ENCYCLOPEDIA OF EXPLOSIVES AND RELATED ITEMS

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VOLUME 3

BY

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PREFACE

This volume represents a continuing effort to cover comprehensively the unclassified subject matter, in the same manner & format, as in Volumes I & II. The reader is urged to obtain the previous volumes and to read both the PREFACE and INTRODUCTION in Volume I in order to understand the authors' way of treating the subject matter

In the writing of the Encyclopedia, the authors have consulted freely with, and have had the cooperation of, many individuals who have contributed their expert knowledge. This fact is acknowledged throughout the text at the end of the subject item. A listing of many others who have been consulted or who have helped in other ways would be impractical. Mr Earle F. Reese of the Propellants Laboratory, Picatinny Arsenal, contributed significantly by collecting some references, writing and reviewing some portions of the manuscript, and by helpful discussions in other areas

Mr L. H. Eriksen, Director, Feltman Research Laboratories, Picatinny Arsenal, has authorized & approved this project, and thru his interest, encouragement & guidance this publication is possible. This Encyclopedia was prepared by personnel assigned to the Explosives Laboratory of FRL, Dr R. F. Walker, Chief. All reference works used (reports, periodicals, journals & books) were made available to the authors thru the cooperation of PA Scientific & Technical Information Branch personnel, Mr M. A. Costello, Chief. Mr John P. Noonan, Chief of the Technical Publications Section, and other personnel of the Section reviewed the manuscript in draft, checked the galley sheets, and reviewed the final copy

Although considerable effort has been made to present this information as accurately as possible, mistakes & errors in transcription do occur. Therefore, the authors welcome the readers to feel free to point out mistakes, errors & omissions of important works, so that corrections & additions can be listed in the next volume. The interpretations of data and opinions expressed are often those of the authors and are not necessarily those nor the responsibility of officials of Picatinny Arsenal or the Department of the Army

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Errata in Volume 1

Page	
XII, line 16 from bottom	Delete the sentence: More information....
Abbr 50-L	subst (instead of subs) for substance
Abbr 58-L	w/o (instead of wo) for without
Abbr 74, line 11	Ladreda v 4 (1945) instead of v 4 (1944)
A123-R, line 4	Replace "permissible expl" with "safe to handle expl"
A290, Table	Insert or betw Red Charcoal and Brown Coal
A545-R	Replace lines 8 & 9 from the bottom by: about 1931 have been restricted to crystalline LA which is desensitized with dextrin. This so-called "dextrinated LA" is now known as "Type I LA." Its purity is usually about 92.5% although the Spec min purity is 91.5% (See Table, p A559). Since about 1957, the "colloidal form of LA," which is not dextrinated, has been manufd in the US on a small scale. This is now known as "Type II LA" and its purity is nearly 100% (See Table, p A559). There is now also "Type RD-1333 LA" (originally Brit) which is obtd by crystn from a soln contg CMC (carboxymethylcellulose, Na salt). The US Spec requirements of LA RD-1333 are given in Spec MIL-L-46225B (March 1964) and of cryst LA in Spec MIL-L-3055A (Sept 1962)
A548-L, 11 & 12th lines from bottom	Delete: where the soln is heated by means of hot water coils
A548-L, 14th line from bottom	Change soln B to soln A
A682-R	Should read: Felixdorf Factory
Index	Ammonals (Austrian) instead of Ammionals

Errata in Volume 2

B 7-R, Ref 28	Should read: 28)H.L.Dryden,
B 20-L, last line	Should read: **Barium Fluoride.** See under Fluorides
B 33-R, **Benite**	Should read: See under Black Powder Modifications, p B173-R
B 92-L, line 20	Dinitrobenzyl Nitrate
B 128-L, lines 30 & 31	Should read: *Diallylpentaerythritol Ether* instead of Diallylpentaerythritol
B 128-L, 4th line from bottom	Should read: 1,3-Bis(β-aminoethanol)-benzene instead of 1,3(β-aminoethanol)-benzene

B 133-R, lines 8 & 9	Should read: Dibenzhydrazidhydrazid instead of Dibenzhydrazidazid
B 147-L, line 17	Should read: Bis(hydroxymethyl)-methylmethane instead of Bis(hydroxymethyl)-methane
B 152-R, 19th line from bottom	**N,N' -Bis(phenylazo)-cyclo-2,3,5,6,7-pentamethylene-1,4-diamine,** instead of.... -2,3,5,7-pentamethylene-....
B 155-L, line 16	Should read Di(picrylthiol)-xylene instead of Di(picrylthiol)-zylene
B 183-R	Should read: **Blast Effects in Water** (Underwater Blast) instead of (Underground Blast)
B 252-L	Delete: *Boron Chloride.* See Boron Trichloride
B 326-L, lines 13 & 25	Should read: Minie Ball instead of Minnie Ball
B 326-L, line 14	Should read: Capt Minié instead of Capt Minnie
B 364-L, **Burster**	Item should appear before **Bursterberger Explosives**
C 28-R, top line	Insert after *Recoilless Rifle,* called *Leaking Gun* by the British
C 79-R, Ref 29	Should read: T. Zimmerman, E. Wurzel & G. Demitrack
C 155-L	Insert before **CHEDDITES AND STREETITES:** **Chauyaky** (Jap). Castable 50/50 Cyclotol *Ref:* G.C.Tibbets et al, PB Rept 50394(1946)
C 157, Table 3	French Cheddite On°5 should read: DNT 23 instead of 2.3
C 160	Refs out of order
C 215-L, Ref 9	(1953) instead of (1949)
C 215-R, Ref 35	Zaehringer instead of Zaeringer
C 220-R	Cobaltous Acetylide instead of Cobaltous Azetylide

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Initiation of Detasheet	D100

SUPPLEMENT TO ABBREVIATIONS, CODE NAMES AND SYMBOLS GIVEN IN VOL 1, pp Abbr 1 to 65 AND IN VOL 2, p IX

AMCEDHS	Army Materiel Command Engineering Design Handbook Series, published by the (US) Army Research Office–Durham, Box CM, Duke Station, Durham, North Carolina 27706
AMCP	Army Materiel Command Pamphlet (US) Washington, DC, 20315
AMCRD	Army Materiel Command Research and Development
AmmPer	Ammonium Perchlorate
AmmPic	Ammonium Picrate
ANFO(Explosives)	Ammonium Nitrate-Fuel Oil Explosives
ASA	Brit abbr for Azide-Styphnate-Aluminum. It refers to LA, LSt & Al mixts which are used as priming compns in detonators having Tetryl or PETN as the base chge [McAdam & Westwater (1958), 52]
BPD	Bombrini Parodi-Delfino. Italian ammo plant located at Colloferro, near Rome
CFD	Consorzio Fabricanti Dinamiti (Ital). (Consists of BPD, SAEPCM & SIPE)
CHAPARAL	Amer Army forward-area air-defense system using a modified *Sidewinder* (an infrared guided missile), mounted on M548 self-propelled tracked vehicle *Ref:* AOA Bull **302** "The Common Defense," Dec 1965, p 4
CRIPE	Centre de Recherches Scientifiques et Techniques pour l'Industrie des Produits Explosifs, Bruxelles, Belgium
CTF	Capitolato Tecnico Fomiture (Ital) (Technical requirements for materials used in Army proplants)
CTI	Institute für Chemisch-Technische Untersuchungen (Institute for Chemical-Technical Investigations), West Germany
DAS	Deutsche Auslegeschrift (German description of patent)
DBP	Deutsches Bundes Patent (German State Patent)
DDCSTI	Defense Documentation Center for Scientific and Technical Information, Cameron Station, Alexandria, Virginia
EqS	Equivalent Sheathed (Explosive). An expl as safe as a sheathed expl of the same type

InfoCirc	Information Circular of US BurMines
MD	Ministerio Difesa (Ital). (Defense Ministry)
MMRBM	Mobile Midrange Ballistic Missile [Ordn **48**, 486(1964)]
NOLM	Naval Ordnance Laboratory Memorandum, US Naval Gun Factory, Washington 25, DC
NOLR	Naval Ordnance Laboratory Report
PA	Pentrite-Acetato di pentaeritrite. Ital expls of BPD consisting of 65/35 or 75/25 mixts of PETN & PETA. These expls are as powerful as TNT but more expensive
PAN	Pentrite-Acetato di pentaeritrite-Nitrato di ammonio. Ital mixts of PETN, PETA & AN developed by BPD. Such a mixt is as powerful as 80/20 Amatol but more expensive
PANA	PAN-Alumino. Ital mixts of PETN, PETA, AN & Al developed by BPD. These mixts are similar in strength to mixts of TNT, AN & Al but more expensive
Percn	Percussion
RARDE,GB	Royal Armament Research and Development Establishment, Great Britain (See also ARDE in Vol 1, p Abbr 5-L)
ReptInvest	Report of Investigation of US BurMines
RSIC	Redstone Scientific Information Center, US Army Missile Command, Redstone Arsenal, Alabama
SAP/BC	Semi-Armor Piercing Ballistic Cap
SpA or SPA	Società per Azioni (Ital) (Stock Company or Corporation)
SRAM	Short-Range Attack Missile for US Air Force version of TFX aircraft [Ordn **48**, 490(1964)]
SSBS	Sol-Sol-Balistique-Strategique (Fr). An ICBM-type ground-to-ground weapon [Ordn **48**, 490(1964)]
TechPaper	Technical Paper of US BurMines
TNAnd	Trinitroanilide
TNPhlGl	Trinitrophloroglucinol

SUPPLEMENT TO THE LIST OF ABBREVIATIONS FOR BOOKS AND PERIODICALS GIVEN IN VOL 1, pp Abbr 65 to 76 AND VOL 2, pp XI to XII

Andreev & Belyaev(1960)	K.K.Andreev & A.F.Belyaev, "Teoriya Vzryvchatykh Veshchestv" (Theory of Explosives), Oboronghiz, Moskva(1960)
Bandurin & Rukin(1959)	M.K.Bandurin & L.G.Rukin, "Sbornik Zadach po Teorii Vzryvchatykh Veshchestv" (Collection of Problems on the Theory of Explosives), GosIzdatOboronProm,Moskva(1959)
Berliner, Expls(1953)	J.J.Berliner & Staff, "Explosives," Pamphlet, NY(1953)
Berthmann(1960)	A.Berthmann, "Explosivstoffe," C.Hanser, München(1960)(Part of Vol 4 of Winnacker-Küchler, "Chemische Technologie")
Carpio 1(1948) Carpio 2(1949)	C.Carpio,"Corso di Esplosivi," Scuola Salesiana del Libro, Roma, Vol 1(1948) & Vol 2(1949)
Chalon(1911)	P.F.Chalon, "Explosifs Modernes," C.Béranger, Paris(1911)
DictUSMilTerms(1963)	Anon, "A Dictionary of United States Military Terms," Public Affairs Press, Washington, DC (1963)
FFK,Vol 1(1958) and Vol 2(1958)	Dr Alfred Langhans, "FFK Feuer-Fundamental -Klassifikation in Frieden und Krieg," Selbstverlag Dr Langhans, Bamberg, Germany, Vol 1(1958) Stoffbearbeitung and Vol 2(1958) Sach- und Namen-Register
FLACS	Journal published by the Florida Section of ACS
Giorgio(1964)	C.Giorgio, "Tecnica degli Esplosivi," Del Bianco, Udine(1964)
JETP	Journal of Experimental and Theoretical Physics, English translation: Soviet Phys JETP. See ZhEksp Teoret Fiz
Jiménez, Explosivos(1951)	J.M.Jiménez, "Explosivos," Ediciones Ejército, Madrid(1951)
Karpov(1945)	P.P.Karpov, "Sredstva Initsiirovaniya" (Initiating Agents), GosIzdatOboronProm, Moskva(1945)
Lusar(1958)	R.Lusar, "Die dentschen Waffen und Geheimwaffen des 2. Weltkrieges und ihre Weiterentwicklung," Lehmanns Verlag, München(1958)

McAdam & Westwater(1958)	R.McAdam & R.Westwater, "Mining Explosives," Oliver & Boyd, Edinburgh(1958)
Merriam-Webster's(1961)	"Webster's Third New International Dictionary," Merriam Co, Springfield, Mass(1961)
Piantanida(1940)	E.Piantanida, "Chimica degli Esplosivi e dei Gas di Guerra," Accademia Navale, Livorno (1940)
Propellant Manual SPIA/M2(1962)	"Propellant Manual," Solid Propellants Information Agency, JohnsHopkinsUniv, Silver Spring, Md(1962)
Simon(1947)	L.E.Simon, "German Research in World War II," Wiley, NY(1947)
Smith & Smith(1960)	W.H.B.Smith & J.E.Smith, "Small Arms of the World," Stackpole, Harrisburg, Pa(1960)
StdMethodsChemAnalysis **1**(1962)	N.H.Furman, Edit, "Standard Methods of Chemical Analysis," Vol **1**, VanNostrand, NY(1962)
StdMethodsChemAnalysis **2A**(1963)	F.J.Welcher, Edit, "Standard Methods of Chemical Analysis," Vol **2**, Part A, VanNostrand, NY (1963)
StdMethodsChemAnalysis **2B**(1963)	F.J.Welcher, Edit, "Standard Methods of Chemical Analysis," Vol **2**, Part B, VanNostrand, NY(1963)
Urbański(Vol & Year)	Tadeusz Urbański, "Chemi und Technologie der Explosivstoffe," Vol **1**(1961) (Translated from Czech), 389 pp; Vol **3**(1964) (Translated from Czech), 527 pp, VEB Deutscher Verlag für Grundstoffindustrie. English Translation from Polish "Chemistry and Technology of Explosives," Vol **1**(1964), Vol **2**(1965), 517 pp and Vol **3**(1966, tentative), Pergamon Press, Long Island, NY, USA
UspFiz	Uspekhi Fiziki(Progress in Physics) (sometimes called in CA Soviet Phys–Usp)
UspFizNauk	Uspekhi Fizicheskikh Nauk (Progress in Physical Sciences)
UspKhimTeknol-Polimerov, Sbornik	Uspekhi Khimii i Teknologii Polimerov, Sbornik (Progress in Chemistry and Technology of Polymers, Collection)
Vaskovskii(1957)	S.F.Vaskovskii, "Praktícheskoye Rukovodstvo po Obrashcheniyou s Vzryvchatymi Materialami" (Instruction for Handling Explosive Materials), GosGeologTekhIzdat, Moskva(1957)
ZhEksp Teoret Fiz	Zhurnal Eksperimental'noi i Teoreticheskoi Fiziki. See JETP
ZhPMTF	Zhurnal Prikladnoi Mekhaniki i Tekhnicheskoi Fiziki (Journal of Applied Mechanics and Technical Physics)

SUPPLEMENT TO THE LIST OF BOOKS ON EXPLOSIVES AND PROPELLANTS GIVEN IN VOL 1, p A676 AND VOL 2, pp C215 to C216-L

1) L.Vennin & G.Chesneau, "Les Poudres et Explosifs. Les Mesures de Sécurité dans les Mines de Houille," C.Béranger, Paris(1914)

2) N.A.Shilling, "Vzryvchatyiye Veshchestva i Snariazheniye Boyepripassov " (Explosive Substances and Loading of Ammunition), Oboronghiz, Moskva(1946)

3) M.A.Budnikov et al, "Vzryvchatyiye Veshchestva i Porokha " (Explosives and Propellants), Oboronghiz, Moskva(1955)

4) A.G.Gorst, "Porokha i Vzryvchatyiye Veshchestva" (Powders and Explosive Substances), GosIzdatOboronProm, Moskva(1957)

5) S.I.Sil'vestrovich, "Vzryvchatyiye Veschestva i Usloviya ikh Bezopasnogo Khraneniya" (Explosives and Their Safe Storage), GosIzdatLit po Stoitel'-Materialam, Moskva(1957),104pp

6) Dr A.Langhans, "FFK Feuer-Fundamental-Klassifikation in Frieden und Krieg," Selbstverlag Dr Langhans, Bamberg, Germany, Vol 1(1958) Stofferbearbeitung and Vol 2(1958) Sach- und Namen-Register

7) R.Lusar, "Die Deutschen Waffen und Geheimwaffen des 2. Weltkrieges und ihre Weiterentwicklung," Lehmanns Verlag, München(1958)

8) S.S.Penner & B.Mullins, "Explosions, Detonations, Flammability, and Ignition," Pergamon Press, NY(1959), 300pp

9) D.S.Avanesov, "Praktikum po Fizikokhimicheskim Ispytaniyam VV (Practical Manual on Physicochemical Experiments with Explosives), GosIzdatOboronProm, Moskva(1959), 165pp

10) V.M.Buzinov & V.P.Savelov, Anglo-Russkii Artilleriyskii Slovar',' (English-Russian Artillery Dictionary), VoyenIzdat, Moskva(1959)

11) Ye.Yu.Orlova, "Khimiya i Technologiya Brizantnykh Vzryvchatykh Veshchestv" (Chemistry and Technology of High Explosives), GosIzdatOboron-Prom, Moskva(1960), 396pp (English Translation by Technical Documentation Liaison Office, MCLTD, WP-AFB, Ohio, MCL-844/1+2, June 1961)

12) Z.G.Pozdnyakov, "Izsledovanie Stabil'nosti Promyshlennykh VV" (Testing the Stability of Industrial Explosives), IzdatInstGornDela, Moskva(1961), 23pp

13) F.Weichelt, "Handbuch der Sprengtechnik," Deutscher Velag Grundstoffind, Leipzig(1961), 547pp

14) Coll, "Les Ondes de Détonation," Colloques Internationaux du Centre National de la Recherche Scientifique No 109, 15 Quai Anatole-France, Paris (VII^{e})(1962)

15) M.E.Serebriakov, "Vnutrenniaiya Ballistika Stvol'nykh Sistem i Porokhovykh Raket" (Interior Ballistics of Gun Barrel Systems and of Solid Rockets), Oboronghiz, Moskva(1962)

16) B.Il.Belkovski & G.T.Dyakov, "Vzrivni Veshchestva: Teoriya, Khimiya, Tekhnologiya i Upotreba" (Explosive Substances: Theory, Chemistry, Technology and Use), Tekhnika, Sofia(1962), 339pp

17) D.B.Chidsey, "Goodbye to Gunpowder," CrownPublg, NY(1963), 224pp

18) K.K.Andreev et al, "Teoriya Vzryvchatykh Veshchestv" (The Theory of Explosives), Oboronghiz, Moskva(1963), 578pp

19) V.A.Assonov, "Vzryvchatyie Veshchestva i Ikh Primeneniye v Gornoi Promyshlennosti" (Explosives and Their Use in Mining Industry), GosNauchin-TekhnIzdLitGornDelu, Moskva(1963), 139pp

20) C.Casci, Editor, "Fuels and New Propellants," Macmillan, NY(1964), 355pp

21) A.A.Shidlovskii, "Osnovy Pirotekhniki" (Fundamentals of Pyrotechnics), Oboronghiz, Moskva(1964) (Translated by US Joint Publications Research Service, PATM 1615 (May 1965)

22) Anon, Commission des Substances Explosives, Service des Poudres, Book of Instruction, 4th Part, Articles 189-210 (obtainable thru the État-Major de l'Armée, 2e Bureau, Paris, France)

C

Alphabet letter C begins in Volume 2, p C 1

CHLORIDES

Chlorides. The salts of hydrochloric acid (HCl) containing the monovalent chloride ion (Cl^-) are called chlorides. They are usually a binary compd in which chlorine is the negative constituent. They are mostly well crystd compds, readily sol in w, readily stable towards w; and not, as a rule, decompd by heat. The toxicity of the chlorides varies widely (Ref 5). They are used in some expls & pyrotechnic mixts as cooling agents

Detection & Determination. Chlorides are detected qualitatively in soln by the formation of a wh ppt when $AgNO_3$ is added to the test soln acidified with nitric acid. This ppt is sol in dil aq ammonia, but is repptd on acidifying with dil HNO_3. A convenient method for the detection of chlorides in the solid state is to heat a small quantity, mixed with manganese dioxide & concd H_2SO_4. If chloride is present, chlorine gas will evolve. Further confirmation can be obtd by mixing a test sample with potassium dichromate & H_2SO_4; on warming, red vapors of chromyl chloride (CrO_2Cl_2) will evolve and condense in blood- red drops on cooler parts of a test tube

The reaction with silver nitrate serves to determine the presence of chlorides both gravimetrically & volumetrically; in the former case, chlorine is weighed as AgCl; in the latter, the vol of std $AgNO_3$ soln reqd for complete pptn of the chloride present in a known vol of soln is detd (Refs 2 & 4) (See also Refs 2a, 6 & 7)

Refs: 1)Mellor **2**(1922), 214ff 2)Mellor(1939), 505 2a)Scott & Furman(1939), 269-81 3)Hackh's(1944), 192 4)Kirk & Othmer **3** (1949), 677 & 680 5)Sax(1957), 461 6)Vogel(1961), 313, 361, 399 & 571 7)StdMethodsChemAnalysis **1**(1962), 324-49

List of Chlorides

Acetyl Chloride or Ethanoyl Chloride. See Vol 1, p A56-R

Aluminum Chloride, $AlCl_3$; mw 133.35, yel-wh delq hex crysts, mp 194° at 5.4 atm, bp 183° at 752mm, d 2.44 at 25°; sol in cold w, sl decompd by hot w; sol in eth, chlf & CCl_4; insol in benz; can be prep by passing chlorine or HCl vapor over heated Al or by other methods. It is a powerful desiccant and must be handled with precaution. When opening containers of $AlCl_3$ stored for a long time, extra care should be taken because some containers might explode (Ref 4). It reacts with w with expl violence

Aluminum chloride is an important reagent in org chem; it is used as a catalyst in many reactions, such as the cracking of petroleum. It is also used in pyrotechnic smoke producing compns (Ref 8)

Refs: 1)Mellor **5**(1924), 312-14 1a)Gmelin, Syst Nr **35**(1934), 163-217 2)C.A.Thomas et al, "Anhydrous Aluminum Chloride in Organic Chemistry", Reinhold, NY(1941) 3)Hackh's(1944), 39 4)P.V.Popov, ZavodskayaLab **13**, 127(1946) & CA **41**, 6723(1947) 5)Kirk & Othmer **1**(1947), 632-34 6)Sidgwick, Chem Elems **1**(1950), 424-25 7)Sax(1957), 261 8)J.DeMent, USP 2995526(1961), p 5 9)US Spec MIL-A -10389B (Aluminum Chloride, Anhyd, Technical)

Aluminum Chloride - Nitrobenzene Complex, $AlCl_3.C_6H_5NO_2$; lt yel ndls(Ref 1); grn-yel crysts(Ref 2); mp 90°(Refs 1 & 2). Was first prepd in 1910 by Menshutkin(Ref 1) and recently by Grossman (Ref 2), by adding dry purified nitrobenzene to dry $AlCl_3$. This complex has been used as a catalyst in some organic reactions.

In one of the fairly recent numbers of C & EN it was reported that an expln took place at one of the American Cyanamide plants when this complex was contained in an autoclave filled with gaseous olefin at 26° and 90 psi

Refs: 1)B.N.Menshutkin, JRussPhChemSoc **42**, 58(1910) & CA **5**, 1420(1911) 2)R.F. Grossman, JOC **22**, 581-2(1957) & **52**, 1090 (1958)

Ammonium Chloride(Sal-ammoniac), NH_4Cl; mw 53.50, wh crysts with sharp saline taste; mp,vaporizes at 340-50° & sublimes without melting; d 1.520; sol in w or alc. It is manufd from the ammoniacal liquor of gas works by neutralizing with HCl; the soln is evapd until crystn occurs; and the crude product is purified by sublimation. The toxicity of ammonium chloride is given in Sax (Ref 7)

It is used as a chemical reagent, in medicines, as a filling for dry batteries, for soldering flux, and in textile printing. It

has been used also as an ingredient of mining expls: a)K chloride 39, K borate 18, NH_4Cl_2, coke or wood charcoal 2, glycerol 2, refined petroleum 2 parts (Ref 1); and b) NH_4Cl, $KClO_4$, NaCl mixt (in a 3/1/1 ratio for any combination of the three salts) 60-80 pts, nitroglycol 5-12 pts and TNT 6-10 pts (Ref 2)

Yamamoto et al (Ref 6) detd the ignition temp of various mixts of $NaNO_2$ & NH_4Cl and found that such mixts, when stored over $CaCl_2$, undergo spontaneous expln after 30-50 days

Refs: 1)(?) Golovine, FrP 353299(1905) & CA **1**, 1343(1907) 2)C.E. Bichel, FrP 356845(1905) & CA **1**, 1344(1907) 3)Naoúm, NG(1928), 405 3a)Gmelin, Syst Nr **23**(1936), 150-92 4)Mellor(1939), 398 5)Hackh's(1944), 47 6)S. Yamamoto et al, JIndExplsivesSoc-Japan **15**, 77-80(1954) & CA **49**, 11282(1955) 7)Sax(1957), 276 8)US Spec O-A-491C [Ammonium Chloride(Sal Ammoniac), Technical]

Antimony Chlorides. Two compds are of interest: *Antimony Trichloride*(Butter of Antimony), $SbCl_3$; mw 228.13, col cryst solid, mp 73.4°, bp 219-23°, d 3.14; sol in concd HCl & in org solvs; insol in w or dil HCl. It is prepd by the reaction of Sb on a limited amt of Cl; or by dissolving the sulfide in concd HCl, distilling the excess acid, and purifying the chloride by sublimation. The compd is very hygr and is hydrolyzed to *Antimony Oxychloride*, SbOCl, used as a smoke-producing agent

Antimony Pentachloride, $SbCl_5$; mw 299.05, col liq, yel when impure, fr p 2.8°, bp 140° (sl dissociated), d 2.37. It is prepd by burning Sb in chlorine; or by the action of excess chlorine on the trichloride, oxide or sulfide. It is used as a chlorine carrier in org syntheses and as a catalyst in some polymerizations

Refs: 1)Mellor **9**(1929), 469(trichloride) & 486(pentachloride) 2)Thorpe **1**(1937), 447 (trichloride) 3)Mellor(1939), 754 4)Hackh's (1944), 65(pentachloride) & 66(trichloride) 5)Kirk & Othmer **2**(1948), 61 6)Gmelin, Syst Nr **18**(1949), 408-38(trichloride) & 439-61(pentachloride) 7)Sax(1957), 307 (pentachloride) & 309(trichloride) 8)US Spec MIL-A-10667B(Antimony Trichloride, Technical) 9)US Spec MIL-A-10824 (Antimony Chloride, Reagent)

Arsenic Trichloride(Arsenious Chloride), $AsCl_3$; mw 181.28, col oily liq, fr p -18°, bp 130.2°, d 2.16 at 25°, vap pres 10mm at 24°; sol in w, alc, eth or oils. It is formed when arsenic burns in chlorine, or by distilling a mixt of As trioxide with HCl, or by heating As_2O_3 with sulfur chloride in a current of chlorine. It is used in ceramics, in the prepn of pharmaceuticals, and in the prepn of chloro derivs of arsine (qv) or chemical warfare agents (CWA's)(qv). See Vol 1, p A491

Refs: 1)Mellor **9**(1929), 237 2)Thorpe **1** (1937), 479 3)Mellor(1939), 745 4)Hackh's (1944), 77 5)Kirk & Othmer **2**(1948), 119 6)Gmelin, Syst Nr **17**(1952), 367-92 7)Sax (1957), 320 8)US Spec MIL-A-10839B (Arsenic Trichloride, Technical)

Azidoacetyl Chloride. See Vol **1**, p A57-L

Barium Chloride, $BaCl_2$; mw 208.27, monoclinic or cubic crysts; mp 960° (transition to cubic at 925°), bp 1560°, d 3.856; sol in w; insol in alc; it absorbs w avidly forming hydrate: *Monohydrate*, $BaCl_2 \cdot H_2O$; mw 226.29; col plates; formed by heating the dihydrate at 60-65°, or by shaking the dihydrate with 99% MeOH, filtering & crystallizing. *Dihydrate*, $BaCl_2 \cdot 2H_2O$; mw 244.31; fine wh crysts, mp (loses 2 mols H_2O at 100°), d 3.097; produced commercially by treatment of Ba sulfide with HCl; the aq solns of $BaCl_2$ deposits crysts of the dihydrate. The anhydrous salt is obtd by drying the crysts at high temp. Barium chloride is poisonous (Ref 6), but it is used widely in industry in a variety of ways.

