest angina (l'angine de pointrine,
in Fr). For this purpose finely pulverized ErTeN
was triturated in a mortar with powdered lactose or
sugar which was a dangerous operation resulting in
some cases in violent exps. One of such exps,
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 patent
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 prep'n than the original Stenhouse method of
1849, proposed to use ErTeN as a chief sensiti-
sing expl constituent (Ref 8). Vest & Kuhn (Ref
15) of BRL prep'd some solvent double-base-propel-
lets contg ErTeN in lieu of NG and observed slight
"blowing" on the surface of such proplnts. It
was suggested to try the prep'n of "solventless"
proplnts contg ErTeN in order to avoid blooming
(Ref 15)

Accdg to the unpublished write-up obtd by us
from Dr Picard (Ref 13), ErTeN could be consid-
ered 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 (lichenes) used for prep'n of erythritol.
The problem of scarcity seems to be now eliminat-
ed after development of synthetic methods of
prep'n of erythritol from butadiene and acetylene
outlined here under Erythritol. This, however,
did not reduce the cost of ErTeN to such an ex-
tent 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) (Prep'n
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 phar-
macerist lost his life while engaged in triturating ErTeN with lacrosse powder in a mortar. 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) Nauem, NG (1928), 237-38 (Prep and some props of ErTeN) 7) F.H. Bergeim, USP 1691994 (1928) & CA 23, 708 (1929) (Method of prep 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 Tetratinotrioxytita) 13) Dr Jean P. Picard of PicArsn; private communication (about 1950) (Some props and uses of ErTeN; synth methods of prep 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. Vegst & L. P. Kuhn, BRL (Ballistics Research Laboratory) Tech Note 757, (Nov 1952) (Investigation of propnits 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 propnits 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 NC) 17) J. Büchi & R. Alther, PharmActaHelv 31 121 (1956) & CA 50, 13368 (1956) (Prepn) 18) J. Carol, JAssocAgriChemists 43, 256-61 (1960) & Explosivst 1964, 45 (Infrared detn for ErTeN) 19) CondChemDoc (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-Verfahren 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 Abbt 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% H2O) 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 Verteigerungszünder

Eschbach (Eschbach Primer or Delayed Action Primer of Eschbach). Two models are described in Beiling-Dreikopf (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) Beiling-Dreikopf (1936), 232-35
2) W. Taylor et al, BIOS Final Rep 644 (1945), 3-16
**Eschko Mixture.** A mix of 2 parts Mg oxide and 1 part dried Na carbonate, used as a reagent for determining sulfur in coal, coke or petroleum 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


**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 exp, the excess being driven out again on pressing. Most of the reported experiments were carried out with TNT incorporated with 4 to 14% paraffin wax pressed at 400 kg/cm² to pellets of 25mm diam weighing 27g each, but the conclusion was arrived that is better to use t alc 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/cm² 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 Encycl, p C493. The measure of effectiveness of the detonation was the amount 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 (Reps 3&4), a pellet 41 to 42mm high was used, and the hole for the detonator was 25mm deep. Completeness of detonation 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 detonation 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 of the detonator (See also Ref 5)


**ESP.** Abb for End of Sustained Pressure (in Rocketry)

Ref: 1)

**Espodon.** An early (1945), French, experimental rocket airplane. It was Europe’s first postwar rocket-powered airplane. Propellant—nitric acid and furanaline

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) Rejs: 1) Hackh's Dict (1944), 314-R 2) "The American College Dictionary", Random House, NY (1952), 410-L 3) E.Ott, Edir, "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. Rejs: CondChemDict (1961), 449-L

Espar Powder. Brit. prot. patented in 1885: Na nitrate 60%, sulphur 14% & sawdust 26%. It was hygroscopic due to presence of Na nitrate. Rejs: 1) Daniel (1902), 283 2) Fedoroff & Clift 4 (1946), p 31