Refs: 1)Mellor **3**(1923), 697ff 2)Gmelin, Syst Nr **30**(1932), 170-209 & **30**(1960), 179 & 323-69 3)Mellor(1939), 638 4)Hackh's (1944), 98 5)Kirk & Othmer **2**(1948), 312-14 6)Sax(1957), 331

Boron Chloride Pentahydride. See Vol 2, p B252-L

Cadmium Chloride, $CdCl_2$; mw 183.32, col hexagonal crysts, mp 568°, bp 960°, d 4.05; sol in w, MeOH & alc; forms hydrates with 1, 2, 4 & 5 moles of H_2O. It can be made by dissolving the metal in aq HCl and evapg the soln in a stream of hydrogen chloride gas, or by dissolving the oxide or carbonate in

HCl. It is used in a variety of ways in industry, and in smoke producing pyrotechnic compns (Ref 5)
Refs: 1)Gmelin, Syst Nr **33**(1925), 82-91 & **33**(1959), 462-512 2)Hackh's(1944), 154 3)Kirk & Othmer **2**(1948), 734 4)Sax(1957), 418 5)J. DeMent, USP 2995526(1961), p 7

Calcium Chloride, $CaCl_2$; mw 110.99, col, delq cubic crysts, mp 772°, bp >1600°, d 2.512, n_D 1.52 at 20°; sol in w or alc; forms mono-, di-, tetra- & hexa-hydrates. This salt is obtd as a by-product in several manufg processes, such as the ammonia-soda process & the production of KCl. It is used as a drying agent in the lab, and commercially as an antifreeze, antidust & conditioning agent
Refs: 1)Mellor **3**(1923), 697ff 2)Mellor (1939), 631 3)Hackh's(1944), 157 4)Kirk & Othmer **2**(1948), 759-61 5)Gmelin, Syst Nr **28**(1956), 74-83 & **28**, Teil B, Lieferung 2(1957), 431ff 6)Sax(1957), 425 7)US Spec O-C-106A(2)(Calcium Chloride, Hydrated, Technical Grade); US Spec MIL-C-13573A (Calcium Chloride, Anhydrous); US Spec O-C-104(Calcium Chloride, Anhydrous, Technical Grade); US Spec O-C-105A(1) (Calcium Chloride, Dihydrate and Calcium Chloride, Anhydrous, Technical)

Chromium Chloride*(Chromic Chloride or Trichloride),* $CrCl_3$, mw 180.05; pink crysts, mp 1150°, dec 1200-1500°; d 2.75 at 15°; insol in cold w, alc, acet & CS_2; v sl sol in hot w with decompn; can be prepd by passing chlorine over mixt of chromic oxide & carbon. Its *hexahydrate,* $CrCl_3 6H_2O$, mw 266.48; deliq crysts either greenish-blk(mp 83°) or violet(mp 95°); d 1.835 at 25/4°; very sol in w, alc; insol in ether; can be prepd by the action of HCl on Cr hydroxide(Refs 1, 2, 3, 5 & 6). Toxicity of Cr chlorides is the same as other Cr compds(Refs 3 & 4). US Military requirements and tests for reagent grade Cr chloride are discussed in Ref 7. Industrial uses include chromium plating, catalyst for polymerizing olefins, prepn of other Cr salts, in calico printing and as a mordant for cotton and silk.
Refs: 1)Mellor **11**(1931), 371-2 2)Kirk & Othmer **3**(1949), 943.4 3)Ullmann **5**(1954), 592-4 4)Sax(1957), 488 5)CondChemDict (1961), 273 6)Lange(1961), 240-1 7)US Military Specification **MIL-C-11336**(Chrominum chloride, reagent)

Chromyl Chloride or Chromium(VI) Dioxychloride*(Chromium Oxychloride or Chlorochromic Anhydride),* CrO_2Cl_2, mw 154.92; dk-red, heavy liq resembling bromine; fr p -96.5°, bp 117.6°, vap pressure 20mm at 20°, d 1.923 at 20°; sol w/o decompn in ether, CCl_4, CS_2, MNB & tetrachloroethane; but is rapidly hydrolyzed by w. Can be prepd by distg a mixt of anhyd Na chromate(or dichromate) with coned H_2SO_4 & NaCl(Refs 1, 1a, 2, 5 & 6). Its toxicity and fire hazard are discussed by Sax(Ref 4)

Chromyl chloride is a very powerful oxidizer and causes many combustible materials to burst into flame on contact. It has been used(freshly prepd) in org oxidations & chlorinations, and in prepn of Cr complexes & dyes
Refs: 1)Mellor **11**(1931), 391 1a)Inorg-Synthesis **2**(1946), 205-7 2)Kirk & Othmer **3**(1949), 394 3)Ullmann **5**(1954) not found 4)Sax(1957), 487 5)CondChemDict(1961), 276 6)Lange(1961), 242-3

Cobalt Chlorides: *Cobaltous,* $CoCl_2$; mw 129.85, dk-blue crysts, mp 735°, bp 1050°, d 3.356; gives pink solns when dissolved in cold w; formed from the hexahydrate by heating it to 120°.
Hexahydrate, $CoCl_2 \cdot 6H_2O$; mw 237-97, ruby red monoclinic crysts, mp 87°, d 1.924 at 25°; obtd from solns of the oxide or carbonate & HCl; forms the *Dihydrate,* $CoCl_2 \cdot 2H_2O$, d 2.477 at 25°, on heating to 56°; and the *Monohydrate,* $CoCl_2 \cdot H_2O$, violet-colored crysts, on heating to 100°. The anhyd salt reverts to various hydrated forms on exposure to moist air
Cobaltic Chloride, $CoCl_3$; mw 165.31, blue crysts, mp sublimes, d 2.94, is listed in Refs 2, 4 & 6. Cobalt chloride has been proposed for use in smoke producing compns(Ref 7)
Refs: 1)Gmelin, Syst Nr **58**(1932), 269-301 & **58**(1961), 576-80 2)Mellor **14**(1935), 611ff (cobaltous) & 653(cobaltic) 3)Mellor(1939), 827 4)Hackh's(1944), 208(cobaltic) & 209 (cobaltous) 5)Kirk & Othmer **4**(1949), 204-05 6)Sax(1957), 493(cobaltic) & 494(cobaltous) 7)J. DeMent, USP 2995526(1961), p 8

Copper Chlorides.
Cuprous Chloride, Cu_2Cl_2; mw 198.05, wh

cubic crysts, mp 422°, bp 1366°, d 3.53; readily sol in HCl & NH_4OH; insol in w, H_2SO_4, dil HNO_3 & alc; prepd by boiling a soln of cupric chloride with HCl & copper, or by reducing a $CuCl_2$ soln with sulfur dioxide. On exposure to air & sunlight, the moist powd changes color to yel, dirty violet and finally to blue-blk. A wh vapor forms when CuCl is heated in air. It has various uses in the lab and in chem manuf processes; used for the denitration of NC, and in some pyrotechnics, smoke-producing & gas generating compns. For example the following mixt was developed at PicArsn for blue light rifle signal: Cu(I) chloride 10, K perchlorate 56, Cu(II) acetoarsenite 24 & Cu(II) resinate 10%; burning rate 2.6in/min & CP 480/sq in (Ref 4). DeMent(Ref 8) patented some smoke producing compns, such as: a)Cu(I) chloride 1.5, K chlorate 4.0, sulfur 2.0 & Amm acid fluoride 2.0 parts and b)Cu(I) chloride 1.5, K chlorate 3.0, sulfur 1.0 & monochloracetic acid 1.0 part. Ellern(Ref 9) stated that a mixt of Cu(I) chloride with guanidine nitrate & vanadium pentoxide was patented in solid gas-generating compns

US Navy Blue Light, Mark 1, Formula 1 contains: Cu(I) chloride 22.0, Ba chlorate 56.0, CuO 13.0, shellac 7.0 & stearic acid 2.0%(Ref 4a)

Cu(I) chloride absorbs acetylene to form an explosive *Cuprous Acetylide Chloride.* It was briefly discussed in Vol 1, p A74-L

Cupric Chloride, $CuCl_2$, mw 134.48, yel-brn delq pdr, mp 498°, bp 993°(decomp), d 3.05; insol in alc, w or NH_4Cl; can be prepd by direct union of copper and chlorine. The *Dihydrate,* $CuCl_2.2H_2O$, long grn delq ndls, mp 110°(with loss of w), d 2.38, can be obtd by treating Cu(II) hydroxide or carbonate with HCl and evapg the soln. It has been used in dyeing & printing of textiles in metallurgy & photography. Davis(Ref 4) stated that the color of commercial MF can be improved if a small amt of $CuCl_2$ is added to the nitric acid soln of Hg before it is poured into the alcohol in the balloon

Refs: 1)Mellor **3**(1923), 157ff(Cuprous) & 168ff(Cupric) 1a)G.J. Schladt, PATR **357** (1933) 2)Thorpe **3**(1939), 356 3)Mellor (1939), 592(Cuprous) & 594(Cupric) 4)Davis (1943), 406 4a)L.Lo Fiego & G. Butters, NOLM **9292**(1947), US Naval Gun Factory, Washington, D.C. 5)Kirk & Othmer **4**(1949), 469(cuprous & 470(cupric) 6)Sax(1957), 503 (cupric) & 518(cuprous) 7)Gmelin, Syst Nr **60**, Teil B(1958), 203ff(cuprous) & 253ff (cupric) 8)J. DeMent, USP 2995526(1961), p 9 9)Ellern(1961), 120

Cyanogen Chloride. See CK under CHEMICAL AGENTS

Iron Chlorides.

Ferrous Chloride, $FeCl_2$; mw 126.76, col rhmb crysts, mp 672°, bp 1030°, d 2.988 at 18°; sol in w, alc or acet; insol in eth; is formed by the action of HCl on iron at red heat, and by hydrochloric acid on iron in absence of air. It forms a series of hydrates contg one, two, four & six moles of H_2O. The transition points of the latter two hydrates are 76.5°& 12.3°, respectively. On heating in air, ferrous chloride forms ferric chloride & ferric oxide

Ferric Chloride, $FeCl_3$; mw 162.22, dk hexagonal crysts, appearing red by transmitted light, & grn by reflected light, mp 300° (sublimes), bp 510°, d 2.8; very sol in w, MeOH, EtOH, acet & many org solvs; sl sol in CCl_4; insol in glycerin, eth acet, phosphorus trichloride or stannic chloride. It is formed by action of dry chlorine on red hot iron, by action of HCl on heated ferric oxide, or by oxidg $FeCl_2$ with chlorine or HNO_3. Ferric chloride is very hygro and forms a series of hydrates. It is the most important ferric compd and has numerous applications in the chem & allied industries; it has been proposed as an ingredient of pyrotechnic smoke-producing compns(Ref 8)

Refs: 1)Gmelin, Syst Nr **59**(1932), 184-221 (ferrous) & 221-3 19(ferric) 2)Mellor **14**(1935), 9ff(ferrous) & 40ff(ferric) 3)Mellor(1939), 817(ferrous) & 819(ferric) 4)Hackh's(1944), 335(ferric) & 337(ferrous) 5)Thorpe **7**(1946), 57(ferrous) & 59(ferric) 6)Kirk & Othmer **8** (1952), 58(ferrous) & 59(ferric) 7)Sax(1957), 713(ferric) & 717(ferrous) 8)J. De Ment, USP 2995526(1961), p 11 9)US Spec MIL-F-14580 (Ferric Chloride, Anhydrous)

Mercury Chlorides.

Mercurous Chloride(Calomel), Hg_2Cl_2; mw 472.14, wh rhmb crysts or powd, mp sublimes ca 400°, d 7.150; insol in w; can be prepd by pptn from a soln of mercurous nitrate by NaCl,

or by direct combination of Hg & Cl. It is used as a reagent, medicinally as a cathartic; and in pyrotechnic green light compns. De Ment (Ref 6) patented its use in smoke-producing compns

Mercuric Chloride (Corrosive Sublimate), $HgCl_2$; mw 271.52, wh rhmb crysts, mp 277°, bp 304°, d 5.440; sol in w, alc or eth; produced in USA chiefly by direct chlorination of mercury, older processes were based on reaction between mercurous sulfate & NaCl. It is extremely poisonous (antidote: 10% sodium thiosulfate soln). It is used in agriculture as a fungicide, in org reactions as a catalyst, and its solns are used medicinally as an antiseptic

Refs: 1)Mellor **4**(1923), 796-816(mercurous) & 816-39(mercuric) 2)Mellor(1939), 651 (mercurous) & 652(mercuric) 3)Hackh's(1944), 524(mercuric) & 525(mercurous) 4)Thorpe **7**(1946), 571(mercurous) & 574(mercuric) 5)Sax(1957), 857(mercuric) & 866(mercurous) 6)J. De Ment, USP 2995526(1961), p 13 7)US Spec JAN-M-201 [Mercurous Chloride (Calomel)(For Ordnance Use)]

Monochloramine. See Chloramine

Nitroacetyl Chloride. See Vol 1, p A57-L

Nitrogen Chloride (Nitrogen Trichloride), NCl_3; mw 120.38, N 11.64%; bright yel oil with a powerful irritating odor & vapors which attack the eyes, fr -40°, bp 71°, explodes at 93°, d 1.653; sol in benz, CS_2, chlf, eth & sulfur dichloride; insol in w. It was accidentally obtd in 1811 by Dulong(See in Refs 1, 2 & 4)(who sustained serious injury by an expln) by action of chlorine on a soln of NH_4Cl. Numerous methods of its prepn are given in Thorpe(Ref 4). Its toxicity & expln hazards are discussed in Sax(Ref 6). This compd is very sensitive and decomp with expl violence. Solns of NCl_3 in inert solvs, up to 20% strength, are not expl (See W.A. Noyes cited in Ref 5). The addn of chlorine or hypochlorite to ammonia is not hazardous under alkaline conditions, but if the solns become neutral or acid, nitrogen trichloride may form(Ref 5)

Refs: 1)Mellor **8**(1928), 599 2)Mellor(1939), 401 3)Hackh's(1944), 577 4)Thorpe **8**(1947), 511ff 5)Kirk & Othmer **3**(1949), 665-67 6)Sax(1957), 950

Nitrosyl Chloride (Nitrogen Oxychloride), NOCl; mw 65.47, orn-red gas with a chlorine-like odor, condenses to a cherry-red liq, fr p -59.6°, bp -5.8°, d(liq) 1.250 at 30°; the liq is miscible in all proportions with chlorine & nitrogen tetroxide, and is sol in chlf, CCl_4 & methylene chloride. The prepn & props of NOCl have been the subject of a recent review(Ref 5). This compd is one of the components of "aqua regia"(See Vol 1, p A476-L), being evolved with chlorine when strong nitric & hydrochloric acids are mixed. Its uses include bleaching of flour, in preparative org chemistry, and as a chlorinating agent

Refs: 1)Mellor **8**(1928), 612 2)Mellor(1939), 424 3)Hackh's(1944), 577 4)Thorpe **8**(1947), 531 5)L.J. Beckham et al, ChemRevs **48**, 319-96(1951) 6)Kirk & Othmer **9**(1952), 462 7)Sax(1957), 959

Nitryl Chloride (Nitroxyl Chloride), NO_2Cl; mw 81.47, col gas, condenses to a liq, fr p -145°, bp -16°, d(liq) 1.37; is formed by reaction between nitrosyl chloride & ozone (Ref 1). Its toxicity & expln hazards are given in Sax(Ref 4). Ogg & Wilson(Ref 2) studied its stability in the gaseous state. This compd has been investigated as a nitrating agent but this reagent must be handled with great care because of danger of explns(Ref 3)

Refs: 1)Thorpe **8**(1947), 541 2)R.A. Ogg, Jr & M.K. Wilson, JChemPhys **18**, 900-1(1950), & CA **44**, 6759(1950) 3)Kirk & Othmer **9**(1952), 319 & 421 4)Sax(1957), 962

Potassium Chloride, KCl; mw 74.56, wh cubic crysts having a strong saline taste, mp 776°, bp 1500°, d 1.987(Refs), Q_f^v 1396 cal/g(Ref 8); sol in w, sl sol in alc, insol in abs alc. It occurs naturally as *sylvite* and is extracted from certain lake brines & purified by recrystn; and is obtd also by fusion or extraction of *carnallite* ($MgCl_2 \cdot KCl \cdot 6H_2O$) with a soln of $MgCl_2$. It is used as a chem reagent, as a constituent of fertilizer, as a flash reducing agent in proplnts, and in permissible AN expls as a cooling agent(Ref 5). (See also Ammonium Nitrate Blasting Explosives, Vol 1, p A341ff)

Refs: 1)Mellor **2**(1922), 521-61 2)Thorpe **5** (1924), 371-87 & **10**(1950), 155 3)Gmelin, Syst Nr **22**(1938), 349-71 4)Mellor(1939), 576 5)Davis(1943), 323-25 & 352-53 6)Hackh's (1944), 677 7)Kirk & Othmer **11**(1953), 24-5 8)P. Tavernier, MP **38**, 312 & 331(1956) 9)CondChemDict(1961), 927

Silver Chloride, AgCl; mw 143.34, wh granular

powd which darkens on exposure to light & finally turns blk, mp 455°, bp 1550°, d 5.561; sol in NH_4OH, concd H_2SO_4, Na thiosulfate, & K bromide solns; v sl sol in w. It occurs in nature as the mineral *horn silver;* and is formed when a soln of a sol chloride is added to a soln of a Ag salt. Silver chloride in contact with metallic Na or K is exploded by percussion (Ref 3). Its ammoniacal soln absorbs acetylene, forming *Silver Acetylide Chloride* complexes which are expl (See Vol 1, p A80-L). Silver chloride is used in medicine as an antiseptic & nerve sedative, in the manuf of pure Ag & Ag salts, and in photography & photometry(Refs 1, 2, 4, 5 & 9)

Refs: 1)Mellor **3**(1923), 390-408 2)Mellor (1939), 602-3 3)Davis(1943), 402 4)Hackh's (1944), 773 5)Thorpe **10**(1950), 765 6)O.L. Williams, JChemEduc **30**, 347(1953)(Reclaiming silver from AgCl residues) 7)Kirk & Othmer **12**(1954), 439 8)Sax(1957), 1104 9)Cond-ChemDict(1961), 1022-23

Sodium Chloride(Salt), NaCl; mw 58.45, col cubic crysts, mp 801-4°, bp 1413-90°, d 2.161, η_D 1.554 at 20°; sol in w or glycerol; very sl sol in alc. It occurs in nature as the mineral *halite or rock salt* as its most abundant form, except as it occurs in sea water. It is derived from its natural sources by: a)soln of rock salt in w, filtration & crystn b)evapn & crystn of brines and c)evapn & crystn of sea water. Salt is a dietary necessity and is an extremely important commodity indispensable to animal life, but it is even more important as a chem raw material since more salt is used in the manuf of chemicals than any other basic material(Ref 9).Salt is used in some permissible AN expls as a cooling agent(See Ref 12) similar to the use of KCl, and as a flash reducer in proplnts(Ref 4). A Ger Patent(Ref 3) was issued for expl compns contg more than 65% of an inert material, such as NaCl. Eg: NG 30 & NaCl(to pass a Ger sieve DIN NO. 100) 70%. An expl contg TNT 40-50 & NaCl 60-50% was used in Germany during WWII(Ref 11a) Taylor(Ref 8) proposed using a special low d NaCl, as a quenching agent, in the prepn of a low d AN blasting expl

In the electrolysis of soln of NaCl, Dodero & Behroun(Ref 10) reported that expl mixts of H & Cl can form at the mercury cathode. The remedy consisted of operating at current densities sufficiently low to allow diffusion of Na into the Hg cathode

Refs: 1)Mellor **2**(1922), 521-61 2)Gmelin, Syst Nr **21**(1928), 305-73 3)WASAG, GerP 639404(1936) & CA **31**, 1616(1937) 4)Mellor (1939), 562-65 5)Davis(1943), 325 & 351-53 6)Hackh's(1944), 780 7)Thorpe **10**(1950), 848-49 8)J. Taylor, BritP 682209(1952), & CA **48**, 6700(1954) 9)Kirk & Othmer **12** (1954), 67-82 10)M. Dodero & M. Behroun, Proc 6th Meeting Intern CommElectrochem-Thermodynam and Kinet **1955**, 290-3 & CA **50**, 9907(1956) 11)Sax(1957), 1114-15 11a)PATR **2510**(1958), p Ger 44 12)N.E. Hanna et al, US Bur Mines RI **5463**(1959)(Effect of small amts of NaCl on the incendivity of permissible expls) 13)D.W. Kaufmann, Edit, "Sodium Chloride", Reinhold, NY(1960)(ACS Monograph 145) 14)CondChemDict(1961), 1037-38

Chlorinated Hydrocarbons & Other Chlorinated Compounds. The substitution of chlorine atom for hydrogen in a compd greatly increases the anesthetic action of the derivative. In addn, the chlorine deriv is less specific than the parent hydrocarbon in its action, and may affect other tissues along with those of the central nervous system of the body. The chlorine deriv is generally quite toxic and may cause liver, heart & kidney damage. As a rule, unsaturated chlorine derivs are highly narcotic but less toxic than saturated derivs. Sax(Ref 4) has discussed in detail the toxicities & hazards of a number of chlorinated compds, including *Chlorinated Diphenyls; Chlorinated Hydrocarbons, Aromatic & Aliphatic; Chlorinated Naphthalenes; Chlorinated Phenols; Chlorinated Triphenyls &* others.

Clogston(Ref 2) reported that a bomb contg a mixt of Al & carbon tetrachloride(CCl_4) exploded violently on heating for 53 min at 152°. The expl decompn of several chlorinated methyl-siloxanes & chlorinated methylchlorosilanes when heated to ca 200° was reported by Zimmermann(Ref 3). A previously undescribed exothermic reaction of chlorinated rubber with zinc oxide was held responsible for an expln that leveled themanufg area of Dayton Chemical Products Laboratories(Ohio) in April 1962(Ref 5)

The use of a w-insol, chlorinated hydrocarbon (such as chloronaphthalene or chloro-p-cymol)

and a resinous material(such as rosin or tar -distn residue) was patented by Matter(Ref 1) as thickening agent for the nitrate, chlorate or perchlorate of an expl mixt

See also Chlorates, Chlorides, Chlorine, Chlorites, and individual chloro compds

Refs: 1)O. Matter, Swiss P228653(1943) & CA **43**, 2437(1949) 2)C.C. Clogston, Underwriters' LabBullResNo **34**, 5-15(1945) & CA **40**, 209-10(1946) 3)W. Zimmermann, ChemBer **87**, 887-91(1954) & CA **49**, 9496(1955) 4)Sax(1957), 457-85 5)Anon, C & EN **40**, No 37, 79(10 Sept 1962)

CHLORINE AND COMPOUNDS

Chlorine(Chlor in Ger), Cl_2, mw 70.91, greenish-yel, poisonous gas having an irritating & suffocating-pungent odor, readily liquefied, fr p -101°, bp -34.6°, d(liq) 1.47 at 0° & 3.65 atms, vap press 4800 at 20°, vap d 2.49; sol in w or CCl_4. Chlorine is the most abundant halogen, occurring as chlorides(in sea water & salt deposits), in many minerals, and in all vegetable & animal tissues. It was first isolated in 1774 by Scheele(cited in Refs 1, 2, 3, 4 & 5) by the action of manganese dioxide on HCl. It is manufd today either by oxidn of HCl or by electrolysis of NaCl(Refs 3, 6 & 8) Chlorine consists of two stable isotopes: Cl^{35} & Cl^{37}, which occur in 75.4 & 24.6% respectively, hence giving an average at wt of 35.457. Sax(Ref 9) discusses in detail the hazard & toxicity of chlorine: the MAC is 1ppm in air or 2.9mg per cubic meter of air; concns of 50 ppm are dangerous & 1000 ppm may be fatal, even for brief exposure

Chlorine is mono-, tri- or penta-valent; hence, it gives the following series of compds: *chlorides* (Cl^- as in NaCl), *hypochlorites*(ClO^- as in NaOCl), *chlorites*(ClO_2^- as in $NaClO_2$), *chlorates*(ClO_3^- as in $NaClO_3$), and *perchlorates* ClO_4^- as in $NaClO_4$). The interaction of Cl_2 & H_2 has been the subject of a great deal of research. In sunlight or when exposed to the light of burning Mg, or at 250°, equal volumes of Cl_2 & H_2 combine with expl violence forming HCl. The expl limits of H-Cl mixts have been detd by Umland(Ref 7). All metals, under suitable conditions, can combine directly with chlorine forming the stable chlorides. Ammonia or ammonium salts in acidic soln react with chlorine producing a highly expl oil, *Nitrogen Chloride or Nitrogen Trichloride*(See under CHLORIDES)

Moist chlorine or *chlorine water* is a powerful oxidg agent and will bleach many org coloring matters. Chlorine is used as a bleaching agent for textiles, straw & sponges; as a disinfectant, germicide & insecticide; as a poison gas; and is particularly important in the synthetic-organic-chemical industry (See Ref 10)

(See also CL under CHEMICAL AGENTS)

Refs: 1)Mellor **2**(1922), 15ff 2)Gmelin, Syst Nr **6**(1927), 1-183 3)Mellor(1939), 489-99 4)Thorpe **3**(1939), 36-81 5)Hackh's(1944), 193 6)Kirk & Othmer **1**(1947), 358 & **3**(1949), 677ff 7)A.W. Umland, JElectrochemSoc **101**, 626-31 (1954) & CA **50**, 1315-16(1956) 8)Faith, Keyes & Clark(1957), 257-64 9)Sax(1957), 463-64 10)J.S. Sconce, Edit, "Chlorine: Its Manufacture, Properties and Uses," Reinhold, NY(1962)(ACS Monograph 154)

List of Chlorine Compounds

Chlorine Acetate, $CH_3 \cdot COOCl$; mw 94.49, exists only in soln. This dk-red soln is unstable and frequently explodes when warmed from -70 to -20°; it contains both chlorine monoxide (Cl_2O) & chlorine acetate. It was prepd by MacKenzie et al(Ref 2) according to the method of Schützenberger by addg ClO_2 to acetic anhydride. This soln cannot be distilled without expln and is reported likely to explode even without warming it

Refs: 1)Beil **2**, 170 &{318} 2)J.C. MacKenzie et al, CanJRes **26B**, 141, 149(1948) & CA **42**, 4919(1948)

Chlorine Azide or Chloroazide. See Vol 1, p A529-L

Chlorine Dioxide. See under Chlorine Oxides

Chlorine Fluorides. Although in 1891, H. Moissan (cited in Ref 2) easily prepd bromine & iodine fluorides by direct action of the corresponding elements, he claimed that it was impossible to obt any chlorine fluoride. Other investigators among them Lebeau(1906, cited in Ref 2) and Ruff & Zedner(1909, cited in Ref 2) also tried, but failed. It was not until 1928 that Ruff et al (Ref 3) succeeded in prepg the monocompd: *Chlorine Monofluoride,* ClF, mw 54.46; colorless gas, fr p -154, bp 100.8, d 1.62 at -100°; critical temp -14°, Q evapn 2.27 kcal/mol; was prepd by action of sl moist chlorine on fluorine at RT; if the gases are dry they do

not react below 250°. Fredenhagen & Krefft (Ref 5) found that if a mixt of fluorine & chlorine is sparked, in the presence of moisture, an expln takes place producing ClF. Chlorine monofluoride is very reactive, acting on some metals even more vigorously than fluorine itself(Refs 4, 7, 8 & 11)

Chlorine Trifluoride, ClF_3, mw 92.46; colorless gas, fr p -83°, bp 11.3°, d 1.77 at 13°, vap d 3.14; MAC 0.1 ppm of air(Ref 11); reacts vigorously with w, making a noise like the crack of a whip(Ref 9). It was prepd in 1930 by Ruff & Krug(Ref 6) from a mixt of chlorine or ClF & excess fluorine passing thru a tube heated to 250°. It is an extremely reactive compd; attacking glass, igniting spontaneously, and reacting violently & sometimes explosively in contact with org materials, metals or oxidizable materials(Refs 11 & 12). It appears that the Germans may have used ClF_3 during WWII for military purposes

Refs: 1)Mellor **2**(1922), 113 2)Gmelin, Syst Nr **6**(1927), 442 3)O. Ruff et al, ZAng-Chem **41**, 1289(1928) & **42**, 807(1929) 4)O. Ruff et al, ZAnorgChem **176**, 258(1928); **183**, 214(1929); **198**, 375(1931) & **214**, 82(1933) 5)K. Fredenhagen & O.T. Krefft, ZPhysChem **141**, 221(1929) 6)O. Ruff et al, ZAnorgChem **190**, 270(1930) & **207**, 46(1932) 7)Mellor(1939), 534 8)Partington(1950), 341 9)Sidgwick, ChemElems **2**(1950), 1148 & 1156 10)Kirk & Othmer **6**(1951), 694 11)Sax(1957), 465 & 466 12)CondChemDict(1961), 255 13)North American Aviation Inc, Rocketdyne Div Rpt **R-3136**, "Chlorine Trifluoride Handling Manual"(Sept. 1961) 14)H.Allen, Jr & M.L. Pinns, "Relative Ignitability of Typical Solid Propellants with Chlorine Trifluoride", NASA Tech Note **D-1533**(Jan 1963)

Chlorine Heptoxide. See under Chlorine Oxides

Chlorine Hexoxide. See under Chlorine Oxides

Chlorine Monofluoride. See under Chlorine Fluorides

Chlorine Monoxides. See under Chlorine Oxides

Chlorine Oxides. The oxides of chlorine are all endothermal compds, i.e, they are formed with absorption of energy and are so unstable that they readily decomp spontaneously with evolution of heat, sometimes with expl violence. None of the oxides can be prepd by the direct union of chlorine & oxygen, but several have been prepd indirectly. They are listed below in order of increasing oxygen content:

Chlorine Monoxides, Cl_2O; mw 86.91, O 18.41%; pale orn-yel gas with a slgreenish tinge, condenses to a red-brn liq, fr p -20°, bp 2.2°, d 3.89 g/liter at 0°; readily sol in w, forming a golden-yel soln hypochlorous acid (HClO). It was discovered in 1834 by Balard(cited in Refs 1, 4 & 12); can be prepd by passing dry chlorine gas over ppt mercuric oxide. Other methods of prepn are given in Ref 2 & 10. The gas explodes readily on heating and the liq may explode on scratching a tube cont it, or by pouring from one vessel to another; in contact with paper, org matter, sulfur, phosphorus, potassium or carbonaceous compds expl reactions may result(Ref 1, p 240; Ref 4, p 227; Ref 5; Ref 7, p 506; Ref 8; Ref 10, p 678; Ref 11; and Ref 12, p 265)

Chlorine Dioxide(Chlorine Peroxide), ClO_2; mw 67.45, O 47.44%; reddish-yel gas, condenses to a dk-red liq, fr p -59°(solidifies to orn-red crysts), bp 11°, d 3.09 g/liter at 11°, vap d 2.32; sol in w forming a yel soln of chlorous acid ($HClO_2$). It was discovered in 1815 by Davy(cited in Refs 7 & 12) in the products of reaction between $KClO_3$ & concd H_2SO_4; can be also prepd by action of oxalic acid ($H_2C_2O_4$) on $KClO_3$, or by action of chlorine gas on sodium chlorite($NaClO_2$)(Ref 10). The liq & solid ClO_2 are slowly decompd by light; and in any form can explode spontaneously & violently, particularly in the presence of org matter(Refs 5, 7, 10, 12, 13, 14, 15, 16, 17 & 18). Chlorine dioxide was first used as a lab reagent; industrial application has been made in the bleaching of cellulosic materials such as pulp, paper & flour; and in a number of other uses given in Kirk & Othmer (Ref 10, pp 704-5)

Chlorine Hexoxide(Dichlorine Hexoxide), Cl_2O_6; mw 166.91, O 56.50%; red-brn oily liq, fr p -1°, d 1.65; dissolves unchanged in water. It was first discovered in 1843 by Millon, but overlooked until rediscovered in 1925 by Bodenstein(cited in Ref 7) on exposing a mixt of ClO_2 & ozone to light. Kalina & Spinks(Ref 6) on reexamination of its props warned that Cl_2O_6 is a dangerous compd and must be handled with care. It explodes violently in contact with org matter, and also by heat

of impact(Ref 4,p 247; Ref 7; and Ref 12, p 276)
Chlorine Heptoxide(Perchloric Anhydride), Cl_2O_7; mw 182.91, O 61.24%; col volatile oil, fr p -91.5°, bp 82° (explodes), vap press 100mm at 29°; reacts with w, forming perchloric acid($HClO_4$); toxicity & expln hazards are given in Sax(Ref 18). It was prepd in 1900 by Michael & Conn(cited in Refs 4 & 12) by dehydrating anhyd perchloric acid with P_2O_5 cooled to -10°. According to Meyer & Kessler (cited in Refs 4, 7 & 12), a safer method is to heat $KClO_4$ with chlorosulfonic acid, ($HO{\cdot}SO_2{\cdot}Cl$), under reduced pressure. This compd decomp on prolonged storage; it explodes violently when struck or heated; but it does not explode in contact with org materials, or sulfur or phosphorus(Refs 4, 7 & 12)
Chlorine Tetroxide, Cl_2O_8; mw 198.91, O 64.35%; was claimed to have been prepd by Gomberg(Ref 3) in ethereal soln by treating anhyd $AgClO_4$ with iodine. Its ethereal soln is colorless & does not volatilize with ether vapors; it remains unchanged when stored in ethereal soln for several days. The apparatus & detailed description of its prepn are given in Ref 3. No confirmation of this substance has been reported(See Refs 4 & 7). Sax(Ref 18) gives its fire & expln hazards.
Refs: 1)Mellor **2**(1922), pp 240, 286 & 380 2)W.A.Noyes & T.A. Wilson, JACS **44**, 1635 (1922) 3)M.Gomberg, JACS **45**, 398-421(1923) 4)Gmelin, Syst Nr **6**(1927), pp 227, 234 & 247 -49 5)J.W.T. Spinks & H.Taube, JACS **59**, 1155-56(1937)(Photochemical decompn of ClO_2 in CCl_4) 6)M.H. Kalina & J.W.T. Spinks, CanJRes **16B**, 381-89(1938)(Explosive props of Cl_2O_6) 7)Mellor(1939), 506-8 8)F. Taradoire, Bull Fr **8**, 860-64(1941) & **9**, 615-20(1942); CA **37**, 5669(1943) & **38**, 525 (1944)(Contact of ClO_2 with S may produce explns) 9)Hackh's(1944), 193 10)Kirk & Othmer **3**(1949), pp 678, 679, 696-99 & 702-7 11)Z.G.Szabó, MagyarKémFolyóirat **56**, 109-11 (1950) & CA **45**, 862(1951)(Decompn of Cl_2O as a characteristic expln process) 12)Partington(1950), pp 265, 270, 274 & 276 13)A.E. Cameron & A.V.Faloon, C & EN **29**, 3196(1951) (Expln of liq ClO_2) 14)R.F.Stedman, C & EN **29**, 5030(1951)(Expln of ClO_2) 15)W.D. Harrison, SouthernPulpPaperMfr **16**, No 5, 32, 34, 36(1953) & CA **47**, 7213(1953)(Prepn, toxicity & expln hazards of ClO_2) 16)F. Casciani, PaperTradeJ **136**, No 10, 21-5(1953) & CA **47**, 7123(1953)(Uses of ClO_2 in pulp & paper bleaching) 17)R.I.Derby & W.S. Hutchinson, InorgSynth **4**(1953), 152-8(Lab prepn of ClO_2) 18)Sax(1957), 465 & 466

Chlorine Trifluoride. See under Chlorine Fluorides

CHLORITES

Chlorites. Salts of chlorous acid, ($HClO_2$), are called chlorites. According to Kirk & Othmer(Ref 7), several chlorites have been prepd in the lab but only Na chlorite, ($NaClO_2$), has been produced in coml quantities

Chlorites do not explode on impact if the metal surfaces are carefully cleaned and org matter is not present; the film of oxide normally occurring on a hammer is sometimes sufficient to cause deton. In other respects chlorites should be handled with the same precautions reqd for hypochlorites or chlorates(qv). A chlorite soln should be removed from any surface at once by thorough washing and should not be allowed to dry on a fabric as this combination is very flammable. Also strong acids should not come in contact with chlorites as the chlorine dioxide(qv) evolved is very expl. In addn, strong chlorite solns react violently with sulfur so that a rubber stopper should not be used in a bottle contg chlorite solns(Ref 7)

Chlorites have been used to pulp wood & other fibrous materials. The yield of pulp is high, because not only is cellulose conserved, but also the hemicelluloses remain in the pulp. Their presence gives unusual props to a pulp which becomes ready for making into paper with less mechanical treatment. An important use of Na chlorite is in the prepn of chlorine dioxide for direct use(See Refs)

Analysis: When it is known that a chlorite is the only active oxidg agent present, it is easily determined by titration of the iodine liberated from KI in an acidified soln. A chlorite does not liberate iodine until the soln is acidified; this serves as a qualitative distinction from a hypochlorite. The quantitative analysis of a soln contg chlorite & hypochlorite requires detn of the total oxidg power. Hypochlorite is detd on a separate sample by addg an excess of Na arsenite, after making certain the soln is alk. This soln is saturated with

$NaHCO_3$ and titrated in KI using starch as indicator. This gives the hypochlorite; the chlorite is obtd by difference. Chlorates do not interfere with these analyses but the presence of other oxidg & reducing agents may(Ref 7)(See also Ref 2, pp 306-7; Refs 4a & 10)

Refs: 1)Mellor **2**(1922), 283 2)Gmelin, Syst Nr **6**(1927), 298ff 3)G.R.Levi, Atti-CongrNazlChimPuraApplicata **1936,** Part 1,pp 382-86 4)Mellor(1939), 512 4a)Scott & Furman(1939), 272 & 276 5)J.F.White et al, IEC **34**, 782-92(1942)(Chemistry of Chlorites) 6)Hackh's(1944), 193 7)Kirk & Othmer **3** (1949), 696-701 8)Sidgwick, ChemElems **2** (1950), 1224-25 9)Partington(1950), 272 10)Scott & Furman(1962), 325 & 337

List of Chlorites

Ammonium Chlorite, NH_4ClO_2, pale-yel, transparent crysts, mp dec rapidly on heating. According to Mellor(Ref 1), this compd was first obtd in soln by N.Millon(1843) on saturating an aq soln of chlorous acid with ammonia. Levi(Ref 2) prepd the compd in the form of delq ndls which exploded on percussion on an anvil

Refs; 1)Mellor **2**(1922), 284 2)G.R.Levi, Gazz **52** I, 207(1922) & JCS **122** I, 527(1922) 3)Gmelin, Syst Nr **6**(1927), 300(Gives a table listing solid chlorites)

Barium Chlorite, $Ba(ClO_2)_2$; was prepd in soln by action of nascent $HClO_2$ on the basic carbonate. The salt could not be separated from soln because of its instability

Refs: 1)Mellor(1939), 512 2)Partington (1950), 272 3)G.R.Levi & R.Curti, Ricerca-Sci **23,** 1798-801(1953) & CA **48,** 13509(1954) 4)C.B.Riolo, AnnChim(Rome) **43,** 564-7(1953) & CA **49,** 6014(1955) 5)R.Curti & E. Montaldi, Gazz **83,** 748-53(1953) & CA **49,** 6554-55(1955)

Calcium Chlorite, $CaClO_2$, wh crysts, dec completely in contact with a hot wire; was first prepd in soln by Millon(1843) from $Ca(OH)_2$ & aq $HClO_2$ (Ref 1), and later by Levi(Ref 2) from Ca peroxide in w & chlorine dioxide. The salt exploded on percussion

Refs: 1)Mellor **2**(1922), 284 2)G.R.Levi, Gazz **52** I, 417(1922) & JCS **122** II, 567(1922)

Chloropentamminecobalt(III) Chlorite. See Compd Co 20, Table D, under Ammines, Vol 1, p A280

Cupric Chlorite, $Cu(ClO_2)_2$, yel-brn ppt, dec in a dry state & explodes violently on percussion; was prepd by mixing molar solns of $CuSO_4$ & $Ba(ClO_2)_2$, separating the basic Cu chlorite and treating the filtrate with 9 vols of abs alc. The *Basic Cupric Chlorite,* $[Cu(ClO_2)_2.3Cu(OH)_2]$, does not explode by percussion; the *Potassium Cupric Chlorite,* $[Cu(ClO_2)_2.2KClO_2.2H_2O]$, obtd by addg concd KCl soln to a soln of $Cu(ClO_2)_2$ is an impact-sensitive expl salt

Ref: G.R.Levi & C. Cipollone, Gazz **53,** 200-3(1923) & CA **17,** 2999(1923)

Hexamminecobalt(III) Chlorite. See Comp Co 24, Table D, under Ammines, Vol 1, p 280

Hydrazine Chlorite, $N_2H_4.HClO_2$, spontaneously flammable when dry; was prepd by suspending $N_2H_4.H_2SO_4$ in a little H_2O contg $Ba(OH)_2$, addg $Ba(ClO_2)_2$ at 0-2°, filtering rapidly, and pouring at once into alc at -10°

Refs: G.R.Levi, Gazz **53,** 105-8(1923) & CA **17,** 2264(1923)

Lead Chlorite, $Pb(ClO_2)_2$, monocl yel crysts, mp explodes at 100°; can be pptd when a soln of Na chlorite is acidified with acetic acid and treated with Pb nitrate. Lead chlorite mixed with sugar detonates violently on percussion, and such a mixt has been used in detonators (Ref 5)

Refs: 1)Mellor **2**(1922), 283 2)G.R. Levi, AttiAccadLincei, **32,** 623-6(1923); JCS **124** II, 767-8(1923) & CA **18,** 326(1924) 3)Mellor (1939), 512 4)T.Dupuis & C.Duval, CR **229,** 51-3(1949) & CA **43,** 8971(1949) 5)Partington(1950), 272 6)Sax(1957), 817

Mercury Chlorites. *Mercurous,* $HgClO_2$, canary-yel compd, unstable & decompg on percussion; was prepd by addg $HgNO_3$ to a concd soln of an alkali or alk earth chlorite, filtering & rapidly washing the ppt with alc(Refs 1 & 2). *Mercuric,* $Hg(ClO_2)_2$, red ppt; explodes violently when dry, by percussion; dec without dissolving in warm w. Using $Hg(NO_3)_2$ and a similar method, the mercuric salt was prepd(Refs 1 & 2)

Refs: 1)Mellor **2**(1922), 224 2)G.R. Levi, Gazz **53,** 245-9(1923) & CA **17,** 3000(1923)

Nickel Chlorite, $Ni(ClO_2)_2.2H_2O$, does not explode easily on percussion, but does so when heated on a w-bath; was prepd by mixing equiv amts of $Ba(ClO_2)_2$ & $NiSO_4$,

filtering and fractionally pptg with abs alc
Ref: G.R. Levi, Gazz **53**, 245-9(1923) & CA **17**, 3000(1923)

Potassium Chlorite, $KClO_2$, col delq crysts, mp dec violently on heating with evolution of flame & heat; was first prepd by Millon(1843) by addg chlorous acid to a soln of KOH and evapg(Ref 2). Bruni & Levi(Ref 1) prepd the salt by reducing a soln of $KClO_3$ to chlorite with oxalic acid
Refs: 1)G. Bruni & G.R. Levi, Gazz **45** II, 177(1915) & JCS **110** II, 27(1916) 2)Mellor **2**(1922), 283

Silver Chlorite, $AgClO_2$, yel crysts, detonates on heating; was pptd when an acidified soln of alk chlorite was treated with Ag nitrate
Refs: 1)Mellor **2**(1922), 283 2)G.R. Levi, AttiAccadLincei **32**, 623-6(1923); JCS **124** II, 767-8(1923) & CA **18**, 326(1924) 3)Partington(1950), 272

Sodium Chlorite, $NaClO_2$, wh crysts, exists in anhyd & tri-hydrated forms, mp 175-200° (decomp, temp dependent upon moisture content). The solubility of $NaClO_2$ is reported by Taylor et al(Ref 1). Commercial production of $NaClO_2$ in the US is based upon the reduction of $Ca(ClO_3)_2$ by HCl. The chlorate is reduced to chlorine dioxide, and the hydrochloric acid is oxidized to chlorine:

$$Ca(ClO_3)_2+4\,HCl \longrightarrow 2ClO_2+Cl_2+CaCl_2+2H_2O$$

The chlorine dioxide is passed into a soln of NaOH contg carbonaceous matter & lime:

$$4\,ClO_2+4\,NaOH+Ca(OH)_2+C \longrightarrow 4\,NaClO_2+CaCO_3+3H_2O$$

The ppt of $CaCO_3$ is removed by filtration, the soln of $NaClO_2$ is concd by evapn & drum dried(Refs 2 & 3)

The lab prepn of $NaClO_2.H_2O$ is conducted by treating Na sulfate with Ba chlorite

According to Sax(Ref 4), Na chlorite is virtually nontoxic; it is a powerful oxidg agent ignited by friction, heat & shock; and can react vigorously on contact with reducing materials
Refs: 1)M.C. Taylor et al, IEC **32**, 899-903 (1940) 2)Kirk & Othmer **3**(1949), 697-701 3)Shreve(1956), 311 4)Sax(1957), 1115

Strontium Chlorite, $Sr(ClO_2)_2$, wh crysts, dec with evolution of heat when touched with a hot wire; was first prepd by Millon(1843) by slowly addg $Sr(OH)_2$ to aq chlorous acid(Ref 1). Levi (Ref 2) prepd the compd by action of strontium peroxide on chlorine dioxide. The salt exploded on percussion
Refs: 1)Mellor **2**(1922), 283 2)G.R. Levi, Gazz **52** II, 417(1922) & JCS **122** II, 567 (1922).

Tetramethylammonium Chlorite, $N(CH_3)_4ClO_2$, col delq crysts; forms neutral aq solns; was prepd by Levi(Ref). This compd explodes on percussion
Ref: G.R. Levi, Gazz **52** I, 207(1922) & JCS **122** I, 528(1922)

Thallous Chlorite, $TlClO_2$, col crysts, dec slowly at RT & very rapidly at 70-80°; sol in w; was prepd by Levi(Ref) from barium chlorite & thallous sulfate in the same manner as in the prepn of $NaClO_2$. This salt explodes on percussion
Ref: G.R. Levi, Gazz **52** I, 417(1922) & JCS **122** II, 567(1922)

Zinc Chlorite, $Zn(ClO_2)_2.2H_2O$, grn-yel crysts; was prepd by Levi & Cipollone(Ref). This salt was not exploded by percussion
Ref: G.R. Levi & C. Cipollone, Gazz **53**, 200(1923) & JCS **124** II, 492(1923)

Chlor-knallgas. Ger name for an expl mixt consisting of equal vols of chlorine & hydrogen. This mixt, first examined by Gay-Lussac & Thénard before 1811, explodes on heating, by flame, or by elec spark
Ref: Gmelin, Syst Nr **6**(1927), 86

Chloroacetophenone. See CN under Chemical Agents

Chloroacetylene(Chloroethine or Chloroethyne), CH:C.Cl; mw 60.49, col gas, fr p -30.2°; ignites on contact with air, explodes violently when mixed with air; other props & methods of prepn are given in Beil(Ref 1)

When reacted with ammoniacal solns of Cu & Ag, it forms expl salts(Ref 2). Other expl salts of chloroacetylene include: *Mercuric Chloroacetylene,* $Hg(C{:}Cl)_2$, pltlts (from chlf), dec at 160°, explodes violently at 170°+; and *Silver Chloroacetylene,* Ag.C:Cl col ppt, becoming rapidly brn on exposure to light, explodes violently even when wet(Ref 2)
Refs: 1)Beil **1**, 244, (106), [221] &{917} 2)K. Hofmann & H. Kirmreuther, Ber **42**, 4234-36(1909) 3)L.A. Bashford et al, JCS **1938**, 1358-64 & CA **33**, 5131(1939)(Oxidation of

chloroacetylene & bromoacetylene) 4)A. Roedig, Ann **569**, 161-83(1950) & CA **45**, 2419(1951)(Describes a Mercurous Salt, ClC:CHg, exploding above 183°) 5)Sax (1957), 237

Chloroacetyl Peroxide. See Dichloroacetyl Peroxide

Chloroaniline and Derivatives

Chloroaniline, C_6H_6ClN. Its three isomers of the formula $Cl.C_6H_4.NH_2$ are described in Ref 1. There is also an isomer, $C_6H_5.NHCl$, called N-Chloroaniline or Phenylchloramine (Ref 2)

Refs: 1)Beil **12**, 597,602,607(297,300,304) & [314, 319, 322] 2)Beil **12**, [295]

Chloroazidoaniline, $Cl.C_6H_3(N_3)NH_2$ - not found in Beil or in CA thru 1956

Mononitrochloroanilines, $C_6H_5ClN_2O_2$. Nine isomers are described in Beil

Ref: Beil **12**, 619, 729, 730, 731, 732, 733, (355, 356, 357) & [396, 397, 398]

Dinitrochloroanilines, $C_6H_4ClN_3O_4$; mw 217.58, N 19.34%; Nitro-N 12.89%. Four isomers are described in Beil

Ref: Beil **12**, 730, 759, 760, (366, 367) & [414, 415, 416 & 417]

Trinitrochloroanilines, $C_6H_3ClN_4O_6$; mw 262.58, N 21.34%, Nitro-N 14.22%. One isomer, *2,4,6-Trinitro-3-chloroaniline, 3-chloropicrylamide,* or *3-chloropicramide,* $Cl.C_6H(NO_2)_3.NH_2$, is described in the literature as brn-violet ndls(from alc), mp 185-86°; it was first prepd by Borsche&Feske(Ref 2) and then by De Monchy(Ref 3). Its expl props were not detd

Refs: 1)Beil **12**, [427] 2)W. Borsche & E. Feske, Ber **61B**, 697(1928) 3)M. de Monchy Rec **53**, 148(1934) & CA **28**, 5418(1934)

Tetranitrochloroaniline, $C_6H_2ClN_5O_8$ - not found in Beil or CA thru 1956

Chloroanilino-propanediol and Derivatives

Chloroanilino-propanediols or *Chlorophenylamino-propanediols,* $C_9H_{12}ClNO_2$. The 3-chloro isomer is mentioned in Ref 2. They may be considered as parent compds of the two dinitro derivs described below, although they were not used for their prepn

Refs: 1)Beil - not found 2)J.B. Dickey & E.B. Towne, USP 2683708(1954) & CA **49**, 1337(1954)

Dinitrochloroanilino-propanediols, $C_9H_{10}ClN_3O_6$; mw 291.66, N14.41%. Two isomers are known:

3'-(2,4-Dinitro-5-chloroanilino)propane-1'2'diol or *3-(2'4'-dinitro-5'-chlorophenylamino)propane-1,2-diol,* $Cl(O_2N)_2C_6H_2.NH.CH_2.CHOH.CH_2OH$; yel crysts, mp 90°; was obtained by heating under reflux for several hours an alcoholic soln of 3-aminopropane-1,2-diol with 2,4-dinitro-1,5-dichlorobenzene & Na acetate, filtering off the NaCl formed, and evapg the filtrate under vacuum(Ref 2, pp 20 & 22)

2'-(2,4-Dinitro-5-chloroanilino)propane-1,3-diol or *2-(2',4'-Dinitro-5'-chlorophenylamino)propane-1,3-diol,* $Cl(O_2N)_2C_6H_2.NH.CH(CH_2OH)_2$; yel crysts, mp 126-27°; was obtained by heating under reflux for several hrs an alc soln of 2-aminopropane-1,3-diol, with 2,4-dinitro-1,5-dichlorobenzene & Na acetate, filtering off the NaCl formed and evapg the filtrate under vacuum(Ref 2,pp 15-17 & Ref 3)

Trinitrochloroanilino-propanediol Dinitrates, $C_9H_6ClN_7O_{14}$, mw 471.56, N 20.78%. Two isomers are known:

3'-(2,4,6-Trinitro-5-chloronitranilino)propane-1',2'-diol Dinitrate or *3-(2',4',6'-Trinitro-5-chlorophenylnitramino)propane-1',2'-diol Dinitrate* (called 3-Chloro-N,2,4,6-tetranitro-anilino-propanediol Dinitrate in CA Decennial Index), $Cl(O_2N)_3C_6H.N(NO_2).CH_2.CH(ONO_2).CH_2(ONO_2)$; yel solid melting ca 50° to a very viscous liq; exploded with a loud report when struck with a hammer, or heated in a flame or in a sealed tube; insol in w, petr eth, chlf, CCl_4; sol in methanol, ethanol, ether, acetone, benz & NB. Was prepd by nitrating with fuming nitric acid in cold the 3'-(2,4-dinitro-5-chloroanilino)propane-1,2-diol(Ref 2, pp 20-22 & Ref 3)

2'-(2,4,6-Trinitro-5-chloronitranilino) propane-1',3'-diol Dinitrate or 2-(2',4',6'Trinitro-5'chlorophenylnitramino) propane-1,3-diol Dinitrate, $Cl(O_2N)_3C_6H.N(NO_2).CH(CH_2.ONO_2)_2$; lt yel substance melting ca 40° to a very viscous resinous liq; expl with a loud report when struck with a hammer or heated in a flame or sealed tube; insol in w, petr eth, ether, chlf, CCl_4, benz; sol in acetone, AcOH & NB. Was prepd by nitrating in cold with fuming nitric acid the 2'-(2,4-dinitro-5-chloroanilino) propane-1,3-diol (Ref 2,pp 15-17 & Ref 3)

Refs: 1)Beil - not found 2)H.P. den Otter, Rec **57**, 13-24(1938) 3)CA **32**, 3354(1938)
Note: Compare the above compds with Anilinopropanediol derivs described in Vol 1,p A434-R

Chloroanisole and Derivatives

Chloroanisole or Methylchlorophenyl Ether, $Cl.C_6H_4.O.CH_3$. Exists as o-, m- and p- isomers
Ref: Beil **6**, 184, 185, 186, (99, 100, 101) & [171, 173 & 175]

3-chloro-2-azido-anisole, $Cl.C_6H_3(N_3).O.CH_3$; mw 183.60, N 22.89%; lt yel prisms(from petr eth), mp 35°; volatile with steam; was prepd by treating 3-chloroanisole-2-diazonium perbromide with ammonia. Its expl props were not detd
Refs: 1)Beil **6**, [284] 2)H.H. Hodgson & A.Kershaw, JCS **1928**, 193 & CA **22**, 1338(1928)

Mononitrochloroanisole, $C_7H_6ClNO_3$. Seven isomers are described in literature
Ref: Beil **6**, 238, 239, 240, (122) & [226, 228, 229]

Dinitrochloroanisole, $C_7H_5ClN_2O_5$. Five isomers are described in the literature
Ref: Beil **6**, 259, 260, (128) & [247, 248]

Trinitrochloroanisole, $C_7H_4ClN_3O_7$, mw 305.60, N 15.14%. Two isomers are known:

2,4,6-Trinitro-3-chloroaniline or Methyl-(3-chloro-2,4,6-trinitro-phenyl-ether), $Cl.C_6H(NO_2)_3.O.CH_3$; col crysts(from alc), mp 86-88°; can be prepd by nitrating 3-chloronitroanisole with nitric-sulfuric acid or by other methods. Its expl props were not detd
Refs: 1)Beil **6**, 292 & [283] 2)J.J.Blanksma, Rec **21**, 323(1902) [Called Oxymethylchlorotrinitrobenzene(1.3.2.4.6.)] 3)H.H.Schlubach & F. Mergenthaler, Ber **58**, 2734(1925) & CA **20**, 1395(1926)

3,4,6-Trinitro-5-chloroanisole, orn-yel prisms (from alc), mp 128°; was prepd by nitrating 5-chloronitroanisole with nitric-sulfuric acid. Its expl props were not detd
Refs: 1)Beil - not found 2)H.H.Hodgson & W.E.Batty, JCS **1934**, 1434 & CA **29**, 133(1935)

Tetranitrochloroanisole, $C_7H_3ClN_4O_9$ - not found in Beil or CA thru 1956

Chloroazobenzene and Derivatives

Chloroazobenzene, $C_{12}H_9ClN_2$; mw 216.66, N 12.92%. Two isomers are described in Beil **16**, 35, 36 & [12, 13]

Chloromononitroazobenzene, $C_{12}H_8ClN_3O_2$; mw 261.66, N 16.05. Five isomers are described in Beil **16**, 51,54,57 & [17, 18]

Chlorodinitroazobenzene, $C_{12}H_7ClN_4O_4$; mw 306.67, N 18.26. Four isomers are described in Beil **16**, 57, 58 & [18]

Chlorotrinitroazobenzone, $C_{12}H_6ClN_5O_6$; mw 351.67, N 19.92. Six isomers are described in Beil **16**, 58, 59 & [18]

Chlorotetranitroazobenzene, $C_{12}H_5ClN_6O_8$; mw 396.67, N 21.19. The following isomers are described in the literature:

4'-Chloro-2,4,6,3'-tetranitro-azobenzene, $(O_2N)_3C_6H_2.N{:}N.C_6H_3(NO_2).Cl$; red-yel lfts (from AcOH), mp dec ca 184-5°; readily sol in alc, AcOH, chlf & benz; was prepd by boiling for 3-4 hrs 1 part 4'-chloro-2,4,6-trinitroazobenzene with 3 parts mixed acid(HNO_3-H_2SO_4)(Ref 1,p 59 & Ref 2); and

3'-Chloro,2,4,6,4'-tetranitro-azobenzene, yel lfts(from AcOH), mp dec 124-5°; readily sol in AcOH; less sol in alc, eth or chlf; was prepd by prolonged boiling of 3'-chloro-2,4,6-trinitroazobenzene with a large excess of mixed acid(Ref 1,p 60 & Ref 3). The expl props of tetra nitro derivs were not detd
Refs: 1)Beil **16**, 59,60 2)C.Willgerodt & A.Böhm, JPraktChem **43**, 488,(1891) & JCS **60** II, 906(1891) 3)C.Willgerodt & E.G. Mühe, JPraktChem **44**, 455(1891) & JCS **62** I, 454 (1892)
Note: Higher nitro derivs of chloroazobenzene were not found in Beil or in CA thru 1956

Chloroazodine. See Azobis-(chloroformamidine) Vol 1,p A652-R

Chlorobenzazide. See Chlorobenzoyl Azide under Chlorobenzoic Acid

Chlorobenzene and Derviatives

Chlorobenzene, Monochlorobenzene or Phenyl Chloride(called Chlorbenzol in Ger),.C_6H_5Cl, mw 112.56, clear, col liq, fr p -55.6°, bp 132°, flash p 85°F(CC), d 1.105 at 25°, vap press 10 mm at 22.2°, vap d 3.88, n_D1.5217 at 25°; sl sol in w; very sol in alc, eth, benz, chlf or CCl_4. It is toxic by inhalation, ingestion or absorption; the MAC is 347 mg/cu m of air or 75 ppm in air(Ref 6). Sax(Ref 6) gives some details as to toxicity & hazard, and reports the expl range 1.8% at 212°F to 9.6% at 302°F. Chlorobenzene can be prepd either by chlorin-

ation of benz in the liq phase with gaseous chlorine, or by vapor phase oxychlorination of benz with HCl & air(Ref 2). It has been used as a starting material in one process for making PA. Various other uses are given in Ref 7. On nitration it yields expl derivs

Technical grade chlorobenzene intended for use by US Dept of Defense as a solvent or as a raw material in the manuf of various chemicals, shall meet the following requirements: specific gravity 1.101-1.112 at 20/4°; acidity, as HCl 0.015%; moisture 0.1%; and distillation range 131-32° at 760 *mm Hg*. Tests are described in Ref 8

Refs: 1)Beil **5**, 199,(108) & [148] 2)Ullmann **2**(1928), 268 3)Pepin Lehalleur(1935), 166 4)Kirk & Othmer **3**(1949), 813-17 5)Ullmann **5**(1954), 461-66 6)Sax(1957) 7)CondChem-Dict(1961), 258 8)US Spec MIL-C-12038B (Tech grade chlorobenzene)

Chloroazidobenzenes(Called Chlorophenylazides by Maffei & Coda), $Cl.C_6H_4N_3$, mw 153.62, N 27.36%. All three isomers, colorless oils, are known. The *ortho-* compd, bp 58-60° at 1mm Hg is described in Refs 2 & 3; the *meta-*, bp 49-51° at 0.7 mm is in Ref 1; and the *para-*, bp 44-6° at 1 mm & 90° at 15 mm is in Refs 1, 2 & 3

Expl props of chloroazidobenzenes were not detd

(Compare with the expl compd, 1-Azido-2,4-dichloro deriv described in Vol 2, p B44-R, under Azido-Halogen Derivatives of Benzene)

Refs: 1)Beil **5**, 277 & [208] 2)H.O. Spauschus & J.M. Scott, JACS **73**, 208 & 210 (1951) & CA **45**, 7002(1951) 3)S. Maffei & L. Coda, Gazz **85**, 1301-02(1955) & CA **50**, 9330(1956)

Note: No nitro- or dinitro- derivs of chloroazidobenzenes were found in Beil or CA thru 1956

Mononitrochlorobenzene or Chloronitrobenzene, $Cl.C_6H_4.NO_2$, mw 157.56, N 8.89%. The o-(or 1,2), m-(or 1,3-) &p-(or 1,4-) nitrochloro derivs are described in the literature(Refs 1, 2, 3, 5 & 7) These comps are non-expl(Ref 6)

Refs: 1)Beil **5**, 241-45, (129-30) & [180-4] 2)Ullmann **2**(1928), 276 3)Pepin Lehalleur (1935), 166-67 4)P.Aubertein, MP **32**, 7-9 (1950)(Rapid method of detg small quantities chlorobenzene in mononitrochlorobenzene) 5)Kirk & Othmer **9**(1952), 393 6)Sax(1957), 476 7)Ullmann **12**(1960), 798

Dinitrochlorobenzene (DNCB) or Chlorodinitrobenzene, $Cl.C_6H_3(NO_2)_2$; mw 202.56, N 13.83%. Six isomers are described in Beil, two of which are important:

2,4-Dinitro-1-chlorobenzene or 1,3-Dinitro-4-chlorobenzene. Three polymorphs exist: orthorhmb crysts or alpha(stabile form), mp 53.4°, bp 315°, d 1.697 at 22°/4°; v sol in hot alc, eth, benz or CS_2; sl sol in alc; insol in w; can be prepd by nitration of chlorobenzene with mixed acid(Refs 1, 2a, 5, 9, & 10); rhmb crysts or beta(labile form), mp 43°, d 1.680 at 20°/4°; solubility same as alpha; can be prepd by rapid cooling of the alpha form(Ref 3); and gamma, mp 27°(Refs 1 & 10). Halpern(Ref 8) reported an expln occurred during distillation of 2,4-DNCB at 1 mm press

2,6'-Dinitro-1-chlorobenzene or 1,3-Dinitro-2-chlorobenzene, yel nds(from alc), mp 87.8°, d 1.6867 at 16.5°; sol in alc & eth; insol in w; forms in small quantities during heating of o-chloronitrobenz with mixed acid, or can be prepd from 2,6-benzenediazonium nitrate & $CuCl_2$(Refs 1, 2 & 5). A new method of prepg 2,6-DNCB is reported by Molard & Vaganay (Ref 11)

Commercial DNCB, yel crysts, mp ca 50°, consists mostly of 2,4-DNCB with a small amt of 2,6-DNCB. This product is considered more toxic than NB. It causes dermatitus & systemic poisoning(Ref 12). DNCB is an expl sl more powerful than DNB and comparable in sensitivity to TNT(Ref 4). The expl props of DNCB & its mixts with PA, TNT or AN were studied by Burlot & Tavernier(Ref 7). It has been reported that phosgene is produced on low order deton of DNCB(Ref 6). DNCB is used as an intermediate in one method for the manuf of tetryl. It may also be further nitrated to 2,4,6-Trinitro-1-chlorobenzene(abbreviated TNCB) and also known as Picryl Chloride(See below)

Refs: 1)Beil **5**, 262-64, (137-39) & [196-97] 2)G.Koerner & A. Contardi, AttiAccadLincei-Rend **23**, 464-71(1914) & SS **10**, 77(1915) 2a) Ullmann **2**(1928), 277-78 3)J. Mason, JCS **1931**, 3154-57 4)L.Wöhler & O.Wenzelberg, AngChem **46**, 173-76(1933) 5)Pepin Lehalleur (1935), 167 6)All & EnExpls(1946), 157 7)E.Burlot & P.Tavernier, MP **31**, 33-41(1949) & CA **46**, 11684-85(1952) 8)B.D.Halpern, C & EN **29**, 2666(1951) & CA **46**, 3279(1952)

9)I. Stojkovič, KemZbornik **1951**, 137-38 & CA **48**, 11369(1954) 10)Kirk & Othmer **9**(1952), 395 11)L.Molard & J.Vaganay, MP **39**, 111-21 (1957) & CA **52**, 19989(1958) 12)Sax(1957), 472 & 630 12a)Ullmann **12**(1960), 799 13)US Spec JAN-D-666(DNCB intended for use in manuf of dinitro- or trinitro- phenol, shall consist essentially of $Cl.C_6H_3(NO_2)_2$, be not darker than lemon yel in color and have setting point 50.0 ±3.0°, as detd in app described in Vol 1, p A613-L. 14)Dr. H. Walter, formerly of PicArsn (1963) [DNCB was originally manufd in Germany for the purpose of converting it to HNDPh, according to the reactions:

$Cl.C_6H_3(NO_2)_2 + C_6H_5.NH_2 \longrightarrow C_6H_5.NH.C_6H_3(NO_2)_2$

$\xrightarrow[H_2SO_4]{HNO_3} (O_2N)_3C_6H_2.NH.C_6H_2(NO_2)_3]$

Note: HNDPhA, known also as Hexa, hexamine or Hexyl was used by Ger Navy as an ingredient of expl fillers for torpedoes, sea mines, depth charges etc. [See also PATR **2510**(1958), p Ger 88]

Trinitrochlorobenzenes, $Cl.C_6H_2(NO_2)_3$; mw 247.56, N 16.98%, OB to CO_2 & Cl_2 -45.2%. Following isomers are described in the literature:

1,2,3-Trinitro-4-chlorobenzene, yel ndls(from alc), mp 68-9°; can be prepd by heating 1,2-dinitro-4-chlorobenzene with fuming nitric acid and oleum. Its expl props were not detd (Ref 1; Ref 2,p 613)

1,2,3-Trinitro-5-chlorobenzene, yel crysts(from benz), mp 168°; can be prepd by treating 2,6-dinitro-4-chloro-1-benzene diazonium nitrate with Cu sulfate and Na nitrite in w. Its expl props were not detd (Ref 1; Ref 2,p 608)

1,2,4-Trinitro-5-chlorobenzene, yel-crysts (from alc), mp 116°; can be prepd by treating 5-chloro-2,4-dinitro-1-benzenediazonium nitrate with Na nitrite in w or by other methods. Its expl props were not detd (Ref 1; Ref 2, p 609)

1,2,4-Trinitro-6-chlorobenzene, plates (from alc), mp,105-6°; can be prepd by heating 1,2-dinitro-6-chlorobenzene with fuming nitric acid and oleum. Its expl props were not detd (Ref 1; Ref 2, p 610)