Esplodenti: Same as Esplosivi

Esplosioni. Ital for Explosions. They can be lente (slow) or 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 (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 polvere (pressed explosives), such as TNT, Tetryl, PA, /fiammatizzato (phlegmatized) T4;
- and polverulenti sciliti (loose powders) such as Amatol with 80% AN

Esplosivi quasi solvindi (Nearly solid explosives) can be subdivided into: trasfilati o pressati a caldo da miscelle colloidali [wiredrawn (passing "screw press"), such as "sausage machine", shown in Figs 32 & 33 of Nau'm's NG, pp 305-06 and in Fig 43, p 182 of Giorgio (Ref 9) or warm-pressed colloidal mixtures], e.g. Balistiti, Dinamiti e ottenuti per raffreddamento di miscelle pastose (obtained by cooling dough-like mixtures), such as Amatol contg 60% AN

Esplosivi liquidi: nitroglicerina (NG), nitroglicolico (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 prep by blending in a "wheel mill", called molazz or macine, in Italian (see Figs 128 & 129, p 381 in Ref 8). This method of manuf has been used in manuf of BkPdz and some Dynamites

Esplosivi Italiani (arranged in alphabetical order):

Abelli: Vol 1 of Encycl, p A2
Ajosite: 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 (Pemissible Explosives): Vol 1, p A286-R
<table>
<thead>
<tr>
<th>Material Type</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Explosive solid</td>
<td>An explosive solid is a solid material that is designed to release energy when it is ignited or activated. It is typically used in applications where a rapid and controlled release of energy is required, such as in blasting operations, fireworks, and military applications.</td>
</tr>
<tr>
<td>Explosive liquid</td>
<td>An explosive liquid is a liquid material that is designed to release energy when it is ignited or activated. It is typically used in applications where a rapid and controlled release of energy is required, such as in the ignition of rockets and missiles.</td>
</tr>
<tr>
<td>Miscellaneous solid</td>
<td>Miscellaneous solid refers to a variety of solid materials that are not categorized as explosives. These materials may include asbestos, polystyrene, and other insulation materials.</td>
</tr>
<tr>
<td>Miscellaneous liquid</td>
<td>Miscellaneous liquid refers to a variety of liquid materials that are not categorized as explosives. These materials may include solvents, fuels, and other chemicals.</td>
</tr>
</tbody>
</table>

**Table E 16**

<table>
<thead>
<tr>
<th>Material Type</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Explosive solid</td>
<td>An explosive solid is a solid material that is designed to release energy when it is ignited or activated. It is typically used in applications where a rapid and controlled release of energy is required, such as in blasting operations, fireworks, and military applications.</td>
</tr>
<tr>
<td>Explosive liquid</td>
<td>An explosive liquid is a liquid material that is designed to release energy when it is ignited or activated. It is typically used in applications where a rapid and controlled release of energy is required, such as in the ignition of rockets and missiles.</td>
</tr>
<tr>
<td>Miscellaneous solid</td>
<td>Miscellaneous solid refers to a variety of solid materials that are not categorized as explosives. These materials may include asbestos, polystyrene, and other insulation materials.</td>
</tr>
<tr>
<td>Miscellaneous liquid</td>
<td>Miscellaneous liquid refers to a variety of liquid materials that are not categorized as explosives. These materials may include solvents, fuels, and other chemicals.</td>
</tr>
</tbody>
</table>

**Table E 16**

<table>
<thead>
<tr>
<th>Material Type</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Explosive solid</td>
<td>An explosive solid is a solid material that is designed to release energy when it is ignited or activated. It is typically used in applications where a rapid and controlled release of energy is required, such as in blasting operations, fireworks, and military applications.</td>
</tr>
<tr>
<td>Explosive liquid</td>
<td>An explosive liquid is a liquid material that is designed to release energy when it is ignited or activated. It is typically used in applications where a rapid and controlled release of energy is required, such as in the ignition of rockets and missiles.</td>
</tr>
<tr>
<td>Miscellaneous solid</td>
<td>Miscellaneous solid refers to a variety of solid materials that are not categorized as explosives. These materials may include asbestos, polystyrene, and other insulation materials.</td>
</tr>
<tr>
<td>Miscellaneous liquid</td>
<td>Miscellaneous liquid refers to a variety of liquid materials that are not categorized as explosives. These materials may include solvents, fuels, and other chemicals.</td>
</tr>
</tbody>
</table>

Ammonal - Vol 1, p A291-R
Ammonitamite - Vol 3, p C440-L
Ammonite - Listed by Giorgio (Ref 9, p 172) as one of the "Explosivi polverulenti normali" without giving its exact compn
Amongelatina - 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 "Explosivi polverulenti normali" without giving its exact compn
ASN - Vol 1, p A496-L
Astralit - 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 Explosivi.** Non-permissible expls manufd by the Mangiarotti Co, Codroipo (Udine). The following are listed in their catalog obtd for us by Dr Omero Vertori, Aulla (Massa Carrara), who also gave their approx comps

- **BM 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**
- **BM 2C per uso a cielo aperto (for use in open): AN & TNT; brown or yellow 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 "Explosivi al 56-60% NGL con 4-5% nitrocellulosa, 30-35% nitrate, ammonico and other ingredients"

**BM esplosivo da mina con nitroglicerina** listed by Belgrano (1952) in Table of p 281 as formulation Z2 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 CI-R**

**Cadinite - Vol 3, p 440-L**

**Canopus - Vol 2, p C39-R**

**Carica cava direzionale or Carica a punta vuota.** Shaped Charge in Engi, 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 SGMC (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**

**Cremmita - Vol 3, p C556-L**

**Cyclonite.** See T4

**DINAMITI ITALIANI (Explosivi Italiani da Mina con Nitroglicerina).** Italian Dynamites can accdg to Belgrano (Ref 6, p 85) and Giua (Ref 8, p 393), 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 *dinamite 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: NG70, matrice legnose 5 & NaNO₃ 25%; Dinamite Numero 1: NG 70, matrice legnose 8, NaNO₃ 18 & carbon 4%; Dinamite Numero 3: NG 25, matrice legnose 19, NaNO₃ 54 & NaCO₃ with ocher 2%. Belgrano (Ref 6) lists in Table on pp 282-83 thirty formulatons 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 (cotton colloidio) 8% which corresponds to Blasting Gelatin while the 2nd formulation:NG 83.0, CC 5.0, Na nitr rate 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: NG74, 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 nitroglycerin (NGc), iso-butylglycerin tetranitrate, metrol trinitrate and liq DNT

Permissible Dynamites (Dinamiti antigrisoutosi) consist, accdg to Giorgio (Ref 9, p 176) of gris outina contg 13.2% and 10% of NG. Belgrano (Ref 6, p 165) lists grisoutina contg NG10, 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 fossilie 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 "polverulenti", 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 64% CC (colloid cotton)
2) Esplosivi al 56-60% di NGL, with the rest being AN30-35% and small ants 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), trinitolo (TNT), or pantrite (PETN) or T4 (Cyclonite). To this class belong ital expls Superdynamon, 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, Ammondire and Martia a and y

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), miscelle nitro-ntafta (AN/fuel oil mixtures) (p 173), "esplosivi a base di clorati" (p 174), esplosivi "Slurry" (p 174) and "esplosivi nella lotta anti-griandine" (expls to fight against hail) (p 180) do not belong to "dinamiti" because they do not contain NG

As an exception may be mentioned Gelatina Chedditi, which contains 5-7% NG (p 174 of Ref 9) (Refs are listed at the end of the section entitled "ESPLOSIVI ITALIANI."
Dinamite. See Vol 3, p C440-L

Dinamone I ed II. Powdery expls, manufd by the Consorzio Fabricanti Dinamiti. They are listed by Isso (Ref 7, p 32) without giving their compns. Giorgio (Ref 9, p 172) lists them among "esplosivi pulvurienti normali", which are based on 80/20 ratio of nitrate/TNT and might contain 4-6% NG.