Refs: 1)Beil **5**, [205] 2)P.G. Van de Vliet, Rec **43**, 608, 609, 610 & 613(1924)

2,4,6-Trinitro-1-chlorobenzene; 1,3,5-Trinitro-2-chlorobenzene(TNCB) or **Picryl Chloride**; monocl-prismatic crysts(from alc or ligroin); mp 83°, d 1.797 at 20°. Soly in 100g of solvent (Ref 1,p [205]): Acetone 212.0g at 17° & 546.4 at 50°; ethyl acetate 91.5 at 17° & 238.5 at 50°; toluene 89.4 at 17° & 321.1 at 50°; benzene 36.7 at 17° & 428.1 at 50°; chloroform 12.4 at 17° & 233.4 at 50°; methanol 10.2 at 17° & 34.8 at 50°; ethanol 4.8 at 17° & 15.1 at 50°; ether 7.13 at 17° & 10.64 at 31°; CCl_4 0.557 at 17° & 2.45 at 50° and CS_2 0.499 at 17° & 0.95 at 30.5°. It is an acid, which can be prepd by nitration with mixed acid of DNCB or of chlorobenzene; its lab prepn includes heating of PA with phosphorus pentachloride or other methods(Refs 1, 4, 5, 8, 10 & 13). TNCB is readily hydrolyzed to form PA and, similarly to PA, it attacks metals to form expl salts, some of which are very sensitive(Ref 8). Its chloride salt, called *Chloropicryl Chloride*, was prepd without danger of expln(Ref 10). Some of its additive compds are also expl. It is very toxic causing dermatitis(Ref 5 & 11)

TNCB is an expl sl more powerful than TNT and comparable to it in sensitivity. Its expl props have been detd as follows(Refs 3,6,7, 8 & 9): *Brisance* by Copper Compression Test, 114% TNT; *Brisance Value* by calculation(Kast) 128% TNT; *Heat of Combustion*, 643 kcal/mole; *Impact Sensitivity* with 2 kg wt 99% TNT & FI 111-27% PA; *Power* by Trauzl Test 97-102% PA &103 TNT; *Rate of Detonation* 6855 at d 1.70-71, 7130 at d 1.74-75, and 7347 m/sec at d 1.77; *Specific Volume* 620 l/kg; and *Temperature* developed on explosion (calcd) 3370°

Uses: TNCB was used in Germany as a HE filler for various ammunition. In compressed form it was known as Filler No 60, in cast form as Filler No 61 and in mixt with Al as Filler No 64 (Refs 9 & 12). It was used not only on account of shortage of other materials, but also because there existed equipment formerly used by Ger Navy for prepn of DNCB, which served for prepn of HNDPhA(See under Dinitrochlorobenzene). When the Navy stopped using HNDPhA (replacing it with RDX compns), the equipment for manufg DNCB became available and was taken by Ger Army for manuf of TNCB, which is easily obtained by nitrating DNCB (This info was supplied by Dr. H. Walter, formerly of

PicArsn)

TNCB was patented in US for use as a filler of detonating fuses(Ref 2)

Refs: 1)Beil **5**, 273, (140) & [205] 2)E. Burkard, USP 1049655(1913) & CA **7**, 893(1913) 3)B.J. Flürsheim, JSCI **40**, 103T(1921) 4)P.G. Van de Vliet, Rec **43**, 607(1924) 5)Ullmann **2** (1928), 278 6)L. Wöhler & O. Wenzelberg, AngChem **46**, 173-76(1933) 7)A.H. Blatt & F.C. Whitmore, OSRD **1085**(1942), 103 8)Blatt, OSRD **2014**(1944)(Picryl Chloride) 9)All & En Expls(1946), 113 10)H. Feuer & A.A. Harban, USP 2679538(1954) & CA **49**, 4715(1955)(Prepn of TNCB chloride in good yield by passing $COCl_2$ into a soln of the pyridine salt of styphnic acid in acetone) 11)Sax(1957), 948 & 1223 12)PATR **2510** (1958), pp Ger 48 & 204 13)Ullmann **12** (1960), 800

Trinitrochlorobenzene, Analytical Procedures. Accdg to Dr. Hans Walter, formerly of PicArsn, the following procedure was used in Germany:

A weighed sample of TNCB is dissolved in methanol and refluxed for two hours with a measured vol of 0.1N NaOH soln (two equivalents of NaOH, plus a slight excess). The following reactions take place:

$Cl.C_6H_2(NO_2)_3+NaOH=NaCl+HO.C_6H_2(NO_2)_3$

$HO.C_6H_2(NO_2)_3+NaOH=H_2O+NaO.C_6H_2(NO_2)_3$

Then the soln contg Na picrate, NaCl and an excess of NaOH is titrated potentiometrically with 0.1N HCl until the 1st potentiometric break in the curve is observed. Burette reading would indicate the vol(V_1), required to neutralize the excess NaOH. Titration is continued until the 2nd break in the curve is observed. This burette reading(V_2), minus(V_1) indicates the vol(V_3), required to convert Na picrate back to PA

Assuming that the normality of HCl soln is exactly the same as that of the NaOH soln, and if the vol of NaOH soln(added before refluxing) is V, then vol(V_4), required for converting TNCB to PA, can be calcd from the equation.

$$V_4 = V - V_2$$

If TNCB is free of PA, then the vol (V_3) would be equal to (V_4), but as tech grade TNCB usually contains some PA(formed as result of hydrolysis), V_3 would be larger than V_4

If any DNCB is present, the sum of amts V_3 and V_4 would be proportionally greater than for TNCB not containing DNCB. The amt of DNCB present in TNCB can be approx calcd as described below:

Assume that (V_3+V_4) for the sample is equal to V_1, while for compd consisting of pure TNCB it is V_3, and for pure DNCB V_2. If V_1 is larger than V_2 and smaller than V_3, then the percentage (x) of DNCB in sample can be calcd from the equation:

$$x = [(V_3 - V_1) \times 100] / (V_3 - V_2)$$

The amts can also be calcd using the equations derived by N. Gelber & N.Liszt in PicArsn, AnalSectnRept **AL-P-1-60**

It has been suggested by F. Pristera of PicArsn, to supplement the above analysis by detn of amt of nitro-nitrogen groups using standard titanous chloride reaction;

$$Cl.C_6H_2(NO_2)_3 \xrightarrow{18TiCl_3} Cl.C_6H_2(NH_2)_3$$

(This section was reviewed by N.Liszt and F.Pristera of PicArsn)

Tetranitrochlorobenzene, (TeNCB), $C_6HClN_4O_8$; mw 292.56, N 19.15%, mp 97°. Isomer not specified; probably it is 2,3,4,6-tetranitro-1-chlorobenzene; very reactive. Was claimed to be prepd before WWII in England, but its method of prepn is not described

Refs: 1)Beil - not found 2)A.H. Blatt & F.C. Whitmore, OSRD Rept **1085**(1942), 84 3)CA's 1907-1956 - not found

p-Chlorobenzeneazotrinitromethane, $Cl.C_6H_4.N{:}N.C(NO_2)_3$, mw 289.56, N 24.17; bright yel crysts, mp explodes at 68°; its method of prepn is described in Ref 2

Refs: 1)Beil - not found 2)A.Quilico, Gazz **62**, 912-27(1932) & CA **27**, 1348(1933)

Chlorobenzoic Acid and Derivatives

Chlorobenzoic Acid(called Chlor-benzoesäure in Ger), $Cl.C_6H_4COOH$; mw 156.57. The o-, m- & p-isomers are described in Beil **9**, 334, 337, 340, (138, 139, 140) & [221, 223, 225]

Chlorobenzoic Acid Azide, Chlorobenzazide or Chlorobenzoyl Azide(called Chlor-benzazid in Ger), $Cl.C_6H_4.CO.N_3$; mw 181.58, N 23.14%. The following isomers are described in the literature:

3-Chloro or m- deriv, col oil of unpleasant odor, explodes when heated; sol in common org solvs; was prepd by diazotizing m-chlorobenzohydrazide in eth soln (Refs 1 & 2).

4-chloro or p- deriv(Ref 3) and *2-Chloro or o- deriv* (Ref 5) were subsequently prepd by action of $NaNO_2$ on the corresponding hydrazide in glacial AcOH. These azides have been used as reagents in identification of amines(Refs 3, 4 & 5). Their expl props were not detd

Refs: 1)Beil **9**, 339 2)T. Curtius & H. Foerster, JPraktChem **64**, 331(1901) & JCS **82** I, 58(1902) 3)Chen-Heng Kao et al, JChinese Chem Soc **3**, 137-40(1935) & CA **29**, 6172-73 (1935) 4)P.P. T. Sah & Chung-Shu Wu, JChinese ChemSoc **4**, 513-17(1936) & CA **31**, 3891(1937) 5)P.P.T.Sah et al, JChineseChem-Soc **13**, 22-76(1946) & CA **42**, 148(1948)

Monochlorobenzoic Acid Azide or Mononitro-chlorobenzoyl Azide, $Cl.C_6H_3(NO_2).CO.N_3$; not found in Beil or in CA thru 1956

Dinitrochlorobenzoic Acid Azide or Dinitro-chlorobenzoyl Azide, $Cl.C_6H_2(NO_2)_2.CO.N_3$; mw 271.59, N 25.78%. Two isomers are described in the literature:

3,5-Dinitro-2-chlorobenzoyl Azide, col crysts, mp 64°(crude product), explodes when heated on a w bath; slow decompn on recrystg from petroleum ether; was prepd by addg NaN_3 to 3,5-dinitro-2-chlorobenzoyl chloride in glacial AcOH(Ref 3); and

3,5-Dinitro-4-chlorobenzoyl Azide, ndls(from benz), mp 86°, decompd explosively by concd H_2SO_4; readily sol in AcOH, benz or NB; was prepd by action of NaN_3 on the appropriate acid chloride in AcOH(Refs 1 & 2)

Refs: 1)Beil **9**, [284] 2)H.Lindemann & W. Wessel, Ber **58**, 1221, 1225(1925) & CA **19**, 2824(1925) 3)J.J. Blanksma & G. Verberg, Rec **53**, 995(1934) & CA **29**, 4662(1935)

Note: Higher nitro derivs of Chlorobenzoyl Azide were not found in Beil or in CA thru 1956

Mononitrochlorobenzoic Acid, $Cl.C_6H_3(NO_2).COOH$; mw 201.56. All the known isomers are described in Beil **9**, 400-4,(165) & [274-76]

Dinitrochlorobenzoic Acid, $Cl.C_6H_2(NO_2)_2.COOH$; mw 246.57, N 11.36°. Two isomers are known:

3,5-Dinitro-2-chlorobenzoic Acid, col ndls(from w), mp 196-200°, bp 240-41°, dec ca 253°, explodes on heating to higher temp; other props & methods of prepn are given in Beil(Ref 1); and

3,5-Dinitro-4-chlorobenzoic Acid, col prisms (from benz), mp 159°, bp 240° at 12 mm Hg; readily sol in most org solvs; almost insol in petr eth; decomp explosively with evolution of flames, when heated with alkalies or BaO(Ref 3); other props & methods of prepn are given in Beil(Ref 2)

Refs: 1)Beil **9**, 415 & [284] 2)Beil **9**, 416 & [284] 3)H. Lindemann & W. Wessel, Ber **58**, 1223(1925) & CA **19**, 2824(1925),

Note: Higher nitro derivs of Chlorobenzoic Acid were not found in Beil or in CA thru 1956

Chlorobenzonitrile and Derivatives

Chlorobenzonitrile(called Chlor-benzonitril in Ger), $Cl.C_6H_4.CN$; mw 137.56, N 10.18%. Three isomers are known: 2-Chloro-, ndls, mp 42-3°, bp 232°, volatile with steam(Ref 1); 3-Chloro-, ndls, mp 39-40.5°, readily volatile with steam (Ref 2); and 4-Chlorobenzonitrile, ndls(from alc), mp 90-6°, bp 223° at 750 mm Hg, volatile with steam(Ref 3). Other props & methods of prepn are given in Beil & in Sax(Ref 4)

Refs: 1)Beil **9**, 336 & [223] 2)Beil **9**, 339 & [225] 3)Beil **9**, 341, (140) & [228] 4)Sax (1957), 469-70

Mononitrochlorobenzonitrile, $Cl.C_6H_3(NO_2).CN$; mw 182.56, N 15.34%. The following isomers are described in the literature:

2-Nitro-4-chlorobenzonitrile, ndls, mp 97-8°; was prepd by diazotizing 2-Nitro-4-chloroaniline with $NaNO_2$ and addg the diazo soln to a cuprous cyanide soln(Refs 1 & 6);

3-Nitro-4-chlorobenzonitrile, ndls, mp 100-1°; was obtd by application of Sandmeyer's reaction, as above(Ref 2);

3-Nitro-6-chlorobenzonitrile, ndls(from alc), mp 105-9°; was prepd by treating 2-chlorobenzo-nitrile with mixed acid at RT(Ref 3); and

4-Nitro-3-chlorobenzonitrile, ndls, mp 87°; was obtd by Sandmeyer's reaction, as above(Refs 4 & 5). The expl props of the mononitro derivs were not detd

Refs: 1)Beil **9**, 401 & [275] 2)Beil **9**, 403 & [275] 3)Beil **9**, 404 & [276] 4)Beil **9**, 404 5)A. Claus & H. Kurz, JPraktChem **54** I, 197-200(1888) & JCS **54** I, 594(1888) 6)E.B.Hunn, JACS **45**, 1026(1923)

Dinitrochlorobenzonitrile, $Cl.C_6H_2(NO_2)_2.CN$; mw 227.57, N 18.47%. Two isomers are known:

2,4-Dinitro-5-chlorobenzonitrile, yel pltls (from alc), mp 156°; readily sol in boiling alc; diffc sol in cold alc eth, or benz; insol in w; was obtd by boiling 2,4-dinitro-5-chlorobenz-amide with $SOCl_2$ for 24 hrs(Ref 4); and

3,5-Dinitro-2-chlorobenzonitrile, yel crysts

(from alc), mp 139°; was prepd from 3,5-dinitro-2-hydroxybenzonitrile by action of PCl_5(Refs 1 & 3). The expl props of the dinitro derivs were not detd

Refs: 1)Beil **9**, 416 2)A. Claus & H. Kurz, JPraktChem **37**, 197-200(1888) & JCS **54** I, 594(1888) 3)J.J. Blanksma, Rec **20**, 418(1901) & JCS **82** I, 281(1902) 4)H. Goldstein & R. Stamm, Helv **35**, 1332(1952) & CA **47**, 3269 (1953)

Trinitrochlorobenzonitriles, $C_7HClN_4O_6$- were not found in Beil or in CA thru 1956

Chlorobenzoquinone and Derivatives

Chlorobenzoquinone(called Chlorbenzochinon or Chlorchinon in Ger), $O:C_6H_3(Cl):O$; mw 142.53. The 2-Chloro-, 3-Chloro-, and 4-Chloro isomers are described in the literature

Ref: Beil **7**, 630, (338, 346) & [579]

Diazobromochlorobenzoquinone, $C_6H_2BrClN_2O$; mw 233.44, N 12.00%. Two isomers are known:

1-Diazo-4-bromo-6-chloro-o-benzoquinone [called 6-Chlor-4-brom-o-chinon-diazid-(1) or 3-Chlor-5-brom-2-diazo-phenol in Ger], crysts, mp 115-16°, dec at 118°; was obtd by slow decompn of 2,6-dichloro-4-bromobenzenediazonium acetate (Refs 1 & 4); and

4-Diazo-2-bromo-6-chloro-p-benzoquinone [called 6-Chlor-2-brom-p-chinon-diazid-(4) or 6-Chlor-2-brom-4-diazo-phenol in Ger], yel-red ndls (from hot eth, benz or chlf), explodes violently on heating to 150°; v sol in hot alc; sl sol in hot eth or chlf; insol in w; was obtd by addn of bromine water to an aq soln of 4-diazo-6-chloro-phenol-2-sulfonic acid(Refs 2 & 3)

Refs: 1)Beil **16**, 523 2)Beil **16**, 529 3)A. Kollrepp, Ann **234**, 32(1886) & JCS **50** II, 1018 (188) 4)K.J.P. Orton & W.W.Reed JCS **91**, 1555, 1569(1907)

Mononitrodiazochlorobenzoquinone, $C_6H_2ClN_3O_3$; mw 203.56,N 20.64%. Only one isomer is known:

4-Nitro-2-diazo-6-chloro-o-benzoquinone [called Chloronitrodiazophenol in CA **35**, 7716(1941); not found in CA Formula Index(1920-56)] [called 6-Chlor-4-nitro-o-chinon-diazid-(2) or 6-Chlor-4-nitro-2-diazo-phenol in Ger], brn-red scales (from alc), mp explodes vigorously above 100°; sol in concd H_2SO_4 from which soln it is pptd by addn of w; diffc sol in alc, eth or hot w; was first prepd by Griess(Refs 1 & 2) by passing nitrous acid fumes thru a warm alc soln of 4-nitro-6-chloro-2-aminophenol. This compd when mixed with LA was patented as a mixt suitable for detonating rivets(Ref 3)

Refs: 1)Beil **16**, 524 2)P. Griess, Ann **113**, 215(1860) 3)Dynamit-AG, BritP 528299(1940) & CA **35**, 7716(1941)

Note: See also Azido and Azido Halogen Derivatives of Benzoquinone, Vol 1, p B80-L; and See Nitroso Derivatives under Chlorophenol

Chlorobenzotriazolols. See under Benzotriazolol and Derivatives, Vol 2, p B87-R

Chlorobenzoyl Peroxide. See Dichlorobenzoyl Peroxide

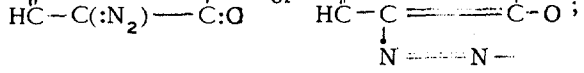
3-Chloro-2-butenylthiocyanate, $H_3C.C(Cl):CH.CH_2.SCN$; mw 147.63, N 9.49%; liq, bp 100-5° at 16 mm Hg; was obtd by reacting equimolar quantities of NH_4CNS (or KCNS) and $H_3C.C(Cl):CH.CH_2.Cl$ (1,3-dichloro-2-butene) in cold alc or on a w bath. Attempts to transpose the compd by distg it at water-pump pressure resulted in an expln

Refs: 1)Beil - not found 2)J. Procházka & O. Wichterle, CollectionCzechChemCommun **14**, 156-61(1949)(in French) & CA **44**, 3442-43 (1950)

5-Chloro-2(chloromethyl)-thiophene. See 5-Chloro-2-thenylChloride

Chlorochromic Anhydride. See Chromyl Chloride under Chlorides

2-Chloro-4-diazo-3-oxidobenzoic Acid [called 2-Chloro-4-diazo-3-oxo-1,5-cyclohexadiene-1-carboxylic in CA 5th Decennial Index, Formulas **41-50**, p 218F(1947-56)] ; $C_7H_3ClN_2O_3$; mw 198.56, N 14.11%;

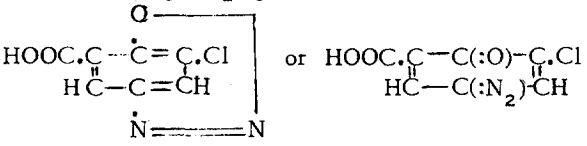


crysts(from w), decomposes violently at 139°, its monohydrate loses H_2O at 80°; was prepd by treating a suspension of 2,3,4-Cl(OH)(NH_2)-C_6H_2.COOH in dil H_2SO_4 with $NaNO_2$(Ref 2)

Refs: 1)Beil - not found 2)G. Berti et al, Gazz **85**, 1636 & 1644(1955) & CA **50**, 10042(1956)

3-Chloro-4-diazo-2-oxidobenzoic Acid [called 3-Chlor-5-diazo-salicylsäure or 6-Chlor

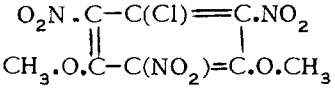
-benzochinon-(1.4)-diazid-(4)-carbonsäure-(2) in Ger], $C_7H_3ClN_2O_3$; mw 198.56, N 14.11%;



ocher-yel scales(from AcOH); mp explodes at 193°; almost insol in w or alc; was prepd by diazotizing 3-chloro-5-aminosalicylic acid dissolved in dil HCl with a cooled soln of $NaNO_2$(Refs 1 & 2)

Refs: 1)Beil **16**, (369) 2)R.Meldola et al, JCS **111**, 543(1917)

1-Chloro-3,5-dimethoxy-2,4,6-trinitrobenzene or 5-Chloro-2,4,6-trinitroresorcinol Dimethylether (Dimethoxypicryl Chloride) [called 5-Chlor-2.4.6-trinitro-resorcin-dimethyläther or 5-Chlor-2.4.6-trinitro-1.3-dimethoxy-benzol in Ger],



; mw 297.63, N 14.12%; yel scales(from alc), crysts (from MeOH); readily sol in alc, acet, benz or MeOH; insol in w or petr eth; was prepd by treating either 1-chloro-2,4-dinitroresorcinol dimethyl-ether or 1-chloro-4,6-dinitroresorcinol dimethyl ether with cold HNO_3 or with mixed HNO_3-H_2SO_4 acid(Refs 1,2 & 3). Its expl props were not detd

Refs: 1)Beil **6** [826] 2)H.H.Schlubach & F. Mergenthaler, Ber **58**, 2735(1925) & CA **20**, 1395(1926) 3)P.J. Van Rijn, Rec **45**, 261 (1926) & CA **20**, 2317(1926)

Chlorodimethylbenzene. See Chloroxylene

4-Chloro-2,6-dinitro-3-hydroxy-benzaldehyde Semicarbazone $HO.C_6HCl(NO_2)_2.CH:N.NH.CO.NH_2$; mw 303.63, N 23.06; dk yel ndls(from dil alc), mp explodes at ca 200°. Its prepn is described in Ref 2

Refs: 1)Beil **8**, [63] 2)H.H.Hodgson & H.G. Beard, JCS **1926**, 2036

Chloroethane and Derivatives

Chloroethane or Ethyl Chloride(called Chloräthan or Äthylchloride in Ger), $CH_3.CH_2.Cl$; mw 64.52; col liq, fr p -139°, bp 12.3°, flash p -58°F(CC), autoign temp 966°F, d 0.9214 at 0°, vap d 2.22, vap press 1000 mm at 20°; expl range 3.6 - 12.0%; MAC 1000 ppm in air or 2660 mg/m³ of air. This liq is harmful to eyes & can cause irritation; it causes narcosis but is the least toxic of all chlorinated hydrocarbons(Ref 2). Other props & methods of prepn are given in Beil (Ref 1)

Ref: 1)Beil **1**, 82, (23), [50] & {133} 2) Sax(1957), 672

Azidochloroethane or Chloroethyl Azide(called Chlorazidoäthan in Ger), $C_2H_4ClN_3$; mw 105.53, N 39.82%. Only one isomer is described in the literature: *1-Azido-2-chloroethane*, $N_3.CH_2.CH_2.Cl$, col liq, having a pleasant odor similar to chloroform, bp 45° at 25 mm press, decrepitates on a hot plate & burns with a violet flame(Refs 1 & 2); vap press & volatility have been reported by Redemann et al(Ref 4); was prepd from 1-azido-2-iodoethane & dry $HgCl_2$ heated at 100° for 3 hrs(Refs 1 & 2). Products of reaction of azidochloroethane with Na phenylacetylene have been studied by Fridman & Lisovs'ka (Ref 3)

Refs: 1)Beil **1**, (133) & {204} 2)M.O.Forster & S.H.Newman, JCS **97** II, 2576(1910) 3)S.G. Fridman & N.M. Lisovs'ka, ZapiskiInstKhim-AkadNauk, InstKhim **6**, Nos 3-4, 353-63(in Russian 364; in German 365)(1940)(& CA **35**, 2470(1941) 4)C.E.Redemann et al, JACS **70**, 2582-83(1948) & CA **42**, 8563(1948)

Mononitrochloroethane, $C_2H_4ClNO_2$; mw 109.51, N 12.80%. Two isomers are known: *1-Chloro-1-nitroethane*, $CH_3.CH(Cl).NO_2$, col liq, bp 127.5°, d 1.2860 at 20°, n_D 1.4264 at 20°; sol in alkalies; insol in w; and *2-Chloro-1-nitroethane* $Cl.CH_2.CH_2.NO_2$, col liq having a sharp taste, bp 173-174°, d 1.405 at 7°; insol in w; decomp in HCl. Other props & methods of prepg mononitrochloroethanes are given in Beil. Their expl props were not detd

Ref: Beil **1**, 101 & {202}

Note: Higher nitro derivs of Chloroethane were not found in Beil or in CA thru 1956

Chloroethanol and Derivatives

Chloroethanol (Ethylene Chlorohydrin, 2-Chloroethyl Alcohol or Glycol Chlorohydrin) [called 2-Chlor-äthanol-(1); 2-Chlor-äthylalkohol; Glykolchlorhydrin or Äthylenchorhydrin in Ger], $Cl.CH_2.CH_2.OH$; mw 80.52, col liq fr p -69°, bp 128°, d 1.202 at 20°, n_D 1.4421 at 20°; miscible with w, alc or eth; MAC 5 ppm or 16 mg/m³ of air; can be prepd by various

methods, for example, by action of hypochlorous acid on ethylene. It is also obtd as a by-product in the manuf of glycol(Ref 1)

Chloroethanol is a narcotic poison affecting the nervous system, the liver, spleen & lungs. Fatal amts can be absorbed thru the skin(Ref 2)

This compd is used as a solvent; its nitrates are expl

Refs: 1)Beil **1**, 337, (170), [333] & {1343} 2)Sax(1957), 677

2-Chloroethanol Nitrite, $Cl.CH_2.CH_2.ONO$; mw 109.51, N 12.79%; col liq, bp 89.5-91°, dec at higher temp, d 1.218 at 20°, n_D 1.4125 at 20°; can be prepd from reaction of chloroethanol, $NaNO_2$ & HCl. Other props & other methods of prepn are given in Beil and in Refs 2 & 3

Refs: 1)Beil **1**, 337, [336] & {1355} 2)H. Petri, ZAnorgChem **257**, 180(1948) & CA **46**, 10996(1952) 3)M.S.Malinovskii & N.M. Medyantseva, ZhObshchKhim **23**, 84-6(1953); 79-81(English translation) & CA **48**, 610 & 12675(1954)

2-Chloroethanol Nitrate, $Cl.CH_2.CH_2.ONO_2$; mw 125.52, N 11.16%; very vol liq, fr p -60°, bp 46° at 8 mm press, d 1.388 at 20°; was prepd by nitrating chloroethanol with HNO_3 (Ref 1)

Chloroethanol Nitrate is an expl less powerful than either NG or Nitroglycol, and is insensitive to impact. Trautzl(Ref 2) thoroughly examined its expl props, some of which are reported by Blatt(Ref 3) as follows:

Brisance, ca 75% that of NG

Explosion Temperature, 242°

Impact Sensitivity with 2 kg wi, 100+ cm vs 6 cm for NG

Power by Lead Block Expansion(Trauzl Test), 307 cc vs 571 cc for NG

Sensitivity to Initiation, can be exploded by a No 8 cap

Stability by Abel Test, 2 min

Trautzl considered Chloroethanol Nitrate a suitable antifreeze additive to NG dynamites. Its props in this respect are comparable to those of NGc. For example, a NG mixt contg 20% Chloroethanol Nitrate freezes at -7°, while one with 20% NGc freezes at -6°

Trautzl(Ref 2) also prepd a blasting gelatin contg Chloroethanol Nitrate 46, NG 46 & Collodion Cotton 8. Its velocity of deton was 2697 m/sec vs 8157 m/sec for a gel contg 98% NG, and 8267 m/sec for one contg 92% NGc. Chlorethanol Nitrate is also a good plasticizer for NC

Refs: 1)Beil **1**, 337 & {1355} 2)K. Trautzl, SS **37**, 146-48(1942) & CA **37**, 5591(1943) 3)Blatt, OSRD 2014(1944) 4)A.M. Pujo et al, Bull Fr **1955**, 974-80 & CA **50**, 13736(1956) (Saponification studies)

2-Nitro-2-chloroethanol, $(O_2N)Cl.CH.CH_2.OH$; mw 125.52, N 11.16%; col liq, bp 94-5° at 8 mm & 103° at 15 mm press, volatile with w + alc vapor; sol in w. It can be prepd by various methods, for example, by treating nitrochloromethane with formaldehyde in w contg some K_2CO_3 (Ref 1)

Its *Sodium salt,* $Na_2C_2H_2O_3NCl+2CH_3.OH$ (pptd from MeOH by acet), hygr crysts, deflagrates vigorously in a flame and is decompd by H_2SO_4 with evolution of flames & deposition of carbon, was prepd by Wilkendorf & Trénel (Ref 2); and its *Sodium salt,* $Na_2C_2H_2O_3NCl+H_2O$, crysts(from alc), was prepd by T & W (Ref 3)

2-Nitro-2-chloroethanol gives, on nitration, an expl nitrate(See below)

Refs: 1)Beil **1**, [339] & {1365} 2)R. Wilkendorf & M. Trénel, **Ber 56**, 611-20(1923) & CA **17**, 2414-15(1923) 3)M. Trénel & R. Wilkendorf, Ber **57**, 2126-30(1924) & CA **19**, 1406-7(1925)

2-Nitro-2-chloroethanol Nitrate, $(O_2N)Cl.CH.CH_2.ONO_2$; mw 170.52, N 16.43%; col oil having a pungent odor, fr p remains liq at -20°, bp decomposes when heated slowly to 150°, detonates violently when heated in a free flame; d 1.568 at 18°; almost insol in w; was prepd by nitrating 2-nitro-2-chloroethanol with mixed acid(Refs 1 & 2)

Refs: 1)Beil **1**, [340] 2)R. Wilkendorf & M. Trénel, Ber **57**, 306 & 308(1924) & CA **18**, 2129(1924)

Note: Higher nitro derivs or nitrates of Chloroethanol were not found in Beil or in CA thru 1956

Chloroethylene and Derivatives

Chloroethylene, Chloroethene or Vinyl Chloride (called Chloräthylen, Chloräthen or Vinylchlorid in Ger), $CH_2:CH.Cl$, mw 62.60; col liq or gas having a faintly sweet odor, fr p -153.8°, bp -13.4°, flash p 108° F(COC), d of liq 0.9195 at 15°, vap press 2660 mm at 25°, vap d 2.15; MAC 500 ppm or 1290 mg/m³ of

air: explosive range 4-22% (Refs 2 & 3). Other props &methods of prepn are given in Beil (Ref 1)
Refs: 1)Beil **1**, 186, (77), [157] & {640} 2)Anon, Safety Maintenance and Production **107**, No 5, 40(1954) 3)Sax(1957), 1249

1-Chloro-1-nitroethylene, CH_2:C.Cl(NO_2); mw 107.46, N 13.02%; heavy oil, bp 149-51° at 760 mm or 54-55° at 14mm press; can be prepd by slowly heating at 150° β-chloro-β-nitroethylnitrate, or by slowly heating to 170° a mixt of β-chloro-β-nitroethanol & P_2O_5 (Refs 1 & 2). It forms an expl polymer described below

Polymer of 1-chloro-1-nitroethylene, $(C_2H_2ClNO_2)_x$; mw $(107.46)_x$, N$(13.02)_x$; yel amor solid, mp 126-30°(dec); sol in hot AcOH; sl sol in alc, eth, petr eth or chlf; insol in w; easily formed when chloronitroethylene is treated with an aq soln of $NaHCO_3$. This polymer explodes mildly when heated in an open flame(Refs 1 & 2)
Refs: 1)Beil **1**, [166] 2)R. Wilkendorf & M. Trénel, Ber **57**, 308-9(1924)

Chloroethyne. See Chloroacetylene and under Halogenated Acetylenes for other salts

Chloroethynyl (Chloroacetynyl) Derivatives of Mercury and Other Salts. See under Chloroacetylene

Chloroform and Derivatives

Chloroform and Trichloromethane (called Chloroform or Trichlormethan in Ger), $CHCl_3$ mw 119.39, col liq having a heavy ethereal odor, fr p -63.5°, bp 61.3°, d 1.498 at 15°, vap press 100mm at 10.4°, vap d 4.12; miscible with many org solvs, such as alc, benz, eth, CS_2, oils & chlorinated solvents; forms azeotropes with w, methanol, alc & hexane. Its MAC is 100ppm or 487mg/m^3 of air. Chloroform causes dilation of the pupils; in the initial stages of inhalation there is a feeling of warmth, then irritation of the mucous membranes; prolonged inhalation will bring on paralysis accompanied by cardiac & respiratory failure and finally death(Refs 1 & 4)

The discovery of chloroform has been credited to Liebig in Germany, to Soubeiran in France, and to Guthrie in the USA at about the same time(1831)(Ref 3). Once widely used as an anesthetic, chloroform has now become increasingly important as a solvent and as a chemical reactant(Refs 3 & 5)

The explosion of chloroform in contact with alkali metals is described by Davis(Ref 2)
Refs: 1)Beil **1**, 61, (9), [14] & {51} 2)Davis (1943), 402-3 3)Kirk & Othmer **3**(1949), 842ff 4)Sax(1957), 473-74 5)CondChemDict(1961), 260 6)US Specification MIL-C-12037B (Tech chloroform)

Mononitrochloroform, Chloropicrin or Nitrotrichloromethane(called Trichlornitromethan or Chlorpikrin in Ger), $O_2N.CCl_3$; 164.39, N 14.01%; col sl oily liq, fr p -64°; bp 112.4°, d 1.6449 at 25°; n_D 1.4595 at 20°, vap press 24.0mm at 25°; readily sol in benz, amyl alc, CS_2, absolute alc; sl sol in eth; v sl sol in w. It was first prepd by an English chemist, Stenhouse, in 1848 by distilling a mixt of PA in water with bleaching powd(Refs 1 & 2). Nitrochloroform is a powerful irritant and affects all body surfaces. It causes lachrymation, vomiting, bronchitis & pulmonary endema. A concn of 1 ppm causes a smarting in the eyes. It is called vomiting gas and has been used extensively by the military as a chemical warfare agent. Four ppm is sufficient to render a man unfit for action, and 20 ppm when breathed from 1 to 2 min causes definite bronchial or pulmonary lesions(Ref 5). See also CWA PS under Chemical, Biological and Radiological Agents

Among the applications of Chloropicrin today are its use in the manuf of dyes(Crystal Violet) and as an oxidg agent instead of NB in the prepn of quinoline & quinoline derivs. Its widest use is as a fumigant and soil disinfectant(Ref 2)
Refs: 1)Beil **1**, 76, (20), [41] & {113} 2)Kirk & Othmer **3**(1947), 882 3)E. Cheylan, MP **32**, 417-21(1950)(Prepn of chloropicrin from nitromethane) 4)J. Fauveau, Chim & Ind(Paris) **67**, 249-52(1952)(Prepn of chloropicrin by action of Javel water on PA) 5)Sax (1957), 479-80 6)US Specification MIL-C-100664A
Note: See also Chloromethane and Derivatives

Chloroformamidine Nitrate. See under Formamidine and Derivatives

Chloroformamidine Perchlorate. See under Formamidine and Derivatives

Chlorohydrins. See Chloropropanediol and Derivatives

Chlorohydroxyanisole and Derivatives

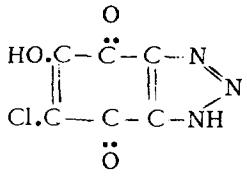
Chlorohydroxyanisole (Chloroxyanisol or 5-Chlor-resorcin-monomethylather in Ger), $HO.C_6H_3(Cl).O.CH_3$; ndls, mp 99°
Ref: Beil **6**, [819]
Azidochlorohydroxyanisde, $HO.C_6H_2(N_3)(Cl).O.CH_3$ - not found in Beil or CA thru 1956
Mononitrochlorohydroxyanisole, $HO.C_6H_2(NO_2)(Cl).O.CH_3$. Three isomers are described in the literature
Refs: 1)Beil - not found 2)H.H.Hodgson & J.S. Wignall, JCS **1928**, 330 3)H.H.Hodgson & W.E. Batty, JCS **1934**, 1434-35
Dinitrochlorohydroxyanisole, $HO.C_6H(NO_2)_2(Cl).O.CH_3$; one isomer 4,6-dinitro-5-chloro-hydroxyanisole, col ndls, mp 126° is described in the literature
Refs: 1)Beil - not found 2)H.H.Hodgson & W.E.Batty, JCS **1934**, 1434
Trinitrochlorohydroxyanisole, $HO.C_6(NO_2)_3(Cl).O.CH_3$, mw 293.59, N 14.31%. One isomer is known:
2,4,6-Trinitro-5-chloro-3-hydroxy anisole or 3-Chloro-5-methoxy-picric Acid, nearly col micro-prisms(from w), mp 147°; was prepd by adding 1g of 5-chloro-3-hydroxyanisole to well-stirred 10 ml of nitric acid(d 1.47) at RT during 15 mins, followed by slow heating to bp, cooling and pouring on ice. Its expl props were not detd
Refs: 1)Beil - not found 2)H.H.Hodgson & W.E. Batty, JCS **1934**, 1435 & CA **29**, 133 (1935)

Chlorohydroxybenzaldehyde-(4-nitrophenylhydrazone and Derivatives

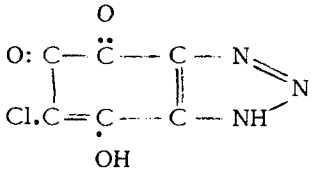
Chlorohydroxybenzaldehyde-(4-nitrophenylhydrazone) [called Chlor-oxy-benzaldehyd-(4-nitro-phenylhydrazon in Ger], $O_2N.C_6H_4.NH.N:CH.C_6H_3Cl.OH$; mw 291.69, N 14.41%. Four isomers are described in the literature: *2-Chloro-3-hydroxy-*, orn-red ndls(from dil alc), mp 244-45°; *4-Chloro-3-hydroxy-*, violet red ndls(from dil alc), mp 226-27°; *6-Chloro-3-hydroxy-*, red ndls(from dil alc), mp 250-51°; and *2-Chloro-3-hydroxy-benzaldehyde-(4-nitro-phenylhydrazone,* red ndls(from alc or AcOH), mp 288°(dec). Other props & methods of prepn are given in the Refs
Refs: 1)Beil **15**, [197 & 200] 2)H.H. Hodgson & H.G. Beard, JCS **1926**, 149 & 152 3)H.H. Hodgson & F.H. Moore, JCS **1926**, 1742
Mononitrochlorohydroxybenzaldehyde-(4-nitrophenylhydrazone, $O_2N.C_6H_4.NH.N:CH.C_6H_2Cl(NO_2).OH$; mw 336.69, N 16.64%. Six isomers are known: *2-Nitro-4-chloro-3-hydroxy-*, dk red-brn ndls(from AcOH), mp 275-76°(dec); *2-Nitro-6-chloro-3-hydroxy-*, orn ndls(from dil AcOH), mp 256-57°(dec); *4-Nitro-2-chloro-3-hydroxy-*, orn ndls(from AcOH), mp 294-95° (dec); *4-Nitro-6-chloro-3-hydroxy-*, brick-red ndls(from AcOH), mp 284-86°(dec); *6-Nitro-2-chloro-3-hydroxy-*, orn-red ndls(from dil AcOH), mp 232-33°; and *3-Nitro-6-chloro-4-hydroxy-(4-nitrophenylhydrazone),* dk orn ndls(from AcOH), mp 266°(dec). Other props and methods of prepn are given in the Refs
Refs: 1)Beil **15**, [198-200] 2)H.H. Hodgson & H.G. Beard, JCS **1926**, 2032 & 2034 3)H.H. Hodgson & T.A. Jenkinson, JCS **1928**, 2274
Dinitrochlorohydroxybenzaldehyde-(4-nitrophenylhydrazone), $O_2N.C_6H_4.NH.N:CH.C_6HCl(NO_2)_2.OH$; mw 381.69, N 18.35%. The following isomers are known:
2,4-Dinitro-6-chloro-3-hydroxy-(4-nitrophenyl-hydrazone), dk red ndls(from AcOH), mp 285-86°(dec)
2,6-Dinitro-4-chloro-3-hydroxy-(4-nitrophenyl-hydrazone), dk orn-red ndls(from dil AcOH), mp explodes at 235-36°
4,6-Dinitro-2-chloro-3-hydroxy-(4-nitrophenyl-hydrazone), dk red ndls(from AcOH), mp 277-79°(dec)(Refs 1 & 4); and
3,5-Dinitro-2-chloro-4-hydroxy-(4-nitrophenyl-hydrazone), reddish-brn ndls(from dil AcOH), mp 267°(dec)(Refs 2 & 5). These derivs were prepd by the general method of Hodson & Beard (Ref 3) in which the appropriate aldehyde, dissolved in dil AcOH, was treated gradually with an aq soln of p- nitrophenylhydrazine until pptn ceased. The hydrazone was formed in quantitative yield
Refs: 1)Beil **15**, [197] 2)Beil **15**, [201] 3)H.H. Hodgson & H.G. Beard, JCS **127**, 880 (1925) 4)H.H. Hodgson & H.G. Beard, JCS **1926**, 2030-35 5)H.H. Hodgson & T.A. Jenkinson, JCS **1928**, 2274
Note: Higher nitrated derivs were not found in Beil

6(or 5)-Chloro-5(or 6)-hydroxy-benzotriazole-4,7

-quinone; or 6(or 5)-Chloro-7(or 4)-hydroxy -benzotriazole-4,5(or 6,7)-quinone,



or



mw 203.56, N 20.64%; yel, lustrous rhmb crysts, contg AcOH(from AcOH), mp detonates at 230 -31°; sol in w, alc, AcOH & acet; sl sol in eth, benz or benzine; was prepd by heating 6-chloro -5-hydroxy-benzotriazolo-4,7-quinone-7-aniline with alc HCl and treating the resulting product with boiling water (Ref 1, p 272; Ref 2, p 305)

This compd also forms salts & addn compds which are, themselves, unstable on heating. Other expl derivs prepd by Zincke et al(Ref 2) include:

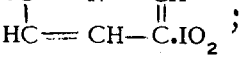
6,7-(or 4,5)-Dichloro-benzotriazole-4,5, (or 6,7)-quinone, $C_6HCl_2N_3O_2$, red ndls (from AcOH), mp blackens when heated & detonating above 260° (Ref 1,p 234; Ref 2, p 300); and

6,6,7 (or 4,5,5)-Trichloro-4,5 (or 6,7)-dioxo -4,5,6,7-tetrahydro-benzotriazole; or 6,6,7 (or 4,5,5)-Trichloro-4 (or 7)-hydroxy-5 (or 6) -oxo-5,6-dihydro-benzotriazole, $C_6H_2Cl_3N_3O_2 + H_2O$, col pltlts (from eth + benz), mp begins to decomp at 130° and explodes above 260° (Ref 1, p 234; Ref 2, p 296)

Refs: 1)Beil **26,** 234 & 272 2)T.Zincke et al, Ann **311,** 296, 300 & 305(1900); JCS **78** I, 524-25(1900)

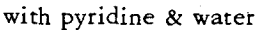
2-Chloro-3-hydroxy-1-methyl 4-isopropyl-benzene -6-diazoniumchloride, $HO.C_6H.Cl(CH_3)[CH-(CH_3)_2].N(\vdots N).Cl$; mw 249.12, N 10.99%; col ndls (from eth-alc), mp explodes on heating; was prepd by treating a cold alc soln of 2-chloro-6 -amino-3-hydroxy-1-methyl-4-isopropylbenzene with nitrous acid(Refs 1 & 2)

Refs: 1)Beil **16,** 533 2)M.Andresen, JPrakt-Chem **23,** 180(1881)

6-Chloro-3-iodoxy-pyridine(called 6-Chlor-3 -jodo-pyridin in Ger),  ;

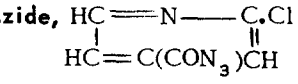
mw 281.43, N 4.98%; col crysts, mp explodes at 205°; was obtd by treating 6-chloro-pyridyl-3-iododichloride with pyridine & water (Refs 1 & 2)

Refs: 1)Beil **20,** [154] 2)A.Pieroni, AttiAccadLinceiRend [6] **2,** 126(1925) & CA **20,** 764(1926)

6-Chloro-3-iodoso-pyridine(called 6-Chlor-3 -jodoso-pyridin in Ger),  ;

mw 265.43, N 5.28%. Its Chloride salt, $ClNC_5H_3.ICl_2$, called *6-Chloro-pyridyl-3 -iodochloride,* yel crysts, mp 104-6°(dec) (Ref 3) & 115°(dec)(Ref 2); was prepd by reacting Cl with 6-Chloro-3-iodopyridine in chlf(Ref 1)

Refs: 1)Beil **20,** [154] 2)C.Rath, GerP 468302(1924) & CA **23,** 612(1929) 3)A. Pieroni, AttiAccadLinceiRend [6] **2,** 126(1925) & CA **20,** 764(1926)

2-Chloro-isonicotinoyl Azide, 

mw 182.57, N 30.69%; wh ndls, mp 46-7°; sublimes in vacuo at 40°; sol in ether; was prepd by treating 2-chloro-isonicotinoyl hydrazide with Na nitrite in w, as described in Ref 2. Its expl props were not detd

Refs: 1)Beil - not found 2)J.Bäumler et al, Helv **34,** 497 & 500(1951); CA **45,** 7572 (1951)

Note: See also 4-Chloro-picolinyl Azide

Chloromethane and Derivatives

Chloromethane or Methyl Chloride(called Chlormethan or Methylchlorid in Ger), $CH_3.Cl$; mw 50.49; col gas, fr p -97.7°, bp -23.7, flash p below 32°, d 0.918 at 20°; vap d 1.78; MAC 100ppm or 209 mg/m³ of air; explosive range 8.1-17.2%. It has very sl irritant props and may be inhaled without discomfort. Its narcotic action is somewhat weaker than that of chloroform (qv)(Refs 1,2 &3). Other props & methods of prepn are given in Refs 1 & 2

Refs: 1)Beil **1,** 59, (8), [11] & {36}

2)Kirk & Othmer **3**(1949), 738 3)Sax(1957), 887

Nitrosochloromethane, $ON.CH_2.Cl$; mw 79.49, N 17.62%; lt yel crysts(bimolecular in soln, monomolecular when melted), possessing a sharp lacrimatory & irritating smell, mp ca 62°(with blue coloration) & decomp vigorously at 65-66°; sol in org solvs; insol in w or cold NaOH; can be prepd from trimolecular formaldoxime, $(H_2C:NOH)_3$, but better from formaldehydeoxime-hydrochloride by reaction with nitrosyl chloride in ether under cooling (Refs 1 & 2)

Refs: 1)Beil **1**, [39] 2)H.Rheinboldt & M.Dewald, Ann **451**, 275(1927) & CA **21**, 1107(1927)

Mononitrochloromethane, $O_2N.CH_2Cl$; mw mw 95.49, N 14.67%.oil, bp 122-23°, d 1.4666 at 15°; sol in 20ps w; mod sol in KOH; can be prepd from Na salt of nitromethane and chlorine water(Ref 2), or by other methods(Ref 1)

Refs: 1)Beil **1**, 76, [41] & {112} 2)J.Tscherniak, Ber **8**, 608(1875)

Dinitrochloromethane, $(O_2N)_2CH.Cl$; mw 140.49, N 19.94%. This compd is known only in the form of its salts & other derivs, some of which are expl:

Potassium Salt, $(O_2N)_2C.K(Cl)$; mw 178.58, N 15.69%; lt yel prisms (from w), mp explodes 83-85°; readily sol in warm w; sl sol in cold w or alc; was prepd by action of alc KOH on dinitrodichloromethane(Ref 1,p [44]; Ref 3)

Silver Salt, $(O_2N)_2C.Ag(Cl)$; mw 247.35, N 11.33%; yel ndls, mp explods 80-81°; insol in w; was prepd by mixing the K salt with $AgNO_3$(Ref 1,p [44]; Ref 3)

Dinitrochlorobromomethane, $(O_2N)_2C.Br(Cl)$; mw 219.39, N 12.77%; col to yel pungent oil, fr p 9.2-9.3°, bp 75-76° at 15 mm, dec on heating at atm pressure, d 2.0394 at 20°, n_D 1.4739 at 20°; mod sol in alc; was prepd by passing chlorine thru an aq soln of K dinitrobromomethane(Ref 1,p 78; Ref 2); or by passing chlorine into equal wts of K dinitrobromomethane & Na acetate(Ref 1,p [44]; Ref 3); or by passing chlorine into an ice-cold aq soln of Amm dinitrobromomethane(Ref 1,p {115}; Ref 4). Alcoholic KOH reacts with dinitrochlorobromomethane to form a yel salt which, when crystd from w, exploded at 147°. This salt possessed a K content intermediate between those of K chloro- & K bromo-dinitromethanes(Refs 2 & 4)

Dinitrochloroiodomethane, $(O_2N)_2C.I(Cl)$; mw 2.66.38, N 10.52%; col oil, having characteristic odor of halogenonitroparaffins, slowly becomes reddish-brn on standing, dec on heating, d 2.1424 at 12°; was prepd by passing chlorine into an ice-cold aq soln of dinitroiodomethane & Na acetate. Alcoholic KOH converts this compd into a mixt of expl salts, K chloro- & K iododinitromethane(Ref 1,p [45]; Ref 3)

Refs: 1)Beil **1**, 78, [44 & 45] & {115} 2)S.N.Losanitsch, Ber **17**, 848(1884) & JCS **46** II, 1107(1884) 3)R.Gotts & L. Hunter, JCS **125**, 447(1924) & CA **18**, 1270(1924) 4)F.C.Schmidt et al, JOC **9**, 421(1944) & CA **39**, 275(1945)

Trinitrochloromethane, $(O_2N)_3C.Cl$; mw 185.49, N 22.66%; yel liq, fr p 4.0-4.5°, bp 51.5 - 52.0 at 30mm, explodes on heating at atm pressure, d 1.6616 at 19°, n_D 1.4560 at 14.9°; v sol in w; sol in most org solvents; can be prepd by several methods such as by treating K trinitromethane with chlorine in ice-cold ether(Ref 1) or in w (Ref 3) or by adding acetone to a mixt of fuming nitric acid and NaCl(Ref 4)

Refs: 1)Beil **1**, [46] & {116} 2)E.Schmidt et al, Ber **54**, 1483(1921) 3)A.K.Macbeth & D.D.Pratt, JCS **119**, 354-55(1921) 4)W.J. Boyd, JCSI **44**, 222T(1925)

Note: See also Chloroform and Derivatives

Chloromethoxy-picric Acid. See Trinitrochlorohydroxyanisole under Chlorohydroxyanisole

Chloromethylacetylene or 1-Chloro-1-propyne, $CH_3C{:}C.Cl$. It was obtd in small quantity, mixed with ethyl bromide, by interaction of p-toluenesulfonyl chloride with methylacetylene -magnesium bromide in dibutyl ether. It was not purified. The pure compd, bp 32.8-33°, n_D 1.4131 at 20°, was prepd by dehydrohalogenation of cis -1,2-dichloro-1-propene or by chlorination of 1 -propyne (Ref 3)

Refs: 1)Beil **1**, {922} 2)F.F.Cleveland & M.J.Murray, JChemPhys **11**, 451-52(1943)

3) A. T. Morse & L. C. Leitch, Can J Chem **32**, 503 (1954)

Chloroaminotoluene or Chlorotoluidine(called Chlor- methylaniline or Chlor-aminotoluol in Ger), C_7H_8ClN; mw 141.59, N 9.89%. The *Chloro-N-methylaniline isomers*, $Cl.C_6H_4.NH.CH_3$, are described in Ref 1; and the *Chloro-C-methylaniline* isomers, $Cl.C_6H_3(NH_2).CH_3$, are described in Ref 2. Most of these compds are liquids or low-melting crysts. Many form salts & addn compds, some of which are unstable. Other props & methods of prepn are given in the Refs

Refs: 1)Beil **12**, 599, 603, 609, (298, 301, 304), & [324] 2)Beil **12**, 835, 836, 870, 871, 988, 989, (388, 389, 404, 435, 436) & [453, 454, 455, 473, 530, 531]

Nitrosochloromethylaniline, $C_7H_7ClN_2O$; mw 170.60, N 16.42%. The following isomers are described in the literature:

4-Nitroso-2-chloro-N-methylaniline or 2-Chloro-1,4-benzoquinone-4-methylimide-1-oxime, $ON.(Cl)C_6H_3.NH.CH_3$ or $HO.N:(Cl)C_6H_3:N.CH_3$, dk-grn prisms(from benz), mp 134-36°, (dec) (Refs 1 & 3)

4-Nitroso-3-chloro-N-methylaniline or 2-chloro-1,4-benzoquinone-1-methylimide-4-oximine, grn prisms(from petr eth), mp 131-32°(Refs 1 & 3)

N-Nitroso-2-chloro-N-methylaniline or (2-Chlorophenyl)-methylnitrosamine, $Cl.C_6H_4.N(NO).CH_3$, golden-yel oil, bp 89° at 1.2mm, d 1.266 at 15° (Refs 2 & 4)

N-Nitroso-3-chloro-N-methylaniline or (3-Chlorophenyl)-methylnitrosamine, lfts, mp 36-38°(Refs 2 & 4); and

N-Nitroso-4-chloro-N-methylaniline or (4-Chlorophenyl)-methylnitrosamine, lt-brn crysts (from gasoline), mp 51°(Refs 2 & 4)

Other props & methods of prepn are given in the Refs

Refs: 1)Beil **7**, (346) 2)Beil **12**, 602, 607, 619, (304) & [319, 322, 332] 3)O.Fischer & P.Neber, Ber **45**, 1096-98(1912) 4)E.Schmidt & H.Fischer, Ber **53**, 1540(1920) & CA **15**, 87(1921)

Mononitrochloromethylaniline, $C_7H_7ClN_2O_2$; mw 186.60, 15.02%. One isomer of *N-Nitro-C-chloro-N-methylaniline*, $Cl.C_6H_4.N(NO_2).CH_3$, is described in the literature(Ref 1). Three isomers of *C-Nitro-C-chloro-N-methylaniline*, $(O_2N)Cl.C_6H_3.NH.CH_3$, are described in the literature (Ref 2). Seventeen isomers of *C-Nitro-C-chloro-C-methylaniline* are known(Ref 3). None of the above monitro derivs is reported as being explosive. Other props & methods of prepn are given in the Refs

Refs: 1)Beil **12**, 619 2)Beil **12**, 729, 730, 733 3)Beil **12**, 849, 850, 1006, 1007(395, 408) & [460, 461, 462, 477, 478, 537]

Mononitro-N-nitroso-chloromethylaniline, $C_7H_6ClN_3O_3$; mw 215.60, N 19.49%. Two isomers are known:

2-Nitro-N-nitroso-5-chloro-N-methylaniline or (2-Nitro-5-chlorophenyl)-methylnitrosamine, $(O_2N)Cl.C_6H_3.N(NO).CH_3$, brn prisms(from hot alc), mp 67.5-68.5; was prepd by continued action of nitrous acid on an alc soln of 3-chloro-N-methylaniline(Ref 1,p 731; Ref 2) and

4-Nitro-N-nitroso-2-chloro-N-methylaniline or (4-Nitro-2-chlorophenyl)-methylnitrosamine, lt yel ndls, mp 94.5-95.5°; was prepd by action of nitrous acid on an alc soln of 2-chloro-N-methylaniline(Ref 1,p 733; and Refs 2 & 3)

The expls props of these C-Nitro-N-nitroso-derivs were not detd

Refs: 1)Beil **12**, 731, 733 2)R.Stoermer & P.Hoffmann, Ber **31**, 2532(1898) & JCS **76** I, 44(1899) 3)D.Z.Zavel'skii & L.A.Lishnevskaya, ZhObshchKhim **25**, 362-71(1955); 343-50(in English) & CA **50**, 2461(1956)

Dinitrochloromethylaniline, $C_7H_6ClN_3O_4$; mw 231.60, N 18.15%. Five isomers are described in the literature:

2,4-Dinitro-5-chloro-N-methylaniline, $(O_2N)_2Cl.C_6H_2.NH.CH_3$, orn-yel ndls(from alc), mp 106-107°; mp 163-65° (Ref 7); sol in alc, eth, acet, benz, chlf or AcOH; sl sol in petr eth; was prepd by reacting 5-chloro-1,2,4-trinitrobenzene with benzaldehyde-methylimide in boiling alc(Ref 1,p [414]: Ref 4)

2,6-Dinitro-4-chloro-N-methylaniline, orn ndls, mp 100-100.5°; was prepd by treating (2,6-dinitro-4-chlorophenyl)-methylnitrosamine with concd HCl(Ref 1,p 760; Ref 2)

4,6-Dinitro-2-chloro-N-methylaniline, yel nds (from alc), mp 133°; was prepd by reaction of 4,6-dinitro-1,2-dichlorobenzene with an alc soln of methylamine(Ref 6)

3,5-Dinitro-2-chloro-4-methylaniline, $(O_2N)_2(Cl)C_6H(NH_2).CH_3$, yel nds(from alc), mp 173°; was prepd by warming 3,5-dinitro-2-chloro-4-methylbenzoic acid azide with AcOH Ref 1,p [539]; Ref 5) and

2,6-Dinitro-3-chloro-4-methylaniline, golden-yel ndls(from alc), mp 137°; v sl sol in cold w -

or in cold concd HCl; was prepd by treating 3,5-dinitro-2-chloro-4-acetaminotoluene with cold H_2SO_4 (Ref 1,p [540]; Ref 3)

The expl props of these dinitro derivs were not detd

Refs: 1)Beil **12**, 760 & [414, 539, 540] 2)R. Stoermer & P. Hoffmann, Ber **31**, 2534 (1898) & JCS **76** I, 44(1899) 3)W. Davies, JCS **119** I, 868(1921) 4)M. Giua, Gazz **53**, 55(1923) & JCS **124** I, 455(1923) 5)H. Lindemann, Helv **11**, 1028(1928) & CA **23**, 827 (1929) 6)C.F. Duin, Rec **51**, 884-85(1932) & CA **26**, 5550(1932) 7)J.L. Robert, Rec **56**, 418(1937) & CA **31**, 4656(1937)

Dinitro-N-nitroso-chloromethylaniline, $C_7H_5ClN_4O_5$; mw 260.60, N 21.50%. Two isomers are known:

2,6-Dinitro-N-nitroso-4-chloro-N-methylaniline or *(2,6-Dinitro-4-chlorophenyl)-methylnitrosamine,* $(O_2N)_2Cl.C_6H_2.N(NO).CH_3$, lt yel ndls(from alc) mp 99-99.5°; was prepd by treating 4-chloro-N-methylaniline in cold alc soln with nitrous acid(Refs 1 & 2)

4,6-Dinitro-N-nitroso-2-chloro-N-methylaniline or *(4,6-Dinitro-2-chlorophenyl)-methylnitrosamine,* compd not recrystd because it decompd when petr eth was added to its benz soln at RT; was obtd by treating an ice-cold soln of 2-chloro-N-methylaniline with HNO_3 & $NaNO_2$ (Ref 3)

Refs: 1)Beil **12**, 760 2)R. Stoermer & P. Hoffmann, Ber **31**, 2533(1898) & JCS **76** I, 44 (1899) 3)C.F. Van Duin, Rec **51**, 885(1932)

Trinitrochloromethylaniline, $C_7H_5ClN_4O_6$; mw 276.60, N 20.26%. Two isomers are described in the literature:

N,2,6-Trinitro-4-chloro-N-methylaniline or (2,6-Dinitro-4-chlorophenyl)-methylnitramine, $(O_2N)_2Cl.C_6H_2.N(NO_2)CH_3$, crysts, mp 100°; was obtd by nitration of 2-nitro-4-chloro-N-methylaniline(Refs 1 & 2) and

N,4,6-Trinitro-2-chloro-N-methylaniline or 4,6-Dinitro-2-chlorophenyl)-methylnitramine, crysts (from alc), mp 91-2°; was obtd by nitration of 4,6-dinitro-2-chlorodimethylaniline on warming with HNO_3 and also by synthesis from 1,2-dichloro-4,6-dinitrobenzene and $CH_3.NH_2$, the resulting 4,6-dinitro-2-chloro-N-methylaniline being converted into the nitramine by concd HNO_3 at RT(Ref 3)

The expl props of these trinitro derivs were not reported

Refs: 1)Beil **12**, 760 2)J.J.Blanksma, Rec **21**, 274(1902) 3)C.F. Van Duin, Rec **51**, 884-85(1932) & CA **26**, 5550(1932)

Tetranitrochloromethylaniline, $C_7H_4ClN_5O_8$; mw 321.60, N 21.78%. Only one isomer is known:

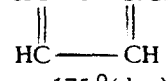
N,2,4,6-Tetranitro-3-chloro-N-methylaniline or 2,4,6-Trinitro-3-chlorophenyl)-methylnitramine, $(O_2N)_3Cl.C_6H.N(NO_2).CH_3$, yel crysts(from chlf), mp 119°; was prepd by nitration of 2-nitro-5-chloro-N-methylaniline(Refs 1 & 2). Its expl props were not detd

Refs: 1)Beil **12**, 771 2)J.J. Blanksma, Rec **21**, 276(1902)

Chloromethylbenzenes. See Chlorotoluene and Benzyl Chloride

N-(Chloromethyl)-N'-[(Methylnitramino)-methyl]-N,N'-dinitromethanediamine. See 1-Chloro-2,4,6-trinitro-2,4,6-triazaheptane

2-Chloromethylthiophene (called 2-Chlormethyl-thiophen in Ger),



mw 132.60; liq, bp ca 175°(dec); when heated to decompn, it emits highly toxic fumes(Ref 4); was first prepd by Biedermann(Refs 1 & 2) by passing chlorine water into α-thenyl alcohol

F.H. Bergeim of the Squibb Institute for Medical Research has reported (Ref 3) that it is hazardous to store this compd even when cold and in the dark. A bottle of 2-chloromethylthiophene stored for several weeks in a dark room at 5°, and in another instance a higher boiling fraction, obtd as a by-product in its synthesis, exploded violently. Since encountering these explosions, Dr Bergeim has found that other laboratories have had similar experiences

Note: See also 5-Chloro-2-thenyl Chloride

Refs: 1)Beil **17**, 37 2)A. Biedermann, Ber **19**, 639 & 1620 Anm 1(1886) 3)F.H. Bergeim, C & EN **30**, 2546(1952) & CA **46**, 9846(1952) 4)Sax(1957), 475

2-Chloromethylthiophene Chloride. See 5-Chloro-2-thenyl Chloride

Chloronitrophenylethene or Chloronitrophenylethylene. See Chloronitrostyrene

Chloropentamminechromium (III) Azide. See p A277 in Vol I, under Ammines

Chloropentamminecobalt(III) Chlorate. See p A280 in Vol I, under Ammines

Chloropentamminecobalt(III) Chlorite. See p A280 in Vol 1, under Ammines

Chloropentamminecobalt (III) Perchlorate. See p A280 in Vol I, under Ammines

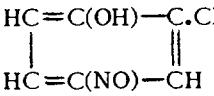
Chlorophenol and Derivatives

Chlorophenol (called Chlorphenol in Ger), $Cl.C_6H_5.OH$; mw 128.60. The 2-Chloro- (Ref 1), 3-Chloro- (Ref 2), and 3-Chloro- (Ref 3), phenols are described in the literature. Sax(Ref 4) gives their toxicity, hazard & some props. Other props & methods of prepn are given in Beil

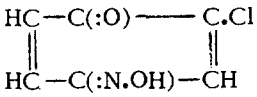
Refs: 1)Beil **6**, 183(98) & [170] 2)Beil **6**, 185, (99) & [172] 3)Beil **6**, 186, (100) & [174] 4)Sax(1957), 478