Dinitrotoluidina esplosiva. See MST or Nougat, Schneiderite and Siperite

Echoes or Escho—Vol 2, p C440-L

Esplosivi all'aria liquida (Liquid Air Explosives). See Belgrano (Ref 6) p 156

Esplosivi antigel o Esplosivi incongelabili (Non-freezing Explosives). See here under DINAMITI ITALIANI in this section.

Esplosivi a base di clorato. See Giorgio (Ref 9, p 174) and Giua (Ref 8, p 390)

Esplosivi antigrissoutosi (Permissible Explosives). See Belgrano (Ref 6, pp 165-72) and Giorgio (Ref 9, pp 177-78)

Esplosivi a base di nitro 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 perchlorato di ammonio. See Giua (Ref 8, p 400)

Esplosivi a base di perchlorato di potassio. See Giua (Ref 8, p 400)

Esplosivi flemmatizzati. Expls such as PETN or RDX phlegmatized with wax, etc

Esplosivi gelatinosi al binitrotolulo. See Esplosivi da mina gelatinosi al binitrotolulo

Esplosivi gelatinosi con nitroglicerina. See Esplosivi da mina gelatinosi con nitroglicerina

Esplosivi geosismici o Esplosivi per prospezione 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 sulfater 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-isobutylglycerinatriacetate 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. Thirty formulations are listed in Table on pp 281-82 of Ref 6

Esplosivi da mina con residuati di polveri di lancia. 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 Explosivi da mina taken from Belgrano are listed in Vol 3, p C451

Explosivo plastico: Cyclonite 85-89, petrolatum 10-12, thiocol 0.5-2 & glycerophthalic resin 0.5-2% [CA 43, 7688(1949)]

Explosivo P - Vol 3, p C440-L

Explosivo S - Vol 3, C440-L

Explosivo S20 - Vol 3, p C440-L

Explosivo Sc. Same as Schneiderite.

FNP (Fillers for bombs). Two formulations are listed in Vol 2, p B118-L

Evangelidi (Explosivo) (1904): KClO₃ 62.5, K ferrocyanide 30.0, charcoal 4.5 & gycerine 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 ochre 0.3% (Ref 6, p 280, formulation 9)

GDIM: NG 38, CC 2.3, AN 50.9, Na nitrate, 6, red ochre 0.5, oil 2 & Na carbonate 0.3% (Ref 6, p 281, form 17)

GDI1: NG 43.2, CC 2.3, wood flour 7, Na nitrate, 45.5, Na carbonate 1 & yellow ochre 1% (Ref 6, p 280, form 3)

GDI2: 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

Goma A: NG 92 & CC 8% (Ref 6, p 281, form, 29)

Goma B: NG 82.5, CC 5.5, wood flour 3, Na nitrate 8.5 & Na carbonate 0.5% (Ref 6, p 281, form 28)

Goma BM. See under BM esplosivi

Goma 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

Manianite - Vol 3, p C440-R

Martia a e y. Mining expls manufd by Sorlini (Antonio), Ghedi-Brescia, N Italy. Giorgio (Ref 9), lists them on p 172 under ”Explosivi polverugenti 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 - Italy 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) KNO₃ 75%, S 10 & C 15% and 2) KNO₃ 70, S 18 & C 12%. For polvere nera di caccia (BKPr) for sporting compn, given on p 292 of Ref 5 is: KNO₃ 75 to 78, S 9 to 12 & C 12 to 15%

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 of expln 1150 kcal/kg, but temp of expln not given. It used to be manufd by Fontana Liri Co for use in arma portatili (pistols and rifles) (Ref 9, p 206)

Giorgio also lists on p 207:
Balistite attenuata al binitrotoluene, 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 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 950 kcal/kg & 2700°C
   2) Polvere 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 Encycl, 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:

Polveri alla nitroacetilcellulosa (NAC): NG 27-32, NC 66-63 & Et Centr 7-4% . Heat of expln 860-960 kcal/kg

Polveri alla Italide: 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: Balistites in the form of cords are sometimes called Filiti

In addn to the above propllnts 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 Societa Dinamite Noble: NG2S, NC60, acetylcellulose 10 & EtCentr 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 compn. 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.