Nitrosochlorophenol or Chlorobenzoquinone -oxime, $C_6H_5ClNO_2$; mw 158.56, N 8.83%. Two isomers are described in the literature:

4-Nitroso-2-chlorophenol or 2-Chloro- p -benzoquinone-4-oxime,

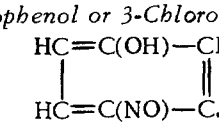


or

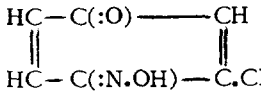


; dk yellowish ndls(from chlf+petr eth), mp 142-48°(dec); mod sol in hot w; sl sol in alc or eth; diffc sol in hot benz, petr eth, acet or CS_2. Other props & methods of prepn are given in Beil(Ref 1); and

4-Nitroso-3-chlorophenol or 3-Chloro-p-benzoquinone-4-oxime,



or



. This nitroso form exists as grn-yel crysts(from benz), rapidly darkening on exposure to light, mp 135.8°(dec); mod sol in eth giving an intensive yel color, sol in cold alc, sol in molten phenol or naphthalene giving a deep red color, sol in aq NaOH with frothing & giving a deep red color ; almost insol in cold benz. The oxime form exists as grn-yel ndls(from benz + AcOH), mp 184° (dec); more sol in w than the nitroso form giving a yel-grn soln in cold w and a pure grn soln in hot w, sol in alc or eth giving a lt-yel color, sol in phenol giving an orn-yel color, sol in NaOH or Na_2CO_3 soln giving a red color which becomes grn on dilution

Other props & methods of prepn are given in Beil(Ref 2)

Refs: 1)Beil **7**, 631, (346) & [580] 2)Beil **7**, [579]

Mononitrochlorophenol, $(O_2N)Cl.C_6H_3.OH$; mw 173.55, N 8.07%. Ten isomers are known and described in the literature . Their props & methods of prepn are given in Beil

Ref: Beil **6**, 238-40, (122) & [226-29]

Mononitrodiazochlorophenol. See Mononitrodiazochlorobenzoquinone under Benzoquinone and Derivatives

Dinitrochlorophenol, $(O_2N)_2Cl.C_6H_2.OH$; mw 218.55, N 12.82%. Eight isomers are known and described in the literature. Their props & methods of prepn are given in Beil

Refs: Beil **6**, 259, 260, (128) & [247, 248]

Trinitrochlorophenol, $(O_2N)_3Cl.C_6H.OH$; mw 263.56, N 15.94%. The following isomers are known:

2,4,5-Trinitro-3-chlorophenol(?), crysts, mp 112.5 - 113.5°, volatile with steam; sol in eth, sl sol in w; was prepd by heating the di-K salt of 4,5-dinitro-3-chlorophenol-2-sulfonic acid with dil H_2SO_4 (Refs 1, 3 & 4) . It forms a yel crysts Ag salt insol in aq ammonia

2,4,6-Trinitro-3-chlorophenol or Chloropicric Acid, yel crysts(AcOH or alc) or prisms(from w), mp 112-19°, not volatile with steam; mod sol in chlf, benz, alc or eth; can be obtd as the ultimate product of nitration of all mono- & di-nitro-3-chlorophenols (Refs 2 & 3). It forms yel salts of Na, Ag, NH_3 (mp 223-24°) & $PhNH_2$ (mp 189-90°). See Refs 2, 3, 5, 6, 7 & 8 for other props & methods of prepn. The Chloride salt, an expl compd called *Chloropicryl Chloride,* is mentioned under Trinitrochlorobenzenes, Derivatives of Chlorobenzene

The expl props of trinitrochlorophenols were not reported

Refs: 1)Beil **6**, [283] 2)Beil **6**, 292 & [283] 3)H.H.Hodgson & F.H.Moore, JCS **127** II, 1603-4(1925) & CA **19**, 2937(1925) 4)H.H.Hodgson & A.Kershaw, JCS **1929**, 2917-23 5)A.I.Titov & N.G.Laptev, ZhObshch-Khim **19**, 267(1949) & CA **43**, 6585(1949) 6)K.F.Keirstead & R.Gaudry, CanJRes **28B**,

750(1950) & CA **45**, 4669(1951) 7)A.I. Titov, ZhObshchKhim **22**, 1929-35(1952) & CA **47**, 6360(1953) 8)H.Feuer & A.H.Harban, USP 2679538(1954) & CA **49**, 4715(1955)

2,3,4,6-Tetranitro-5-chlorophenol, $(O_2N)_4Cl.C_6.OH$; mw 308.56, N 18.16%; col crysts (from chlf or CCl_4), mp 147°, explodes on heating to higher temp; soln of crysts in w or alc becomes yel; was prepd by nitration of 3-nitro-5-chlorphenol using mixed acid(HNO_3-H_2SO_4) and cooling to 50°(Refs 1 & 2)
Refs: 1)Beil **6**, 293 2)J.J.Blanksma, Rec **27**, 36(1908); JCS **92** I, 126-27(1907) & CA **2**, 1133(1908)

Chlorophenylamino-propanediol. See Chloroanilino-propanediol

Chlorophenyl-aminotetrazole. See 1-Amino-5-(o-chlorophenyl)-α-tetrazole, Vol I,p A193-R

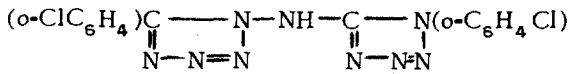
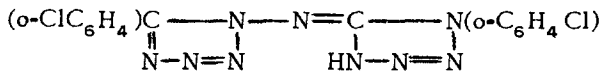
Chlorophenylazides. See Chloroazidobenzenes under Chlorobenzene and Derivatives

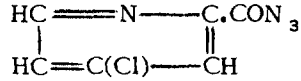
1-(4-Chlorophenyl)-III-cyanoguanyl-triazene or 1-(p-Chlorophenylazo)-3-cyanoguanidine, $Cl.C_6H_4.N{:}N.NH.C(:NH).NH.CN$; mw 222.56, N 37.75%; yel crysts(from alc), reported as brn solid by Bekhli et al(Ref 4), mp-dec at 136-40° on heating slowly, explodes mildly on heating rapidly between 150-60°; was first prepd by Walther & Grieshammer (Refs 1 & 2) from reaction of 4-Chlorobenzenediazonium chloride & dicyanodiamide in alk soln. It was later prepd by others (Refs 3, 4, 5 & 6).Some of its salts are mild expls
Refs: 1)Beil **16**, (405) 2)R. von Walther & W. Grieshammer, JPraktChem **92**, 230(1915) & JCS **110** I, 172(1916) 3)H.L. Bami et al, JIndianInstSci **29A**, 1-8(1946) & CA **41**, 6214-15(1947) 4)A.F. Bekhli et al, ZhPriklKhim **20**, 591-6(1947) & CA **43**, 3793(1949) 5)P.H. Rumpf, FrP 948328(1949) & CA **45**, 5723(1951) 6)Z.V. Pushkareva et al, DoklAkadN **92**, 89-92 (1953) & CA **48**, 10635(1954)

Chlorophenyldinitromethane. See ω,ω-Dinitro-4-chlorotoluene under Chlorotoluene and Derivatives

Chlorophenylethene or Chlorophenylethylene. See Chlorostyrene

5-(o-Chlorophenyl)-tetrazole,  ;
mw 180.60, N 31.03%; col fine ndls, mp 177°, explodes on heating above mp or on impact; readily sol in hot alc; sl sol in eth; v sl sol in w; was obtd when aminochlorophenyltetrazole(See Vol 1, p A193-R) was treated with $NaNO_2$ & HCl(Ref 2)
Refs: 1)Beil - not found 2)R. Stollé et al, JPraktChem **138**, 9-11(1933) 3)F.R. Benson, ChemRevs **41**, 16(1947)

1-(1'-o-Chlorophenyl-5'-tetrazolylamino)-5-o-chlorophenyltetrazole or 1-o-chlorophenyl-5-(5'-o-chlorophenyl-1'-iminotetrazolyl)-tetrazole 4,5-dihydride,

or

mw 374.20, N 33.70%; col ndls, mp 166°(dec), defgr on heating above mp; was obtd by heating for 30 hrs a mixt of di-o-chlorobenzohydrazide chloride & NaN_3 in 95% alc(Ref 2)
Refs: 1)Beil -not found 2)R Stollé et al, JPraktChem **137**, 328 & 333-34(1933) & CA **27**, 4233(1933)

4-Chloro-picolinyl Azide, 
mw 182.57, N 30.69%; crysts, mp 92°, explodes mildlyon heating rapidly; diffc sol in w or dil acids; was obtd from 4-chloropicolinyl hydrazide by reaction in cold HCl with aq KNO_2(Ref 2)
Refs: 1)Beil - not found 2)R. Graf et al, Ber **64 B**, 23(1931) & CA **25**,2429(1931)
Note: See also 2-Chloro-isonicotinoyl Azide

3-Chloropicramide or 3-Chloropicrylamide. See 2,4,6-Trinitro-3-chloroaniline under Chloroaniline and Derivatives

Chloropicrin. See Mononitrochloroform under Chloroform and Derivatives

3-Chloropicrylamide or 3-Chloropicramide. See 2,4,6-Trinitro-3-chloroaniline under Chloro-

aniline and Derivatives

Chloropicryl Chloride. See Chloride Salt of TNCB(or Picryl Chloride) under Chlorobenzene and Derivatives

Chloropropane and Derivatives

Chloropropane or Propyl Chloride(called Chlorpropan or Propylchlorid in Ger), C_3H_7Cl; mw 78.55. The n-isomer (1-chloropropane), col liq, fr p -123°, bp 47.2°, flash p 0°F, d 0.890, is described in detail in Ref 1, and its toxicity & hazards in Ref 3. The isopropyl isomer (2-chloropropane), clear liq, fr p -118°, bp 35.3°, flash p -26°F, d 0.858 at 25°, is described in detail in Ref 2, and its toxicity & hazards in Refs 3, p 802-3

Refs: 1)Beil **1**, 104, (34), [72] & {219} 2)Beil **1**, 105, (34), [72] & {221} 3)Sax (1957), 802-3 & 1062

1-Azido-3-chloropropane, $N_3.CH_2.CH_2.CH_2.Cl$; mw 119.56, N 35.14%; liq, bp 32-4° at 4 mm; was prepd by heating 1-bromo-3-chloro-propane with NaN_3 in alc(Refs 1 & 2). Its expl props were not detd

Refs: 1)Beil **1**, {261} 2)S.G. Fridman, MemInstChem, AcadSciUkrain 4, No 3, 351-55; in Russian 356; in German 356-57(1937) & CA **32**, 5373(1938)

Diazidochloropropane, $N_3C_3H_5(N_3)Cl$; mw 160.58, N 52.35%. Two isomers are known:

1,2-Diazido-3-chloropropane, $N_3.CH_2.CH(N_3).CH_2Cl$, col oil having a faint sweet odor, bp 62° at 0.6mm; and

1,3-Diazido-2-chloropropane, $N_3.CH_2.CHCl.CH_2.N_3$, col liq having a pungent sweet odor, becomes yel on exposure to light, bp 63° at 0.6 mm. Both compds react vigorously with concd H_2SO_4. Their expl props were not detd (Refs 1 & 2)

Refs: 1)Beil **1**, (37) 2)M.O. Forster & J.C. Withers, JCS **101** I, 494-96(1912)

Nitrosochloropropane, $ON.C_3H_6.Cl$; mw 107.54, N 13.03%. Two isomers are known:

1-Nitroso-1-chloropropane, cryst substance having a pungent odor & irritating the eyes to tears; mp 56-7° to a deep blue liq, dec 83°; was obtd by reacting propionaldoxime with NOCl in eth at low temp(Ref 1,p [79]; Ref 2,p 277); and

2-Nitroso-2-chloropropane, dk blue liq having a strong odor, bp ca 70°(w/o decompn); readily sol in ordinary org solvs; insol in w; was prepd from acetoxime & NOCl in eth(Ref 1; Ref 2,p 301)

See also Refs 3 & 4

Refs: 1)Beil **1**, 115, [79] & {255} 2)H. Rheinboldt & M. Dewald, Ann **451**, 278 & 301 (1927) & CA **21**, 1107 & 2872(1927) 3)O. Wichterle & J. Vogel, CollectionCzechChem-Communs **14**, 209-18(in English)(1949) & CA **44**, 1515(1950)(Addn of nitroso compds to conjugated systems) 4)P.Tart, BullBelg **63**, 525-41(1954) & CA **49**, 7976(1955)(Spectroscopic data)

Mononitrochloropropane, $O_2N.C_3H_6.Cl$; mw 123.54, N 11.34%. The following isomers are described in the literature:

1-Nitro-1-chloropropane, col liq having a pleasant aroma, bp 67° at 56mm, d 1.205 at 15°, n_D 1.4251 at 20°;

1-Nitro-2-chloropropane, liq, bp 75° at 15 mm, d 1.2361 at 15°;

1-Nitro-3-chloropropane, liq, bp 197°(with partial decompn), 115-16° at 40 mm, d 1.267 at 20°;

2-Nitro-1-chloropropane, col liq, bp 172-73°, 80-84° at 25 mm, d 1.2 at 18°; and

2-Nitro-2-chloropropane, col liq having a pleasant aroma, separates as prisms from cold CS_2 & eth, bp ca 134°(with some decompn when heated cautiously), explodes violently when heated rapidly; mod sol in alc or eth; v sl sol in w; insol in KOH; was prepdby passing NaClO into a cold aq soln of acetoxime, or by reaction of Cl with 2-nitropropane in alc soln(Refs 2, 3 & 4)

Other props & methods of prepg nitrochloropropanes are given in Ref 1

Refs: 1)Beil **1**, 116, [79] & {259} 2)R. Möhlau & C. Hoffmann, Ber **20**, 1506(1887) 3)L. Henry, ChemZtr **1898** I, 193 4)H. Rheinboldt & M. Dewald, Ann 455, 302(1927)

Mononitronitrosochloropropane, $C_3H_5ClN_2O_3$; mw 152.54, N 18.37%. Two isomers are known:

1-Nitro-1-nitroso-1-chloropropane, $(O_2N)C(NO)(Cl).CH_2.CH_3$; dk grn oil; was prepd by addg 1-nitro-1-chloropropane to a KOH aq soln cooled to 0°, and slowly introducing $NaNO_2$ & H_2SO_4. This compd was patented as an additive to Diesel fuel to improve its cetane number(Ref 3); and

2-Nitro-2-Nitroso-1-chloropropane, $Cl.CH_2.C(NO_2)(NO).CH_3$; blue oil, very unstable; was

obtd when an ethereal soln of chloroacetoxime was treated with liq NO_2. On oxidn with chromic acid in AcOH, a small quantity of dinitrochloropropane was formed(Refs 1 & 2)

The expl props of these compds were not detd

Refs: 1)Beil **1**, 117 2)R.Scholl & G. Matthaiopoulos, Ber **29**, 1554(1896) & JCS **70** I, 520 (1896) 3)T.T.Noland & E.M. Nygaard, USP 2392610(1946) & CA **40**, 2285(1946)

Dinitrochloropropane, $C_3H_5ClN_2O_4$; mw 168.54, N 16.63%. Only one isomer is reported in the literature:

2,2-Dinitro-1-chloropropane, $Cl.CH_2.C(NO_2)_2.CH_3$, oil, bp 103-5° at 15.4mm, dec at 200-2°, readily volatile with steam; was obtd by oxidn of 2-nitro-2-nitroso-1-chloropropane with CrO_3 in AcOH. Its expl props were not reported (Refs 1 & 2)

Refs: 1)Beil **1**, 118 2)R. Scholl & G. Matthaiopoulos, Ber **29**, 1554(1896) & JCS **70** I, 520 (1896)

Note: High nitrated derivs of chloropropane were not found in Beil or in CA thru 1956

Chloropropanediol and Derivatives

Chloropropanediol, Chlorohydrin or Glyceryl Chlorohydrin(Called Chlorpropandiol, Monochlorhydrin, Glycerinchlorhydrin or Chlorpropylenglykol in Ger), $Cl.C_3H_5(OH)_2$; mw 110.54. Two isomers are known, both of which are present in the commercial product:

3-Chloropropane-1,2-diol or α-Chlorohydrin, $Cl.CH_2.CH(OH).CH_2.OH$, col to pale-yel sl viscous liq having a pleasant odor, bp 213° (dec), fl p (OC) 280°F, d 1.3204 at 20°, n_D 1.4810 at 20°; miscible with w & org solvs; immiscible with oils; for method of prepn, see under Commercial Chlorohydrin and in Ref 1;

2-Chloropropane-1,3-diol or β-Chlorohydrin, $CH_2(OH).CHCl.CH_2OH$, col to pale-yel viscous liq, bp 146° at 18mm, d 1.3207 at 20°; mod sol in w or benz; miscible with acet, alc or eth; can be prepd by treating allyl alc with hypochlorous acid, or, in small quantity together with α-isomer, by heating glycerin with HCl at 100° followed by fractionation(Ref 2)

Commercial Chlorohydrin, a mixt of alpha & beta isomers, of which alpha is in a greater proportion; col liq, unstable & hygr, bp 213-18°(dec), d 1.326 at 18°. It was first prepd by Berthelot in 1853; can be obtd by passing HCl gas into glycerin at 100° in the presence of 2% AcOH; or at RT by treating glycerin with sulfur monochloride(S_2Cl_2)

It is used as a solv for acetyl cellulose, cellulose acetate, resins, gums, etc; and serves as an intermediate in org synthesis & for the prepn of some expls

Refs: 1)Beil **1**, 473(246), [537] & {2150} 2)Beil **1**, 476, (247), & [542] 3)Naoúm, NG (1928), 182-83 4)Davis(1943), 220 5)Kirk & Othmer **3**(1949), 857-60 6)Sax(1957), 480 7)CondChemDict(1961), 261

Azido and Nitroso derivs of Chloropropanediol were not found in Beil or in CA thru 1956

3-Chloropropane-1,2-diol-1-nitrate or α-Chlorohydrin Mononitrate(called 3-Chlor-propylenglykol -1-nitrat or Monochlorhydrin-mononitrat in Ger), $Cl.CH_2-CH(OH).CH_2.ONO_2$; mw 155.54, N 9.01%; oil, bp 75° at 7 mm, d 1.4411 at 20°, n_D 1.4751 at 29°(Ref 4); was obtd not completely pure from epichlorohydrin(chloropropylene oxide)by nitration in cold with HNO_3(Refs 1 &3). A practically pure product was obtd by Nichols, Jr et al (Ref 4) by a nitration procedure using NH_4NO_3 & HNO_3 in aq solns at a higher temp(ca 35°), or in an alternate procedure with non-aq solns of epichlorohydrin in $CHCl_3$ and white fuming HNO_3 added simultaneously at 40°

Pujo et al(Ref 5), by treatment of epichlorohydrin with N_2O_4 and methanolysis, obtd a product bp 73-5° at 0.1-0.2mm, n_D 1.4750 at 19°, which presumably was a mixt of $Cl.CH_2.CH(OH).CH_2.ONO_2$ & $Cl.CH_2.CH(ONO_2).CH_2.OH$. The presence of the 2-nitrate isomer was indicated, but not further substantiated, by the strong reducing action toward Fehling's soln of the oxidized product. This product, when oxidized with CrO_3-AcOH, was very unstable(bp 28-57° at 0.1mm)

Chlorohydrin mononitrate can be detonated by strong initiation, but it will not explode under impact of a falling weight(Ref 2)

Refs: 1)Beil **1**, {2154} 2)Naoúm, NG(1928), 190-91 3)IGFarbenind, FrP 846575(1939) & CA **35**, 1071(1941) 4)P.L.Nichols, Jr et al, JACS **75**, 4255-56(1953) & CA **48**, 11296(1954) 5)A.M.Pujo et al, BullFr **1955**, 974-80 & CA **50**, 13736(1956)

3-Chloropropane-1,2-diol-1,2-dinitrate; α-Dinitrochlorohydrin; or Glycerol Monochlorohydrin Dinitrate (called Disalpetersäureester des γ-Chlor-proplyeneglykols, 3-Chlor -propylenglykol-dinitrat or "Dinitromonochlorhydrin" in Ger), $Cl.CH_2.CH(ONO_2).CH_2(ONO_2)_2$;

mw 200.55, N 13.97%, OB to CO_2 and Cl_2 - 20%; lt-yel liq, fr p +5°, can be supercooled to below -30°; bp 190-195°(sl dec),and 121-123° at 15mm press; d 1.541 at 15°, d 1.532 at 20°(Ref 8); vapor density 5.83; dielectric constant 17.50 at 20°(Ref 8), dipole moment 3.46(Ref 8); n_D 1.459 at 20°(Ref 8); sol in alc, eth, acet or chlf; insol in w or dil acids; was first prepd by Henry on treating glycerin-α-monochlorohydrin with a mixt of equal parts of fuming HNO_3 and cond H_2SO_4(Refs 1 & 2). Also can be prepd by the nitration of epichlorhydrin(Ref 6). According to Sax(Ref 10), dinitrochlorohydrin is rated as severely toxic on exposure, ingestion or inhalation; and is dangerous, as shock will explode it; heating causes decompn, burning and emitting of toxic fumes; and it can react vigorously with reducing materials(See also Ref 1)

2-Chloropropane-1,3-diol-1,2-dinitrate or β-Dinitrochlorohydrin, $(O_2NO).CH_2.CHCl.CH_2.(ONO_2)$; yel liq, fr p 16.2°(Ref 5,p 173 & Ref 6, p 185); can be prepd in a fairly pure state by the action of PCl_5 on a chlf soln of K-dinitrochloroglycerin(Ref 6,p 181). It is one of the components of Commercial Dinitrochlorohydrin *Commercial Dinitrochlorohydrin* is a mixt of α-isomer with a small amt of β-isomer; yellowish to red-brn oil, bp 190°(dec); 121.5° at 13 mm; can be prepd by nitrating with HNO_3-H_2SO_4 commercial chlorohydrin. Its soly & other props are almost the same as those of the α-isomer (Refs 5, 6 & 9)

It is a powerful expl possessing the following props: *Brisance,* by Lead Block Compression Test 83% NG; *Explosion Temperature* - defgr mildly at 190° when heated rapidly; dec slowly at 170° with evoln of yel fumes when heated slowly; completely decomp at ca 180°; *Explosion Equations:*

$C_3H_5Cl(ONO_2)_2 \longrightarrow CO_2+2CO+2H_2O+HCl+N_2$ or

$C_3H_5Cl(ONO_2)_2 \longrightarrow 2CO_2+CO+H_2O+H_2+HCl+N_2$;

Heat of Explosion, 1184cal/g with H_2O liq and HCl dissolved, vs 1140cal/g(as detd value) or 72%NG; *Heat of Formation,* 87.8kcal/mol; *Hygroscopicity* - not hygroscopic; *Ignition Sensitivity* - diffc to ignite but, when ignited, it burns quietly without expln; *Impact Sensitivity* - 2kg falling 10-20 cm, vs 4 cm for NG; and FI(Figure of insensitiveness)- 120% PA; *Initiation Sensitivity* - very sensitive to initiation; can be detonated by a No 1 cap; *Power by Trauzl Test* - 10g sample,No 8 cap, gave 475cc vs 600cc for NG or 79% NG and 130%PA; *Thermal Stability* - Slightly more stable than NG(Abel's Test at 72°- 30 minutes); *Velocity of Detonation,* in small diam tubes and under sl confinement, gives higher values than NG under the same conditions; however, under strong confinement and in larger diam tubes, NG gives much higher values; *Volatility,* moderate although appreciably higher than that of NG(a 20g sample at 75° for 24 hrs lost 3.1% vs 0.35% for NG); at RT(20°) volatility is only 0.13% (Refs 5, 6 & 9)

Uses: Dinitrochlorohydrin has been used to a great extent in Europe, in coml NG explosives. It acts not only as an antifreeze in depressing the fr p of NG but also renders NG safer to handle. This was of particular importance for Germany where railroad regulations prohibited the transportation of NG explosives unless they were rendered less sensitive by addn of such substances as dinitrochlorohydrin. It was used also for a short time in the USA by the Aetna Explosives Company, but was replaced first by diglycerin tetranitrate and then by nitroglycols. Thorton, Jr(Ref 7), has described many methods for differentiating aromatic compds, such as nitrobenzene and nitrotoluene, from nitric esters, such as dinitrochlorohydrin and nitroglycols, etc. Although dinitrochlorohydrin does not gelatinize NC by itself, it does so when mixed with NG. When slightly heated, in the absence of NG, it forms with NC a gelatin not very tough and readily exudable(Refs 5, 6 & 9)

Refs: 1)Beil **1**, 474 & {2154} 2)L.Henry, Ann **155**, 165(1870) 3)F.Roewer, ChemZtr **1906** II, 983 4)Westfälisch-Anhaltische Sprengstoff-AG, BritP 1789(1906) & CA **1**, 943 (1907) 5)Kast(1921), 173 6)Naoúm, NG(1928), 179-190 7)W.M.Thorton, Jr, USBurMinesCirc **7155**(1941), 13pp & CA **35**, 5702(1941) 8)L.J. de Kreuk, Rec **61**, 822 & 824(1942); ChemZtr **1943** I, 827; CA **38**, 3523(1944) 9)Blatt, OSRD **2014**(1944) 10)Sax(1957), 630-32 [Comp is listed as both Dinitrochlorohydrin and Dinitromonochlorohydrin]

Mononitrochloropropanediol, $C_3H_6ClNO_4$; mw 155.54, N 9.01%. Only one isomer is described in the literature:

2-Nitro-2-chloropropane-1,3-diol [called 2-Nitro -2-chlor propandiol-(1.3) or β-Nitro-β-chlor -trimethylenglykol in Ger], $HO.CH_2.C(Cl)$-

$(NO_2).CH_2.OH$, rhmb ndls, spears or prisms (from NaCl soln or ethyl acet + chlf), mp 115-16°(begins to sinter), bp 123° at 4 mm; readily sol in w, alc, eth or eth acet; sol in toluene;v sl sol in chlf, CCl_4 or benz; was prepd by reaction of Cl with the Na salt of 2-Nitrotrimethyleneglycol in ether, or from nitrochloromethane & aq formaldehyde soln by treatment with K_2CO_3 soln(Refs 1, 2 & 3). It forms some salts & derivs which are expl
Refs: 1)Beil **1**, [543] 2)E.Schmidt & R. Wilkendorf, Ber **55**, 317(1922) & CA **16**, 2475 (1922) 3)R.Wilkendorf & M. Trénel, Ber **56**, 613 & 616(1923) & CA **17**, 2414(1923) 4)M. Trénel & R. Wilkendorf, Ber **57**, 2129(1924) & CA **19**, 1406(1925)
Note: Higher nitro- or nitrated derivs of Chloropropanediol, other than those described above were not found in Beil or in CA thru 1956

Chloropropylene Oxide. See *α*-Epichlorohydrin

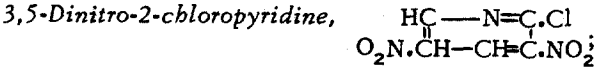
Chloropyridine and Derivatives

Chloropyridine(called Chlor-pyridin in Ger), C_5H_4ClN; mw 113.54, N 12.34%. The 2-Chloro- (Ref 2), 3-Chloro- & 4-Chloro- isomers are described in detail in the literature(Ref 1). These isomers form many addn compds & salts, some of which are unstable on heating
Refs: 1)Beil **20**, 230 & 231, (80) & [152] 2)Sax(1957), 481

Mononitrochloropyridine $C_5H_3ClN_2O_2$; mw 158.54, N 17.68%. Three isomers are known:
3-Nitro-2-chloropyridine, ndls (from w), mp 101-2°; almost insol in cold w; insol in NaOH; sol in concd acids; was prepd by diazotizing 3-nitro-2-aminopyridine with $NaNO_2$ in HCl soln [Ref, p (82)];
3-Nitro-4-chloropyridine, col crysts, mp 45°, bp 95° at 5 mm; was prepd by heating on a water bath 3-nitro-4-hydroxypyridine with PCl_5 & a little $POCl_3$ {Ref, p[154]}; and
3-Nitro-6-chloropyridine, sl fragrant ndls (from w), mp 108-10°; sol in concd acids; v sl sol in cold w; insol in NaOH; was prepd by diazotizing 5-nitro-2-aminopyridine with $NaNO_2$ in concd HCl or by heating 5-nitro-2-hydroxy-pyridine with PCl_5 & $POCl_3$ to 110-20°{Ref, pp (82) & [155]}
Ref: Beil **20**, (82) & [154, 155]

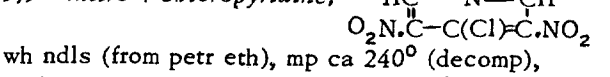
Dinitrochloropyridine, $C_5H_2ClN_3O_4$; mw 203.55, N 20.64%. The following isomers are described in the literature:

3,5-Dinitro-2-chloropyridine,



wh crysts(from petr eth) mp 64° (Ref 4) & 68° (Ref 6); this same compd described in CA (Ref 3) as yel prisms (from MeOH), mp 102-3° is obviously incorrectly identified. It was prepd by reaction of 3,5-dinitro-2(1H) pyridone (pale yel crysts, mp 175°) with PCl_5&$POCl_3$ heated at 120° (Ref 4); and by conversion with PCl_5 of 3,5 -dinitro-2-hydroxypyridine (described as shining yel ndls, mp 176°), obtd by nitration of 5-nitro -2-hydroxypyridine with fuming H_2SO_4 & HNO_3, and reported as hitherto undescribed (Ref 6). Takahashi & Yamamoto(Ref 3) earlier had prepd 3,5-dinitro-2-hydroxypyridine, and by treating it with PCl_5 & $POCl_3$ obtd yel prisms, mp 102-3° (See above). The dinitro deriv reported by Ochiai & Futaki (Ref 5) as ?,?-Dinitro -4-chloropyridine, prisms (from petr eth), mp 68°, and obtd by treating ?, ?-dinitro-4-hydroxy-pyridine with $POCl_3$ & dimethylaniline at 130 -40° is probably the 3,5-dinitro-2-chloro deriv, since other derivs prepd showed that the 4-OH group led to the formation of 3,5-derivs or derivs substituted ortho to the OH position (Ref 5); and

3,5-Dinitro-4-chloropyridine,



wh ndls (from petr eth), mp ca 240° (decomp), preliminary softening occurs at ca 60°, rapidly decomp on standing at RT, reverting to dinitrohydroxypyridine; was prepd by treating 3,5-dinitro-4-hydroxypyridine under reflux with PCl_5 & $POCl_3$ at 160° for 1 hour (Ref 2)

These compds & some of their derivs explode on heating to high temps
Refs: 1)Beil - not found 2)V.A. Petrow & E.L. Rewald, JCS **1945**, 313-14 & CA **39**, 4328(1945) 3)T.Takahashi & Y. Yamamoto, JPharmSocJapan **69**, 409-11(1949) & CA **44**, 1978(1950) 4)A.H. Berrie et al, JCS **1951**, 2593 & CA **46**, 8114(1952) 5)E.Ochiai & K.Futaki, JPharmSocJapan **72**, 274-75(1952) (in German) & CA **47**, 6417(1953) 6)E. Plažek, Rec **72**, 573(1953) & CA **49**, 1038 (1955)
Note: Higher nitro derivs of Chloropyridine were not found in Beil or in CA thru 1956

Chloropyridine Carboxylic Acid Azides.