**Romete cava and Romete galleria.** Mining expls manufd by Polverificio Stacchini and listed by Isso (Ref 7, p 32) without giving their compn.

**Sabulite - Vol 3, p C440-K.**
**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.**

**Toluelammonial - Vol 2, p B118-L.**

**Tritolita.** Mixture of Tritol (TNT) with T4 Tritolial - 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 DB-5 - Vol 3, p C439-R.**

**Vulcanite P - Vol 3, p C439-R.**

**Refs on Esplosivi Italiani:**

1) R. Molina, "Esplosendi'', 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 Chimica Industriale No 14, Esplosivi'', Patron, Bologna (1947) (Lithographed)
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)
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 propne, 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°, Dynarnites No 1 (NG75 & grhr 25%) decom with evolution of nitrogen oxides at 200 to 210°; La(Lead Azide)> 235°, MF (Mercuricfulminate) 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 decomp takes place at 110°, test a new sample at 120°, etc. When the temp is reached at which the sample degrades 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 propint 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 asymptote to this curve will give the minimum temperature of deflagration (ignition, or detonation). Following are some values obt 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 decomp 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 det 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.

Explos 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 (bourroit de bois).

Refs: 1) Vennin, Burlot & Lécorché (1932), 212

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 propint 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 obt when a standard expl or propint is fired in the same manner. It is considered that the work performed by an explosive (travail de l'explosif) is proportional to the range.

Note 1: Accdg to Vennin et al (Ref), a caled 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 propints 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 Canz (Ref 1) and also briefly in Vol 2 of Encycl, p B6-R, under Ballistic Pendulum Chronographs.

A rather complicated method of calcula-
tion of various values is given in Ref 2, pp 270 & 271, but it is stated that the tables prepared in France give in most cases the required values without resorting to calculations.

Refs: 1) Cranz 3 (1927), 39-51  
2) Ven- 
nin, 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 Laboratoire 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 propell 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 (Bygroscopecy of an Explosive in Bulk)

(The term "en vrac" means in bulk, not packed or arranged but loose.

For its determination, 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 saturee
d'eau des explosifs encouche (Hygrosco-
picité de Cartridge Explosives in an Atmos-
phere Saturated with Water).

This test is designed for cartridges of min-
ing explosives wrapped in plain or paraffined
paper and conducted in the following manner:

Place on 8-cm layer of sawdust mixed with
an equal amount of water in a zinc-lined
wooden box and arrange about 2 cm 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 is 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 con-
tainer, 50mm 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, thermostati-
cally controlled at 40 ± 1° or at 50 ± 1°.
Leave for 21 days and reweigh the container
as well as the cardboard. 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) cor-
responds to the amount of exudant. The differ-
ence, P-p, corresponds to the loss in vol-
atiles (Ref 8)

5) Combustion en gouitière de 20mm (Com-
bustion in a 20mm Trough). Also called
Épreuve de chauffage de la gouitière or
Épreuve de propagation dans une gouitière
de 20mm de diamètre. 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 combus-
tion (Combustion Tests), also called Ap-
titudes 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(gouitier), 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 satura-
ted 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
propellant). If it is not very flammable (as TNT)
a few seconds (or minutes) will elapse before
the substance catches fire. This time inter-
val is dead with the aid of a stop watch.

As soon as the substance catches fire
the source of heat is withdrawn and obser-
vation is made as to whether the combustion
continues in the part of the trough which was
not heated. If the combustion does con-
tinue, the times necessary to reach the 50,
100cm, etc marks are recorded and the velo-
city 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 Boul-
langé 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 ton conique (Combustion in
a Conical Pile), also called Épreuve de sensi-
bilité à l'inflammation ou Aptitude à l'inflam-
mation (Sensitivity to Ignition (or Inflammation)
Test). The purpose of this test is to ascer-
tain the ease of ignition (inflammation) of an
expl or a propellant 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 meas-
uring 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 réglementaire du Services des Poudres", described by Muraour (Ref 1), consists essentially in allowing a steel hammer (mouton) of a definite weight (1.2 or 5 kg) to drop from a predetermined height onto a small, thin-walled cap (capsule de Bourges) containing 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 kgm is obtained. This value is known as "insensibilité de l'explosif" 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 30 kg (Sensitivity to Shock with a Big Hammer of 30 kg) (Impact Sensitivity Using a 30 kg 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 100 g) 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±0.1 g sample, which has been previously dried and sifted thru a screen with 1 mm 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 0.01 g a 10 g sample of expl, transferring it to a steel mold of 30 mm 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 obt for drawing pressures-density curve (Ref 8).

10) Transmission de la pression dans un explosif pulseur (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 obt with a particular explosive.

The test consists essentially in transferring a 100 g sample of powdered explosive, weighed to the nearest 0.1 g, 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 obtained (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, 30 mm in diam, loaded with 50 g 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 plate will be somewhat deeper. In this event it will be necessary to repeat the test with a smaller booster (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 booster 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 Travail 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 standardized in 1912 by the CSE (Commission des Substances Explosives) and a detailed description was given in the book of L. Venin, E. Burtol & H. Lécorché, "Les Pouderes et Explosifs", Ch. Réfranger, 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 also in other countries, including USA. It is described as item H in Vol 3 of Encycl., pp C311-R & C312, under CHRONOGRAPHS.


Essence C. Colorless liquid not miscible with water, but misc with alc., acet., or per ether; density 0.690 at 20/20°, nD1.3883 at 20°, range of distn 70 to 90°. Can be used as gelatinizer for NC.


Essence de mirbane. Ft 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
E 141

**Esso flame thrower.** An apparatus that throws a spray of liquid fuel that ignites in the air

**Essolube.** Trademark of Esso Standard Div of Hamble 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 Bk-Pdr in which part or total of K nitrte was replaced by K chlorate, manufd under Bertho-
leer's direction until a disasterous 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 conduc-
ted either by centrifuging or by filtering thru a Nutsche
Ref: 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
Ref: 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 ed (1965), 315-56 (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 Muraur (Ref 1) and with the use of the "capsules de Bourges". The comds tested were: NG, TMGDN (Trimethylene glycoldinitrate); NgC (Nitroglycol); 1,3-BuDDN (1,3-Butanedioldinitrate); 1,4-BuDDN; DEDGN (Dichethyleneglycoldinitrate) (pure); DEDGN (commercial), known in France as NEO and TEGDN (Triethyleneglycoldinitrate)
Ref: 1) H. Muraur, MAF 12, (1933)
2) L. Médard, MP 31, 131-43 (1949)

**Esters, Nitric of Polyhydroxy dicarboxylic Acids.** Krauz & Majrich (Ref) prep'd in the laboratory the dinitrates of the following acids: trihydroglutaric(CHOH)(CO₂H)₂, muacic(CHOH)₄⁻, succinic(CHOH)(CO₂H)₂ and saccharic(CHOH)(CO₂H)₄, 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% SO₃) 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 dinitrates are good stabilizers for NC, and in this respect surpass tartaric acid, (CHOH)₂(CO₂H)₂ or its dinitrate
Ref: C. Krauz & A. Majrich, Collect Czech ChemCommun 11, 639-48 (1939) & CA 35, 2721 (1941)