See 2-Chloro-isonicotinoyl Azide and 4-Chloro-picolinyl Azide

Chloroquinone. See Chlorobenzoquinone

Chloroquinone-oxime. See Nitrosochlorophenol under Chlorophenol and Derivatives

Cholorosilanes, SiH_xCl_y, compds of silicon, chlorine and hydrogen where the total number of atoms of chlorine & hydrogen or hydrogen subs tituted radicals adds up to 4

Summary of Flammability Data

Substance	Diluent Atm	Flammability Limit % by Vol Lower	Upper	Critical O Content % by Vol
Methyl chloride, CH_3Cl	O_2,N_2	8.5	17.2	14.6
Methyltrichlorosilane, CH_3SiCl_3	O_2,N_2	7.6	>20	12.9
Dimethyldichlorosilane, $(CH_3)_2SiCl_2$	O_2,N_2	3.4	>9.5	11.5
	O_2,N_2,CO_2 (N_2/CO_2=5.7)	3.4(?)	>8.8	12.4
Trimethylchlorosilane, $(CH_3)_3SiCl$	O_2,N_2	2.0	>6.2	10.3
Methyldichlorosilane, CH_3HSiCl_2	O_2,N_2	3.4	>24.0	2.9

reduce flammability in the latter cases. Data in the above table show the lower limit rises moderately in the series: trimethylchlorosilane, dimethyldichlorosilane & methyltrichlorosilane (Ref 1)

Refs: 1)E.W. Balis et al, IEC **41**, 1459-61 (1949) & CA **43**, 7229-30(1949) 2)Sax(1957), 481

Their toxicity & hazard are given by Sax (Ref 2). Balis et al (Ref 1) have detd flammability data and established the critical oxygen content - i.e, the O content below which flame propagation is not possible in a gaseous atm, no matter what its chlorosilane content. A summary of these data establishing this critical O content for 4 chlorosilanes & for methyl chloride (chloromethane, qv) by means of a modified BurMines - App is given above:

The outstanding datum in the above table is the low critical oxygen content value for methyldichlorosilane. This value is considerably below previous values reported and supports the view according to Balis et al that the silicon-hydrogen linkage is extremely sensitive toward oxygen when an electrical discharge is present. A striking difference between methyl & silyl compds is the much lower tendency of Cl on the central atom to

Chlorostyrene and Derivatives

Chlorostyrene, Chlorophenylethene or Chlorophenylethylene (called Chlor-styrol, Chlor-phenyl-äthylen or Chlor-vinyl-benzol in Ger), C_8H_7Cl, mw 138.59. Three isomers are described in the literature: alpha-Chloro-, $C_6H_5.CCl:CH_2$; omega-Chloro-, $C_6H_5.CH:CH.Cl$; and 4-Chlorostyrene, $Cl.C_6H_4.CH:CH_2$. The prepn & props of these isomers, all liquids, are given in Beil

Refs: Beil **5**, 476, (230) & [367]

Mononitrochlorostyrene, $C_8H_6ClNO_2$; mw 183.59, N 8.62%. The following isomers are known:

2-Nitro-α-chlorostyrene, $O_2N.C_6H_4.CCl:CH_2$; lt yel oil, dec on distilling (Ref 1,p 479);

2-Nitro-ω-chlorostyrene, $O_2N.C_6H_4.CH:CH.CH.Cl$; pale yel ndls or prisms(from alc) having a pleasant odor, mp 58-59°; sl sol in eth or hot alc (Ref 1,pp 479 & [369]);

3-Nitro-ω-chlorostyrene, $O_2N.C_6H_4.CH:$-

$CH.Cl$; yel prisms (from alc) having a pleasant odor, mp 83°; the crysts darken on exposure to light (Ref 1,p [369]);
4-Nitro-α-chlorostyrene, $O_2N.C_6H_4.CCl:CH_2$; lt yel ndls (from petr eth), mp 63-4°; diffc sol in w, otherwise sol in most solvs (Ref 1,p 480);
4-Nitro-ω-chlorostyrene, $O_2N.C_6H_4.CH:CH.Cl$; lt yel ndls(from alc) having a pleasant odor, mp 126° (Ref 5) & 128° (Ref 1,p [369]); less sol in alc than the 3-nitro-ω-chloro deriv;
α-Nitro-ω-chlorostyrene, $C_6H_5.C(NO_2):CHCl$; yel oil which decomp at 12 mm, and on standing forms a red mass; was prepd from phenylacetylene ($C_6H_5C:CH$) and nitryl chloride(NO_2Cl) in ether with cooling in dry ice. Without a solv the compds react with a violent explosion(Ref 3);
ω-Nitro-α-chlorostyrene, $C_6H_5.CCl:CH.NO_2$; solid (from petr eth), mp 55-6°, bp 127-29° at 13 mm; was obtd with other products when phenylacetylene($C_6H_5C:CH$) was allowed to react in vacuo at 0-20° with nitrosyl chloride (NOCl) for 2-3 days. The reactions of this compd with w, KOH, alcoholates, thiocyanates, amines & other reactants were also reported(Ref 7);
ω-Nitro-ω-chlorostyrene, $C_6H_5.CH:C.Cl(NO_2)$; golden-yel lfts or pltlts(from petr), mp 48-49°; insol in w; sol in other common solvs(Ref1,p 480). Braude et al(Ref 4) detd its UV light absorption data;
ω-Nitro-2-chlorostyrene, $Cl.C_6H_4.CH:CH.NO_2$; yel crysts(from alc), mp 47°; was prepd by cooling o-chlorobenzaldehyde and nitromethane in a freezing mixt, carefully addg aq NaOH, and addg concd HCl & w to the ppt in MeOH(Ref 2). Barret(Ref 6) patented the use of this compd & other derivs of ω-nitrostyrene as retarders in the copolymerization of maleic anhydride & styrene;
ω-Nitro-3-chlorostyrene, $Cl.C_6H_4.CH:CH.NO_2$; yel crysts(from alc), mp 48-9°; was prepd, using m-chlorobenzaldehyde, by the same method as for the 2-chloro deriv(Ref 2); and
ω-Nitro-4-chlorostyrene, $Cl.C_6H_4.CH:CH.NO_2$; elongated yel prisms(from alc), mp 111-14°; was prepd, using p-chlorobenzaldehyde, by the same method as for the 2-chloro deriv(Ref 1,p (230) & Ref 2). Prepn of this compd is also reported by Huitric et al (Ref 10) & by Kamlet(Ref 8). Its UV absorption spectra were detd by Kamlet & Glover(Ref 9)

The props & methods of prepg the mononitrochlorostyrenes, not given above, can be found in the Refs indicated

Refs: 1)Beil **5**, 479, 480, (230) & [369] 2)N. Campbell et al, JCS **1940**, 449 & CA **34**, 5060(1940) 3)W. Steinkopf & M. Kühnel, Ber **75B**, 1328(1942) & CA **37**, 4687(1943) 4)E.A. Braude et al, JCS **1947**, 1104 & CA **42**, 107(1948) 5)E. Müller, IAngChem **61**, 179-83(1949) & CA **44**, 1429(1950) 6)G.R. Barrett, USP 2537015(1951) & CA **45**, 3195(1951) 7)R. Perrot & R. Berger, CR **235**, 185-87(1952) & CA **47**, 5908(1953) 8)M.J. Kamlet, JACS **77**, 4896(1955) & CA **50**, 6466(1956) 9)M.J.Kamlet & D.J. Glover, JACS **77**, 5696(1955) & CA **50**, 2290(1956) 10)A.C. Huitric et al, JACS **78**, 619(1956) & CA **50**, 12890-91(1956)

Dinitrochlorostyrene, $C_8H_5ClN_2O_4$; mw 228.59, N12.25%. The following isomers are described in the literature:
ω,3-Dinitro-α-chlorostyrene, $O_2N.C_6H_4.CCl:CH.NO_2$; no description given, mp 105°; was prepd by reaction of m-nitrophenylacetylene with nitrosyl chloride (NOCl) for several weeks(Ref 7);
ω,4-Dinitro-α-chlorostyrene, $O_2N.C_6H_4.CCl:CH.NO_2$; no description given, mp 118-19°; was prepd by reaction of p-nitrophenylacetylene with NOCl for several weeks(Ref 7);
ω-4-Dinitro-ω-chlorostyrene, $O_2N.C_6H_4.CH:CCl(NO_2)$; lt yel ndls(from aq acet), mp 150-53°; readily sol in acet or ethyl acetate; sol in MeOH, benz or AcOH; diffc sol in petr eth; was obtd, in addn to allo-α-chloro-p-nitrocinnamic acid, when allo-α-chlorocinnamic acid [$C_6H_5.CH:C(Cl).COOH$] was nitrated with fuming HNO_3, or by nitrating normal α-chlorocinnamic acid at 20-25° instead of in the cold(Refs 1 & 3);
ω,4-Dinitro-2-chlorostyrene, $(O_2N)Cl.C_6H_3.CH:CH.NO_2$, pale yel ndls(from alc), mp 149-50°; was prepd by nitrating ω-nitro-2-chlorophenylstyrene(Ref 5);
ω,5-Dinitro-2-chlorostyrene, $(O_2N)Cl.C_6H_3.CH:CH.NO_2$; no description given, mp 142°; was prepd by condensing the appropriate

benzaldehyde with nitromethane(Ref 6); *ω,2-Dinitro-4-chlorostyrene,* $(O_2N)Cl.C_6H_3.CH{:}CH.NO_2$; lt yel crysts, mp 98-99°; readily sol in org solvs, except petr eth; insol in w; was prepd by nitrating 2-nitro-4-chlorocinnamic acid with HNO_3(d 1.52), or by reacting 2-nitro-4-chlorobenzaldehyde with nitromethane & KOH in MeOH-KOH(Refs 2 & 4); and *ω,3-Dinitro-4-chlorostyrene,* $(O_2N)Cl.C_6H_3.CH{:}CH.NO_2$; ndls(from alc), mp 150-51°(dec); mod sol in eth, alc, benz, chlf or acet; insol in petr eth or w; was prepd similar to the ω,2-dinitro deriv using the appropriate reactants(Refs 2 & 4)

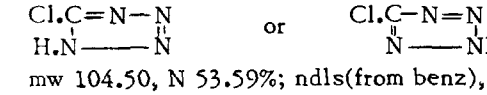
The expls props of these dinitrochlorostyrenes were not detd

Refs: 1)Beil **5**, (231) 2)Beil **2**, [371] 3)P. Pfeiffer et al, Ber **47**, 1767(1914); JCS **106** I, 838(1914) & CA **8**, 2723-24(1914) 4)J. van der Lee, Rec **45**, 692(1926) & CA **21**, 399(1927) 5)D.E. Worrall, JACS **60**, 2845 (1938) & CA **33**, 1284(1939) 6)G.B. Butler & M.E. Carter, JACS **72**, 2303(1950) & CA **44**, 9927(1950) 7)R. Perrot & R. Berger, CR **235**, 187(1952) & CA **47**, 5908(1953)

Note: Higher nitro derivs of Chlorostyrene were not found in Beil or in CA thru 1956

Chlorotetrazole and Derivatives

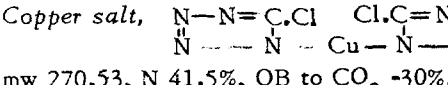
5-Chlorotetrazole(called 5-Chlor-tetrazol in Ger),



mw 104.50, N 53.59%; ndls(from benz), mp 73°, after softening begins to decomp at 120°; readily sol in eth, alc or w; was obtd by appropriate treatment of a diazotized soln of the copper salt; was not attacked by SO_2, Na_2SO_3 or NaOEt even on prolonged heating (Refs 1, 2 & 3). See Ref 4 for acidic props

It forms some expl salts, such as:

Copper salt,



mw 270.53, N 41.5%, OB to CO_2 -30%, OB to CO - 18%, d 2.04, mp - expl at 305° in 5 secs; was prepd by treating tetrazol-5-diazonium chloride with an aq $CuCl_2$ soln (Refs 1 & 2)

Properties: brisance by sand test 57% TNT & 53% when BkPdr fuse was used; density (loading) 1.49 at $10x10^3$psi, 1.63 at $20x10^3$ psi, 1.74 at $40x10^3$psi & 1.86 at $70x10^3$ psi; explosion temp - see mp; exudation-none; friction pendulum test - exploded by fiber or steel shoe; heat of expln 432 cal/g; heat test at 100°,% loss - 2.67 in 1st 48hrs. 0.10 in 2nd 48 hrs, no expln in 100 hrs; hygroscopicity 3.11% at 30° & 90% RH; impact sensitivity, 1-lb wt, PicArsnApp 3" with 9 mg sample (4" for MF); sensitivity to initiation - min detonating charge - 0.20 mg LA & 0.10 mg Tetryl; specific heat, cal/g/°C - 0.155 in temp range 0° -30° for 0.891 g sample; stab sensitivity (see Ref 11, p 61); storage - wet, hazard class (quantity - distance) 9 & compatibility group M(Refs 10 & 11)

Uses: as a primary expl (Ref 11, p 61). Investigation of its use in detonators was made by Wagner(Ref 9)

Silver salt, no formula given, lfts (from concd HNO_3); readily sol in NH_3; was obtd from an aq soln of 5-chlorotetrazole & $AgNO_3$ ammoniacal soln by pptn with concd HNO_3(Refs 1 & 2); and

Sodium salt, $NaClCN_4+1.5H_2O$, crysts(from hot w), mp explodes ca 253-55° depending on rate of heating; was prepd from a slurry of 5-aminotetrazole+H_2O, NaOH soln & $NaNO_2$ added to cold aq concd HCl, and isolated as the ethyl acetate insol residue(Ref 5)

Refs: 1)Beil **26**, [197] 2)R. Stollé et al, Ber **62**, 1123(1929); BrA **1929A**, 828 & CA **23**, 4471(1929) 3)F.R. Benson, ChemRevs **41**, 6 & 34(1947) 4)E. Lieber et al, JACS **73**, 1792 (1955) & CA **46**, 499(1952) 5)R.A. Henry & W.G. Finnegan, JACS **76**, 290(1954) & CA **49**, 12451(1955) 6)R.J. Gaughran & J.V.R. Kaufman, PATR **2136**(1955)(C), "Synthesis and Properties of Halotetrazole Salts" (U) 7) A.C. Forsyth et al, PATR **2146**(1955)(C), "Development of Optimum Explosive Trains. An Investigation Concerning Stab Sensitivity Versus Loading Density of Some Initiating Compounds" (U) 8)A.M. Anzalone et al, PATR **2179**(1955)(C) "Characteristics of Explosive Substances for Application in Ammunition" (U) 9)R.L. Wagner, SFAL, EDS Rept **17**, Project TA 1-2707BK(1957) 10)W.R. Tomlinson, Jr & O.E. Sheffield, PATR **1740**, Rev 1(1958) 11)Anon, "Properties of Explosives of Military Interest", Section 1, AMCP **706-111**(1963) (Revised edition of PATR **1740**)(Reprint of ORDP **20-177**)

5-Chloro-2-thenyl Chloride; 2-Chloromethylthiophene Chloride-(5) or 5-Chloro-2-(chloromethyl)-thiophene, $Cl.C-S-C-CH_2Cl$; $HC-CH$

mw 167.04; clear col liq, bp 95-7° at 15mm, undergoes spontaneous expl decompn; d 1.385 at 25°; n_D 1.5722 at 25°; was first prepd by Clapp et al(Ref 2) by chloromethylation of 2-chlorothiophene; similarly prepd by Cairns & McKusick (Ref 3) and by Kyrides et al (Ref 4) all of whom also studied its reactions. Rosenthal(Ref 5) conducted the chloromethylation in anhyd medium, using trioxane, chlorothiophene, $ZnCl_2$ & HCl at 0-5°. Norris(Ref 6) patented a procedure in which chlorothiophene, HCl & HCHO were heated 2 hrs at 40° at 2 hrs at 60°. It was Rosenthal(Ref 6) who pointed out the spontaneous explosive nature of 5-chloro-2-thenyl chloride and noted that it may be safely stored by placing a loosely stoppered vessel contg this liq within a metal container in a refrigerator

Note: See also 2-Chloromethylthiophene

Refs: 1)Beil - not found 2)R.C. Clapp et al, JACS **69**, 1549(1947) & CA **41**, 5874(1947) 3) T.L. Cairns & B.C. McKusick, JOC **15**, 790-91 (1950) & CA **45**, 1112(1951) 4)L.P. Kyrides et al, JACS **72**, 746(1950) & CA **45**, 2930(1951) 5)N.A. Rosenthal, JACS **73**, 5903(1951) & CA **46**, 11177(1952) 6)H.D. Norris, USP 2623049 (1952) & CA **47**, 9365(1953)

Chlorotoluene and Derivatives

Chlorotoluene or Chloromethylbenzene (called Chlor-toluol or Chlor-methylbenzol in Ger), $Cl.C_6H_4.CH_3$; mw 91.13. The 2-Chloro- (Ref 1), 3-Chloro- (Ref 2), and 4-Chloro- (Ref 3) toluenes are described in the literature (Refs 1,2,3 & 4)

Note: See also ω-Chlorotoluene or Benzyl Chloride, Vol **2**, p B95-R

Refs: 1)Beil **5**, 290, (149) & [224] 2)Beil **5**, 291, (150) & [226] 3)Beil **5**, 292, (150) & [226] 4)Sax(1957), 482-83

Azido, $N_3(Cl).C_6H_3.CH_3$, and *Diazido,* $(N_3)_2$-$(Cl).C_6H_2.CH_3$, derivs of *Chlorotoluene* were not found in Beil or in CA thru 1956

Nitrosochlorotoluene, $ON(Cl).C_6H_3.CH_3$, not found in Beil or in CA thru 1956

Dinitrosochlorotoluene, $C_7H_5ClN_2O_2$; mw 184.58, N 15.18%. A deriv of uncertain structure, called *2,5-Dinitroso-4-chlorotoluene,* $O.N:C_6H_2Cl(CH_3):N.O$ (?), lemon-yel powder, mp dec at 163-65°; insol in w or in alkalies; obtd by oxidn of 5-chlorotoluene-dioxime in dil alk soln with potassium ferric cyanide $[K_3Fe(CN)_6]$, was reported by Kehrmann et al (Refs 1 & 2). This compd could not be recrystd w/o decomp; it was converted by reducing agents into the dioxime & then the diamine; with concd HNO_3, it gave the dinitro compd (qv)

Refs: 1)Beil **7**, (353) 2)F. Kehrmann et al, Ber **48**, 2029(1915) & CA **10**, 767(1916)

Mononitrochlorotoluene, $O_2N(Cl).C_6H_3.CH_3$; mw 171.57, N 8.15%. All possible isomers (10) are known and described in the literature: 2-Nitro- 3-, 4-, 5- & 6- chlorotoluenes(Ref 1); 3-Nitro- 2-, 4-, 5- & 6- chlorotoluenes (Ref 2); and 4-Nitro- 2- & 3- chlorotoluenes(Ref 3). All are low melting cryst solids, except 3-Nitro-4-chloro- & 4-Nitro-3-chlorotoluenes which are definitely liquids at RT

Refs: 1)Beil **5**, 327, (162) & [251] 2)Beil **5**, 328-29, (163) & [252] 3)Beil **5**, 329, (163) & [253]

Dinitrochlorotoluene, $(O_2N)_2(Cl).C_6H_2.CH_3$; mw 261.58, N 12.33%. The following isomers are described in Beil:

2,3-Dinitro-4-chlorotoluene, yel prisms(from benz + petr eth), mp 106.5°(Ref 1, p [262]);

2,3-Dinitro-6-chlorotoluene, ndls or prisms(from MeOH or EtOH), mp 106.5°(Ref 1);

2,4-Dinitro-3-chlorotoluene, yel ndls(from alc), mp 92°(Ref 1,p [262]);

2,4-Dinitro-5-chlorotoluene, yel ndls(from alc), mp 91°(Ref 2). See also Ref 5;

2,4-Dinitro-6-chlorotoluene, prisms(from alc), mp 49°(Ref 2,p [263]);

2,5-Dinitro-4-chlorotoluene, yel prisms(from petr eth) or ndls(from alc), mp 101-7°(Ref 2);

2,5-Dinitro-6-chlorotoluene, ndls(from petr eth), mp 61.5-63°(Ref 2, p [263]);

2,6-Dinitro-3-chlorotoluene, lt yel ndls(from alc), mp 75-7° {Ref 2, pp (168) & [263]};

2,6-Dinitro-4-chlorotoluene, yel ndls(from eth), mp 76-7°(Ref 2,p 344);

3,4-Dinitro-2-chlorotoluene, ndls(from AcOH), mp 89°(Ref 2,p [263]);

3,4-Dinitro-6-chlorotoluene, lfts(from cold alc) or ndls(from warm alc or dil AcOH), mp 87-88.5° (Ref 3,p [264]);

3,5-Dinitro-2-chlorotoluene, lt yel ndls or pltlts(from alc) or prisms(from alc + eth), mp 63-5°(Ref 3);

3,5-Dinitro-4-chlorotoluene, lt yel ndls or short prisms(from alc), mp 114.5-116°(Ref 3); *x,x-Dinitro-2-chlorotoluene,* listed in Beil **5**, [264] but later considered to be probably 3,5-Dinitro-2-chloro-4-methylacetophenone (Ref Beil **7**, [940]). See Ref 5; *2,4-Dinitro-1' (or ω)-chlorotoluene or 2,4-Dinitro-benzylchloride,* $(O_2N)_2C_6H_3.CH_2.Cl$, pltlts(from eth), mp 32-4°(Ref 2, pp 344 & [263]). *Note:* See also ω-Chlorotoluene or Benzyl Chloride, Vol **2**,p B95-R, for other derivs *ω,ω-Dinitro-4-chlorotoluene or (4-Chlorophenyl)-dinitro-methane,* $Cl.C_6H_4.CH(NO_2)_2$ or $Cl.C_6H_4.C(NO_2):NO_2H$, ndls(from petr eth), mp 55°(dec); readily sol in hot solvs, mod sol in cold alc, eth, acet, chlf or benz; diffc sol in cold petr eth; was obtd by action of nitrogen peroxide on 4-chlorobenzaldoxime. Its *Potassium salt,* $KC_7H_4ClN_2O_4$, orn-yel prisms (from w), mp explodes on heating; readily sol in warm w giving a red colored soln; v sl sol in alc; insol in other solvs(Ref 3,p [264]; Ref 4)

Other props & methods of prepg the above Dinitrochlorotoluenes are given in the Refs indicated. Their expl props were not detd
Refs: 1)Beil **5**, 344 & [262] 2)Beil **5**, 344, (168) & [263] 3)Beil **5**, 345, (169) & [264] 4)G. Ruggeri, Gazz **53**, 695(1923) & JCS **126** I, 53-4(1924) 5)W. Qvist & M. Moilanen, Acta-AcadAboensis, MathPhys **14**, No 3, 9 pp(1943); ChemZtr **1943** II, 1268-69 & CA **38**, 5492(1944) [By nitration of 3-chloro-p-cymene and repeated crystn from AcOH, there was obtd *2,4-Dinitro-5-chlorotoluene* (qv), mp 90-90.5°; this compd also resulted by reaction of 3-chlorotoluene with fuming HNO_3 & concd H_2SO_4 and crystn from 85% AcOH. This compd is reported identical with the supposed 3,5-Dinitro-2-chloro-4-methylacetophenone or N. Ganguly & R. LeFèvre, CA **28**, 5416(1934). Q & M report that the latter compd & other derivs of G & LeF have not been prepd and should be removed from the chemical literature]

2,4,6-Trinitro-3-chlorotoluene, $(O_2N)_3(Cl).C_6H.CH_3$; mw 261.58, N 16.07%; wh ndls (from benz + petr eth), mp 148.5-151°; readily sol in AcOH, benz, acet or chlf; less sol in alc, eth or petr eth; was prepd by nitrating, with mixed acids, either 2,4-dinitro-5-chlorotoluene or 3-chlorotoluene(Refs 1 & 3) and by other methods. See Ref 4 if original is available. Its expl props were not detd
Refs: 1)Beil **5**, 349 2)K.L. Ganguly, Ber **58B**, 708-12(1925) [Compd listed in Collective Formula Index **14-40**(1920-46), p 162 but not found in reference abstract CA **19**, 2037(1925) or in original Ref] 3)W. Qvist & M. Moilanen, ActaAcadAboensis, MathPhys **14**, No 3, 9 pp (1943); Chem Ztr **1943** II, 1268-69 & CA **38**, 5491(1944) 4)S.S. Joshi & D.S. Deorha, JIndianChemSoc **29**, 46-8(1952) [Compd listed in 5th Decennial Formula Index **41-50** (1947-56), p 220F but not found in reference abstract CA **47**, 2731(1953)]

2,4,6-Trinitro-ω-chlorotoluene. See 2,4,6-Trinitro-benzyl Chloride under Benzyl Chloride and Derivatives, Vol **2**,p B95-R

Chlorotripentamminecobalt (III) Nitrate. See p A279 in Vol 1, under Ammines

1-Chloro-2,4,6-trinitro-2,4,6-triazaheptane or N-(Chloromethyl)-N'-[(Methylnitramino)-methyl]-N,N'-dinitromethanediamine, $CH_3.N(NO_2).CH_2.N(NO_2).CH_2.N(NO_2).CH_2.Cl$; mw 272.61, N 30.83%; wh solid(from $C_2H_4Cl_2$), mp 140.7-141.7°; was prepd from 1-trifluoroacetoxy-2,4,6-trinitro-2,4,6-triazaheptane in anhyd dioxane & anhyd HCl when allowed to stand at RT for 5 days; was similarly obtd using dioxane or tetrahydrofuran as the solv(Ref 3). Its UV absorption spectra were detd by Jones & Thorn(Ref 2) from analytically pure samples supplied by a university source, but no method of prepn was given. The expl props of this compd have not been reported
Refs: 1)Beil - not found 2)R.N. Jones & G.D. Thorn, CanJRes **27B**, 830 & 835(1949) & CA **44**, 2848(1950) 3)R. Reed, Jr., JACS **78**, 804 (1956) & CA **50**, 12864(1956)

Chlorous Acid(called Chlorige Säure in Ger), $HClO_2$, mw 68.47. This acid, like hypochlorous (HClO) and chloric ($HClO_3$) acids, has never been isolated. It exists only in aq solns, or as salts calledChlorites(qv). Chlorous acid may be considered as a deriv of the unknown anhydride Cl_2O_3. It was first prepd in an impure state by M. Martens in 1836 and by N.A.E. Millon in 1841 by dissolving ClO_2 in water. A better method of prepn is to treat Ba chlorite with an equiv amt of dil H_2SO_4; after filtering off the

pptd $BaSO_4$, the resulting filtrate contains aq chlorous acid free from chloric acid

Chlorous acid is a fairly strong oxidg agent. It is unstable, however, even at RT decompg as follows:

$4HClO_2 = 3ClO_2 + \frac{1}{2}Cl_2 + 2H_2O$,

and turning yel in color. As judged by the conductivity of its aq solns, it is a weak acid. Its ionization constant is 1.1×10^{-2} which is about the same as for oxalic acid or chloroacetic acid(Ref 5). For more detailed description of its props, See Ref 2

Various methods for determining the ClO_2 ion are given in Ref 2, p 306

Refs: 1)Mellor **2**(1922), 281 2)Gmelin, Syst Nr **6**(1927), 296 & 306 3)Mellor(1939), 512 4)Hackh's(1944), 195 5)Kirk & Othmer **3**(1949), 696 6)Partington(1950), 272 7)Ullmann **5** (1954), 520

Chloroxylene and Derivatives

Chloroxylene, Chlorodimethylbenzene, Xylyl Chloride or Methylbenzyl Chloride (called Chlor-xylol, Chlor-dimethyl-benzol or ω-Chlor-xylol in Ger), C_8H_9Cl, mw 140.60. Nine isomers are described in Beil(Ref 1) and three are listed in Sax(Ref 2)

Refs: 1)Beil **5**, 363, 364, 373, 384, (180, 183, 186,) & [283, 291, 299] 2)Sax(1957), 1263

Mononitrochloroxylene or Mononitromethylbenzyl Chloride, $C_8H_8ClNO_2$; mw 185.60, N 7.55%. Nine isomers of formula $CH_3(NO_2).C_6H_2.(Cl)CH_3$ (Ref 1), and two isomers of Nitro-ω'-chloroxylene, $CH_3(NO_2).C_6H_3.CH_2Cl$(Ref 2), are described in the literature

Refs: 1)Beil **5**, , 368, 378, 379, (184) & [286, 295] 2)Beil **5**, [286 & 302]

Dinitrochloroxylene or Dinitromethylbenzyl Chloride, $C_8H_7ClN_2O_4$; mw 230.61, N 12.15%. Only one isomer is known and described in the literature:

4,5-Dinitro-6-chloro-m-xylene, $CH_3(NO_2)_2.C_6H.(Cl)CH_3$, wh crysts(from alc), mp 61°, bp 290-91° & 178° at 27mm; was prepd from 5,6-dinitro-4-amino-1,3-dimethylbenzene thru diazotization & treatment with cuprous chloride (Refs 1 & 2)

Refs: 1)Beil **5**, 380 2)A.Klages, Ber **29**, 313 (1896) & JCS **70** I, 291(1896)

Trinitrochloroxylene, $C_8H_6ClN_3O_6$; mw 275.61, N 15.25%. Two isomers are known:

2,4,5-Trinitro-6-chloro-m-xylene, $CH_3(NO_2)_3.C_6(Cl)CH_3$, crysts, mp 165°; prepd by action of HNO_3 on 5-nitro-4-chloro-m-xylene (Refs 1 & 4); and

2,4,6-Trinitro-5-chloro-m-xylene, $CH_3(NO_2)_3.C_6(Cl)CH_3$, ndls(from alc), mp 218°; diffc sol in common solvs; decompd by alc KOH; was prepd from 5-chloro-1,3 dimethylcyclohexa-3,5-dieme in fuming HNO_3 at RT by treating the soln with equal parts of fuming HNO_3 & H_2SO_4 and refluxing for ½ hr(Refs 1 & 2), and by other methods(Refs 1 & 3)

The expl props of the above trinitrochloroxylenes were not detd

Refs: 1)Beil **5**, 381 2)A. Klages & E. Knoevenagel, Ber **28**, 2046(1895) & JCS **68** I, 654(1895) 3)C.L. Jackson & P.S. Smith, AmChemJ **32**, 181(1904) & JCS **86** I, 803(1904) 4)J.J. Blanksma, Rec **25**, 179(1906)

Chlorozon or "Essence de Boulogne". A disinfectant and bleaching soln consisting of free HClO, NaCl, and small quantities of $NaClO_3$ & chlorine, all dissolved in water

Ref: Gmelin, Syst Nr **6** (1927), 6

Choc, Essai au (Sensibilité au choc; Essai au) (Fr.).Sensitivity to Shock (or Impact) Test. See Impact Sensitivity or Shock Sensitivity Test in Vol 1,p XVII of Encyclopedia

Choc des balles; Essai au(Fr.). Rifle Bullet Test. See Vol 2 of Encyclopedia under BULLET TESTS

Choc, L'onde de(Fr.),Shock Wave. See under Detonation and Shock Waves

Chocolate Powder. Same as Cocoa Powder described in Vol 2,p B173-L

Choke. In shotguns, it is a narrowing in the bore towards the muzzle end of gun in order to limit the speed of the shot(Ref 1)(See also *choke-bore*). In pyrotechnic terminology, it is a constricted nozzle for the discharge of the propulsive gas from a fireworks rocket. In Brit rocket terminology, choke is the restricted opening that performs the function of a metering orifice in a proplnt feed system (Ref 2,p 88). The term choke was previously used for that part of a rocket propulsion unit now known as DeLaval or converging-diverging

nozzle(Ref 2,pp 88 & 112-114)
Refs: 1)Glossary of Ord(1959), 69 2)Rocket Encycl(1959), 88 & 112-14

Choke Bore(Artillery). The gradual diminution in the caliber of a gun due to the deposition of copper from the driving band or to other causes. This term is also used to signify the constriction in a shotgun, near the muzzle end(See also Choke)
Ref: Chamber's Technical Dictionary, Macmillan NY(1954), 158

Choke Bore Powders. BkPdrs formerly manufd by the DuPont Co for use in sporting guns
Ref: Daniel(1902), 142 & 210

Choke Ring. Metal ring used in the reaction chambers of certain recoilless weapons to control the escape of chamber gases
Ref: Glossary of Ord(1959), 69

Choking. A common expression for use of the constricted nozzle("choke") to control the flow of gas from a rocket engine thrust chamber by building up pressure inside the chamber until the upper limit of mass flow is reached, or when the speed of sound is reached in the duct
Refs: 1)Glossary of Ord(1959), 69 2)Rocket Encycl(1959), 88

Choking Gas. A chemical agent(qv) which causes irritation and inflammation of the bronchial tubes and lungs
Ref: Glossary of Ord(1959), 69

Cholesterol, $C_{27}H_{45}OH$. A monatomic alc found in the blood, brain tissue, etc; pearly scales, mp 148°, d 1.067; insol in w; sol in alc, eth, chlf or acet(Ref 2). Caution is advised(Ref 4) in detg cholesterol by the method of Pearson et al(Ref 3) as explns can occur after addn of the p-toluenesulfonic acid
Refs: 1)Beil - not found 2)Hackh's(1944), 197 3)S. Pearson et al, AnalChem **25**, 813-14 (1953) & CA **47**, 9391(1953) 4)B.J. Jones & F.B. Moreland, ClinChem **1**, 345(1955) & CA **50**, 1529(1956)

Choline(Bilineurine or Trimethylethanol Ammonium Hydroxide), $(CH_3)_3.N(OH).CH_2.CH_2.OH$; mw 121.18, N 11.56%; viscous liq occurring in many animal & vegetable tissues; sol in w or alc(Refs 1 & 6)

Choline forms numerous salts, some of which are more or less explosive
Choline Perchlorate, $C_5H_{14}NO.ClO_4$; crysts (from alc or w), mp 273°; obtained by treating choline with 70% perchloric acid(Refs 2 & 5). Its expl props were not detd
Choline Picrate, $C_5H_{14}NO.C_6H(OH)(NO_2)_3$, yel ndls, mp 248-9° (dec)(Refs 3 & 4). Its expl props were not detd
Choline Nitratoperchlorate, $C_3H_9N(ClO_4).CH_2.CH_2.ONO_2$; mw 248.62, N 11.27%, Cl 14.26%; long,thin, rectangular, lustrous plates; mp 185-6°; explodes strongly at higher temps; sl sol in w; obtained by heating choline perchlorate on a water-bath with a small amt of 65% HNO_3, followed by crystn from w(Ref 5)
Refs: 1)Beil **4**, 277, (425) & [720] 2)Beil **4**, (427) 3)Beil **4**, (427) & [723] 4)Beil **6**, (137) & [276] 5)K.A. Hofmann & K. Höbold, Ber **44**, 1766-7(1911) & CA **5**, 3257(1911) 6)Hackh's (1944), 197

Chondrin. A transparent, yel gelatinous mass obtained from cartilage. It is a protein considered to be a mixt of mucin & gelatin(Ref 2). It is sol in hot water, and was used for impregnating "dopes" of dynamites(Ref 1)
(See also Burstenberger Explosives in Vol 2 of this Encyclopedia)
Refs: 1)Daniel(1902), 89 2)Hackh's(1944), 197

Chromammonit. Ger for Chromeammonite(qv)

CHROMATES, DICHROMATES, TRICHROMATES AND TETRACHROMATES

CHROMATES are salts of dibasic *chromic acid,* H_2CrO_4, which is known only in solns. Chromates decomp on heating, giving 3 oxygens per 2 moles of chromate: $2M_2CrO_4 = Cr_2O_3 + 2M_2O + 3O$, where M = monovalent metal. For this reason chromates might serve as oxidizing agents

The primary material for prepn of industrial inorganic chromates is Chromite(chrome-iron ore). For example in the prepn of $CaCrO_4$, the finely ground ore is mixed with $CaCO_3$ and roasted in an oxidizing atmosphere. The roasted mass is treated with water. This

dissolves $CaCrO_4$, leaving a ppt of iron and aluminum oxide

For the prepn of other chromates, such as $PbCrO_4$, a soluble salt of Pb is mixed with $CaCrO_4$ and the resulting $PbCrO_4$ is pptd (See also under individual chromates)

Some chromates are used in expl, and pyrotechnic compns. Discussions on their uses in pyrotechnic heating devices, pyrochemical gas generators and other pyro items are given in Ellern, Pyrotechnics(1961). 137, 147, 160, 172, 173 & 265

Chromates, Analytical Procedures

A)Qualitative Tests: a)A very delicate test for chromate in soln is to add hydrogen peroxide and a little ether and shake in a test tube. A blue color in the ether layer indicates the presence of Cr ion in the form of unstable perchromic acid. For an insol chromate some nitric acid is added together with peroxide (Ref 1,p 282) b)Strong acid solns of chromates react with diphenylcarbazide, $OC(HN.NH.C_6H_5)_2$, to give a violet coloration even though traces of chromate ion are present(Ref 3). For this test 5 ml of a chromate soln is treated with 2 drops of HCl or AcOH, followed by 1 drop of DPhCbz reagent(which is prepd by dissolving 0.2g of DPhCbz in 5 ml of glac AcOH and\ diluting to 20 ml with alc)(Ref 1,p 283) c)In the absence of interfering ions, chromates can be detected by a spot test thru the formation of red Ag chromate or yellow Pb chromate(Ref 3,p 164) d)In phosphoric acid solns chromates oxidize pyrrole to pyrrole blue(Ref 3,p 164) e)Chromates react with 1% soln of strychnine in concd sulfuric acid to give a blue-violet to red color(Ref 3,p 164) f) Chromates in neutral or slightly acidified (with AcOH) solns give yel ppts when treated Ba or Pb acetate, or Ba chloride; red ppts form when chromate solns are treated with Ag nitrate or mercurous nitrate(Ref 1,p 282) g)Reducing agents: H_2S, H_2SO_3 or ferrous salts, form,when added to chromates in acid soln, green chromic compds(Ref 1,p 282)

B)Quantitative Tests: a)Small amts of chromates can be detd by diphenylcarbazide method as described in Ref 1,p 290; or in Ref 3,p 791; or by colorimetric method developed by Dittrich and described in Ref 1,pp 289-90 b)Large amts of chromates may be detd gravimetrically by treating a neutral or slightly acidified(with AcOH) soln of a chromate with Ba acetate or chromate, filtering the ppt of $BaCrO_4$, washing it and gently igniting before weighing(Ref 1,p 287; Ref 2,pp 164-5 and Ref 4, p 522; See also Ref 5) c)Another gravimetric method uses mercurous nitrate to ppt chromate (or dichromate) ion from neutral or faintly acidic soln as a brown, amorphous basic mercurous chromate(Ref 2, p 164 & Ref 4,pp 521-2) d)Chromate ion may also be detd volumetrically either by potassium iodide method or by ferrous sulfate method. In the former method, a chromate is reduced in acid soln by addg solid KI and the liberated iodine is titrated with a std soln of Na thiosulfate (Ref 1,p 288), whereas in the latter method, a chromate is reduced in dil sulfuric acid soln with ferrous sulfate and the excess titrated with std soln of K permanganate or K dichromate (Ref 1,pp 288-9)

Refs: 1)Scott & Furman 1(1939) 2)Treadwell & Hall 2(1942), 164-5 3)Feigl 1(1954), 159 & 164 4)Vogel, InorgAnalysis(1961), 520-3 & 791 5)Std MethodsChemAnalysis 1(1962), 350-76

List of Chromates

p-Aminobenzenediazonium Chromate, $H_2N.C_6H_4.N_2.HCrO_4$. See vol 1,p A187-L of this Encyclopedia

Ammonium Chromate, $(NH_4)_2CrO_4$, mw 152.09; yel monocl crysts; mp dec at 180°(Ref 8); can be exploded by rapid heating or shock(Ref 6); d 1.917 at 12°(Ref 8); d 1.866(Ref 9); 100g of cold w dissolves 40.5g at 30° and hot w decomposes it; sl sol in acet; insol in alc. Can be prepd by the double decompn of Ba chromate with Amm sulfate or by neutralization of aq chromic acid with ammonia, followed by evapn of w over quicklime(See also Refs 1a, 2, 3, 4, 5, 9 & 10). Its toxicity and fire hazard are discussed in Ref 6

Ammonium chromate has been used in some commercial expls(Ref 1a). Colver(Ref 1) gives the following expl compn: Amm picrate 55,K (or Na) picrate 25 & Amm chromate 20%. Ellern(Ref 7) discusses the possibility of its use as a catalyst in pyrochemical gas generators. Other uses of Amm chromate are discussed in Refs 4 & 9

Refs: 1)Colver(1918), 699 1a)C.A.Taylor &

W.H. Rinkenbach, Explosives; Their Materials, Constitution and Analysis", USBurMinesBull **219**, USGovtPtgOff, Washington, DC(1923), 48 2)Mellor **11**(1931), 241 3)Thorpe **3**(1939), 111 4)Kirk & Othmer **3**(1949), 946 5)Ullmann **5** (1953), 585 6)Sax(1957), 277 7)Ellern, Pyrotechnics(1961), 173 8)Lange(1961), 216-17 9)CondChemDict(1961), 72 10)Gmelin, Syst Nr **52**(1962ff)

Ammonium Chromate, Analytical Procedures. Chromate ion may be detected and detd as described under Chromates, Analytical Procedures

Ammonium ion may be detected by heating in a test tube a soln of Amm chromate with a strong base, such as Na, K or Ca hydroxides. The odor of ammonia resulting from such treatment indicates the presence of NH_4 ion. If a strip of moist red litmus paper is held over the neck of test tube, it should turn blue (Ref 1,p 630). Amm ion may also be detected by treating a soln of sample with Nessler's reagent. A yel or reddish-brn coloration or brn ppt is produced accdg to the amt of Amm present(Ref 1,p 630 & Footnote 2)

For *quantitative* detn of Amm ion, the following methods may be used: a)Kjeldahl Method(See Ref 1,pp 632-5; Ref 2,pp 493-5 & Ref 3,pp 256-7) b)Direct Method in which a soln of sample is heated with concd soln of strong base(such as NaOH) in a distillation flask and the expelled ammonia is collected in a flask contg an excess of standard acid. Then the excess of acid is titrated with std NaOH or KOH soln(Ref 1,p 637; Ref 2,p 493 & Ref 3,pp 254-5) c)Indirect Method, in which the sample is boiled in a flask with a known excess of NaOH soln until all NH_3 is expelled with the steam. Then the flask is cooled and the excess of NaOH is titrated with std acid(Ref 3, p 255)

There is no US Military Specification

Refs: 1)Scott & Furman **1**(1939), 630-35 2)Treadwell & Hall **2**(1942), 493-5 3)Vogel, InorgAnalysis(1961), 254-7 4)StdMethodsChem Analysis **1**(1962), 736 & 744-45

Barium Chromate (Lemon Chrome or Baryta Yellow), $BaCrO_4$, mw 253.37; heavy yel, rhombic crysts; very poisonous; mp - may be heated to 1000° without melting or decompg (Ref 2); d 4.498 at 15°; nearly insol in w; sol in acids(Ref 14). Can be prepd by the interaction of Ba chloride and Na chromate or by other methods(Ref 1,3,7,9,12,14 & 15). Its toxicity & fire hazard are discussed in Refs 11 & 12. It is a powerful oxidizer and reacts vigorously with reducing materials. It has been used in paints, ceramics, matches and pyrotechnics(Refs 8,13 & 16) but its main use is as a constituent of corrosion-proof pigments employed in construction of aircraft(Ref 7a)

Hart(Ref 4), patented gasless delay compns for fuzes contg insol chromates(such as of Ba or Sr) as oxidizers and powdered Zr(or Mn) and S(or red P) as fuels. Eg: a)$BaCrO_4$ 74, Mn 24 & P 2% and b)$BaCrO_4$ 90, Zr 5 & S 5%. These powders were claimed to give better performance than the compn described in Ref 3, listed under Lead Chromate. Bennett & Dubin(Ref 5) patented some gasless delay compns, such as $BaCrO_4$ 42-72, Zr(pdr) 4.5-3.0, Ni(pdr) 31-15 & $KClO_4$ 22-7%. $BaCrO_4$ may be replaced by $PbCrO_4$, MnO_2, PbO or Pb_3O_4 and $KClO_4$ by $KClO_3$. Hale & Hart(Ref 6) patented some gasless powders contg insol chromates(such as of Ba, Ca, Sr or Ag) as oxidizers and red P as a fuel. Eg: $BaCrO_4$ 96 & P 4%. More recently Hart(Ref 10) patented gasless delay compns contg $BaCrO_4$ & $KClO_4$ with dichromated powdered alloys Zr/Ni or Ti/Ni. The following pyrotechnic compns based on Ba chromate are listed by Ellern (Ref 13): a)Gasless Ignition Mixture - $BaCrO_4$ 90 & B(amorphous)10%(Formula 74, p 282) b)Delay Composition - $BaCrO_4$ 31, Zr/Ni 54 & $KClO_4$ 15%(Formula 77,p 283) c)Delay Compositions-$BaCrO_4$ 95 & B(amorphous) 5%(Formula 77,p 283) d)Delay Compositons See Formulas 79,80 & 81, under Lead Chromate and e)"Exotic" Delay Mixtures a)$BaCrO_4$ 82-85 & Niobium(Columbium) 18-15% b)$BaCrO_4$ 50 & Niobium(or Tantalum) 50% c)$BaCrO_4$ 71 & Tantalum 50%

US Military requirements for Ba chromate are listed in Ref 16. There are two grades of chromates: *Grade A* - intended for use in the manuf of delay powder and other nongaseous powder and *Grade B*-intended for use in other pyrotechnic mixts

Following are the requirements:

Requirement	Grade A	Grade B
	(percent)	
Volatile matter, max	0.10	0.20
Purity($BaCrO_4$), min	98.5	99.0
Chlorides(as Cl), max	0.05	0.05
Moisture Reabsorption, max	–	0.20
Apparent Density, g/ml	-0.35-0.50	0.35-0.75
Granulation(US Std Sieves)		
Thru No 100, min	–	100
Thru No 200, min	–	98
Thru No 325, min	100	–
Average Particle Diameter, microns, max	2.0	3.5

In US Military Specification MIL-C-13739(Ord) are given compns and analyses of the following delay powders based on Ba chromate:

Composition, %	Types I	II	III
Ba chromate	60.0	60.0	60.0
K perchlorate	14.0	14.0	14.0
70/30-Zr/Ni alloy, powdered, Type I	26.0	9.0	3.0
70/30-Zr/Ni alloy, powdered, Type II	–	17.0	23.0
Average burning rate, sec/inch	2.0	5.0	12.0

Requirements and tests for Zr/Ni alloys are given in Spec MIL-Z-11410 and for K perchlorate in Spec JAN-P-217(2)
(See also Delay Compositions)
Refs: 1)Mellor **11**(1931), 271 2)B. Zagorchev, ZPhysChem(Leipzig) **182A**, 31(1938) 3)Thorpe **3**(1939), 111 4)G.C. Hale, USP 2450892(1948) & CA **43**, 408(1949) 5)O.G. Bennett & J. Dubin, USP 2457860(1949) & CA **43**, 2438(1949) 6)G.C. Hale & D. Hart, USP 2468061(1949) & CA **43**, 5189-90(1949) 7)Kirk & Othmer **3**(1949), 946-7 7a)Thorpe **9**(1949), 635 8)Kirk & Othmer **11** (1953), 323 9)Ullmann **5**(1953), 585 10)D. Hart, USP 2696429(1954) & CA **49**, 5845(1955) 11)Sax(1957), 331 12)CondChemDict(1961), 126 13)Ellern, Pyrotechnics(1961), 137, 144, 146-7,265 & 282-4 14)Lange(1961), 224-5 15)Gmelin, Syst Nr **52**(1962ff) 16)US Military Specification **MIL-B-550A** 17)Picatinny Arsenal Technical Reports, 844, 1239, 1406, 1432, 1512, 1513, 1546, 1686, 1733, 1952, 1976 & 2006

Barium Chromate, Analytical Procedures. Chromate ion may be detected & detd as outlined under Chromates, Analytical Procedures. Barium ion may be detected by heating a small amt of the salt on a platinum wire(or spatula) in a Bunsen burner flame and observing coloration; if Ba is present, the flame is yellowish -green, which appears blue thru a green glass (Ref 1,p 117)

Quantitative detn of Ba ion can be made either gravimetrically(such as pptn as Ba sulfate), or volumetrically(such as titration with std sulfate soln). These methods are described in Ref 1,pp 127 & 129 and Ref 3,p 554

In Ref 2a is described the following procedure for *Determination of Barium Chromate in US Delay Composition T10:*

Transfer a 2g sample, accurately weighed, into a 250 ml flask, add ca 10 ml distl w and 15ml concd HCl. Swirl beaker gently and cover with watch glass. When the reaction has subsided, heat the beaker on a hot plate and keep the soln boiling until complete dissoln of sample(ca 5 mins). Cool the beaker, wash down the cover and sides of beaker with 15-20 ml of w and filter the soln(together with washings) thru No 40 Whatman filter paper, collecting the liquid in an 800ml beaker. Wash thebeaker with several portions of hot w(total 50ml) and pass the washings thru filter into 800ml beaker contg the filtrate. Dilute it with w to ca 500ml, cover the beaker with a watch glass, place it on a hot plate and bring the soln to boiling. Add 50ml of 0.6N sulfuric acid and continue boiling for 5 mins. Transfer the beaker to a closed steam bath and leave for at least 30 mins(preferably overnight). Decant the supernatant liquid thru a tared Gooch crucible, retaining as much of the ppt in the beaker as possible. Wash the ppt in beaker with two 15ml portions of hot distd w and transfer the ppt to the crucible using a stream of cold distd w. After drying the ppt in the crucible for 1 hr at 100°, heat it for 45mins in a muffle furnace at 650°. Cool the crucible, first on an asbestos mat for ca 5 mins, and then in a desiccator. Det the weight of ppt and calculate: $\%BaCrO_4=(108.55 \times A)/(W \times F)$,
where: A=wt of ppt; W=wt of sample and F= purity of Ba chromate as detd in test B, described under Analysis of Barium Chromate, which follows

Analysis of Barium Chromate, which is intended

for use in US delay powders and pyrotechnic compns is described in Ref 5. The method includes the following tests:

A)Volatile Matter. Heat at 100-110° for 1 hr a 2g sample, accurately weighed, in a tared (previously dried at 110° and cooled) weighing dish ca 2" in diam, provided with a glass stopper. Cool in a desiccator and weigh

$$\% \text{ Volatiles} = [(A-B) \times 100] / (A-D),$$

where A = wt of undried sample in dry dish with glass stopper; B = same after drying the sample and D = wt of empty dish with glass stopper

Stopper the weighing dish and save the sample for the "Moisture Reabsorption Test"(See test E, which follows "Water Soluble Matter"

B) *Purity*($BaCrO_4$ content). Weigh accurately a 0.35g sample, transfer it to a 500ml Erlen fl, add 75 ml of distd w with 15ml of concd HCl and swirl to dissolve the sample. Add, with constant swirling ca 2g powdered Na carbonate, in small portions, followed by a soln of 10g KI(iodate-free) in 10ml distd w. Cover the flask with a watch glass to exclude air, and put in a dark place for 10 mins. Dilute to ca 350ml with w and titrate with 0.1N std Na thiosulfate soln until the brn color of liberated iodine begins to fade. Add 3-5ml of starch indicator soln(See Note below) and continue the titration until the blue color of the starch iodine disappears, leaving the grn color of chromic ion

$$\%BaCrO_4 = \frac{8.446 \times V \times N}{W \times (1-0.01 \times A)},$$

where: V = ml of thiosulfate soln; N = its normality; W = wt of sample and A = % of volatile matter

Note: Prepare starch soln by mixing 1g of soluble starch with 10g of red HgI_2 and enough cold w to make a thin paste. Dilute with ca 200 ml of w, boil 1 minute, while stirring, cool and keep in stoppered bottle(Ref 5,pp 3 - 4)

C)*Chlorides.* Shake a 10g powdered sample with 250ml of w in a 500ml Erlen fl, add a small amt of filter paper pulp and filter the soln thru filter(such as Whatman No 42 or equivalent) into a 600-ml beaker. Wash the residue on the filter with several portions of w and add the washings to the filtrate. Add 1 ml of a 5% K chromate soln and titrate with 0.1N Ag nitrate soln to a permanent faint blood-red tinge. Run a blank.

$$\%\text{Chlorides(as Cl)} = \frac{3.55 \times (V-B) \times N}{W}$$

where: V = ml of std Ag nitrate soln used for the sample; B = ml of Ag nitrate soln used for the blank; N = normality of Ag nitrate soln and W = wt of sample(Ref 5,p 4)

D) *Water Soluble Matter.* This test is conducted by detg the soly of sample in distd w previously satd with pure Ba chromate. For prepn of this satd w, boil for 5 mins ca 500 ml distd w contg ca 2g CP $BaCrO_4$ in a 600-ml beaker, cool to RT and filter the liq thru filter paper, such as Whatman No 42 or equivalent. Wash the residue with hot w, transfer to a clean 600-ml beaker, add 500ml hot w, boil for 5 mins, cool to RT and filter, as above. Refilter if filtrate is not quite clear. Transfer exactly 60 ml of combined filtrate to a dry, tared 100-ml beaker and evap first on a hot plate to a vol of ca 2 ml and then to dryness on a steam bath,taking care to prevent any mechanical loss. Dry the beaker and contents in an oven at 100-105° for 1 hr, cool in a desiccator and weigh. Total wt, minus wt of beaker is the wt of residue in 60 ml of liquid. Designate the wt of residue as B. Save the filtrate in Erlen fl for use as a "wash water" in detn of sol matter in specification grade Ba chromate

Procedure: Transfer 5 g of accurately weighed spec grade Ba chromate to a 150-ml beaker, add exactly 50ml of "wash water"(See above). Cover the beaker with a watch glass, heat to boiling and then continue to boil for 5 mins. Cool the mixt to RT and filter the decanted liq thru a filter paper, such as Whatman No 42(or equivalent), catching the filtrate in a tared 100-ml beaker. Refilter if the soln is not quite clear. Rinse the insol matter off filter twice with 5 ml portions of "wash water" and evap the combined w extract and rinsings (total, 60 ml) as described above. Dry the beaker with residue in an oven at 100-105° for 1 hr, cool in a desiccator and weigh. Designate the wt of residue as A

$$\% \text{ Water Soluble Matter} = \frac{100 \times (A-B)}{W}$$

where: A = wt of residue from sample; B = wt of residue from CP $BaCrO_4$ and W = wt of sample(5g)(Ref 5,p 4)

E) *Moisture Reabsorption Test.* Place the dried sample saved after detn of "Volatile Matter" (Procedure A), in a desiccator contg 27% sulfuric acid soln and, after 24hrs at 25°, reweigh. Calc the percent increase as moisture reabsorption(Ref 5,p 5)

The 27% sulfuric acid is prepd by adding slowly with cooling 153ml of H_2SO_4(d 1.84) to 1l of w

F)*Apparent Density.* It can be detd by means of a "Scott Volumeter", described in Ref 4,p 745. The procedure for US specification grade Ba chromate is briefly described in Ref 5,p 5. It is similar to the procedure used in paint industry(Ref 2)

G)*Granulation.* Place a 100g sample(previously dried at 100-105° for 20 mins and cooled) on the specified sieve which has been superimposed on an evaporating dish ca 9" in diam. Wash the material with w contg a small amt of wetting agent, using a camel's brush to facilitate the passage of particles thru the screen. When no more material will pass thru the screen, wash the residue with acetone, dry it in air and transfer to a tared watch glass. Dry in an oven at 100-105° for 20 mins, cool in a desiccator and weigh

Granulation in % thru = (1-A/W)x 100

where: A wt = of residue on sieve and W= wt of sample(Ref 5,p 5)

H) *Particle Size.* Det it by means of a "Fisher Subsieve Sizer"(Ref 4,p 848), in accordance with manufacturer's instructions

The apparatus and procedure were described in old(1948) US Specification JAN-B-550,pp 5 - 7 & 10 - 11

Refs: 1)Scott & Furman(1939), 117, 127, 129 & 286-91 1a)Treadwell & Halt, Vols 1 & 2(1942) 2)H.A. Gardner & G.G. Sward, "Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors", H.A. Gardner Laboratory, Inc, Bethesda, Md(1950) 402-3 2a)Anon, "Control Laboratory Manual", Proplnts & Expls Branch, PicArsn(1956), Method 504.1 3)Vogel, InorgAnalysis(1961), 520-2 & 554 - 4)Fisher Scientific Co, Catalog **63**, "Modern Laboratory Appliances", Pittsburgh, Pa(1962), 745 & 848 5)US Military Specification **MIL-B-550A**, pp 3 - 5 6)StdMethodsChemAnalysis, **1**(1962), 138-59 & 350 - 76

Calcium Chromate, $CaCrO_4$, $CaCrO_4.2H_2O$, mw 192.12; yel, monocl crysts; mp - loses $2H_2O$ at 200°; sol in w(22.2% at 0° & 18.2% at 43°); sol in acids; sl sol in alcohol. Can be prepd from chrome-iron ores as described under CHROMATES. Its toxicity is discussed in Ref 7. It exists also as monohydrate and as anhydrous salts(Refs 1, 3, 4, 6, 8 & 9)

Ca chromate has been used for prepn of other chromates, as a corrosion inhibitor and as a yel pigment in paints(Refs 4, 6 & 8). A pyrotechnic compn contg $CaCrO_4$ for an amber colored signal was described by Schladt (Ref 2). Hale & Hart(Ref 5) patented a gasless delay compn contg $CaCrO_4$ as an oxidizer and Zn(or Zr) and S(or red P) as fuels. Its use in corrosion - inhibiting cartridges for aircraft fuel tanks is discussed in Ref 4a

Refs: 1)Mellor **11**(1931), 267 2)G.J. Schladt, PATR **596**(1935) 3)Thorpe **3**(1939), 111 4)Kirk & Othmer **3**(1949), 947 4a)Thorpe **9**(1949), 635 5)G.C.Hale & D.Hart, USP 2468061(1949), & CA **43**, 589-90(1949) 6)Ullmann **5**(1953), 586 7)Sax (1957), 426 8)CondChemDict(1961), 200 9)Langel(1961), 232-3 10)Gmelin Syst Nr **52** (1962ff)

Calcium Chromate, Analytical Procedures. Chromate group may be detected and determined using the methods listed in Refs under Chromates Analytical Procedures. Calcium ion may be detected and detd as described in Refs 1 & 2

Refs: 1)Vogel, InorgAnalysis(1961), 294, 436-37, 473-77, 551-52 & 1117-18 2)StdMethods ChemAnalysis **1**(1962), 258-78

Note: As there is no US Military specification for Ca chromate and as its props are similar to those of Ba chromate, it is suggested to use analytical procedures given in Spec MIL-B-550A, applying factors suitable to $CaCrO_4$. These procedures are also described in this Vol under Barium Chromate

Cobalt(II) Chromate or Cobaltous Chromate, $CoCrO_4$, mw 174.95 grayish-blk pdr, mp decomp on heating; was prepd by 4-hr heating of $CoCO_3$, CrO_3 & some w in sealed tube at 185°(Refs 1,3 & 5). Its *dihydrate,* $CoCrO_4.2H_2O$, blk microscopic crysts were obtd on adding a cold aq soln of Na chromate to a mixt of $CoCO_3$ & CrO_3 in cold w(Refs 2 & 3). There is also *basic chromate,* which is available in commerce(Refs 3 & 6). Cobaltous chromate was tried at PicArsn as an ingredient of a pyrotechnic compn producing blue light. Its compn was: Co(II) chromate 25, K perchlorate 65 & Cu (II) resinate 10%; rate of burning 2.8 in/min and CP(candle power) only 40/sq in (Ref 4)

Refs: 1)S.H.C. Briggs, ZAnorgChem **63**, 327-28 (1909) 2)S.H.C. Briggs, JCS **1929**, 244 3) Gmelin, Syst Nr **58**, Teil A(1932), 479-80

4)G.J. Schladt, PATR 357(1933) Table IV 5)Sax(1957), 494 6)CondChemDict(1961), 287

Copper(II) Chromate, $CuCrO_4$, brn ppt, mp decomp above 325° and forms ca 650° *Cuprous chromite,* $CuCrO_4$, was obtained, accdg to Mellor (Ref 1) by J. Schulze on heating $Cu(OH)_2$ with an excess $K_2Cr_2O_7$ (in soln) in a sealed tube at 220°. Ullmann(Ref 2) states that $CuCrO_4$ can be prepd as yel-brn ppt on treating $CuSO_4$ with K_2CrO_4, both in soln. *Basic cupric chromate,* $CuCrO_4.2CuO.2H_2O$, light chocolate color pdr, losing w at 260°. Can be prepd by the action of chromic acid on Cu hydroxide (Ref 4). *Copper ammonium chromate* can be prepd by treating copper chromate with ammonia (Ref 2)

Copper chromates have been used as mordants in dying and in some pyrotechnic compns. For example a blue light rifle signal light compn developed at PicArsn(Ref 5) contained: Cu(II) chromate 25, K perchlorate 65 & Cu resinate 10%; it burned at the rate 3.5in/min developing CP 485/sq in

Refs: 1)Mellor **11**(1931), 260 2)Ullmann **5** (1954), 588 3)Sax(1957), 516(Toxicity & fire hazard of basic salt) 4)CondChemDict(1961), 301(Prepn & props of basic salt) 5)G.J. Schladt, PATR **357**(1933)

Lead Chromate *(Crocoite or Chrome Yellow),* $PbCrO_4$, mw 323.22; yel monocl crysts; mp 844° with decompn; d 6.12; practically insol in w & in ammonia; sol in acids & alkalies. Can be prepd by interaction of Na chromate soln with solns of Pb nitrate or acetate(Refs 2, 4, 8, 11, 13 & 14). An industrial method of prepn is given in Ref 6

Pb chromate is a powerful oxidizing agent which sometimes causes explns when in contact with org material. For example, Daniel (Ref 1) reported cases where org objects(such as paper lamp shades) covered with Pb chromate paint ignited spontaneously or even exploded. Toxicity & fire hazards are discussed in Ref 10

Uses: Pb chromate has been used as a yel pigment in paints and as a component of pyrotechnic compns & of delay compns in fuzes(Refs 7,9 & 12). It was also used in pyrotechnic smokes(Ref 4a,p 122) and in some older types expls, such as Cornil and Halakite. Hale(Ref 3) proposed the following delay compn for fuzes: Pb chromate 88, powdered Al or Si 11 & linseed oil 1%. Another delay compn of Hale contg Pb chromate is mentioned by Ellern(Ref 12, p 139). Bennett & Dubbin(Ref 5) patented some gasless delay compns, such as: Pb chromate(or Ba chromate) 42-72, Zr (powder) 4.5-3.0, Ni 31-15 & K perchlorate 22 - 7%. Burning rates 4.5 -15sec/in. Ellern (Ref 12,p 144) states that Pb chromate & K perchlorate have been employed successfully as burning rate accelerators. The following delay compns(Formulas 79,80 & 81) using $PbCrO_4$ are given on p 283 of Ref 12:

	Formulas		
	79	80	81
PbChromate, %	53	43	36
Ba Chromate, %	3	20	31
Mn(powder), %	44	37	33
Burning rate, sec/in	4	8	13

Davis(Ref 4a,p 458), lists a non-erosive primer compn: $PbCrO_4$ 40, MF 36, powd glass 4 & Sb_2S_3 20%. Hart (Ref 4b) developed the following smoke compn for fuze of practice chemical mine, T20: Se 65, Mg 10 & $PbCrO_4$ 25%. Shidlovskii(Ref 9) lists the following non-gaseous pyrotechnic compns: a)$PbCrO_4$ 40, PbO 44 & $FeSi_2$ 16% and b)$PbCrO_4$ 63, Pb_3O_4 25 & Si 12%; NC 0.3% added to serve as a binder

US Military requirements for $PbCrO_4$ intended for use in the manuf of non-gaseous powders are listed in Ref 15. The requirements are: A)Moisture, max 0.2% B)Purity, min 99.2% $PbCrO_4$ C)Water Soluble Matter, max 0.2% D)Grit, max 0.002% E)Apparent Density, min 0.90g/ml and F)Granulation, min 100% thru No 325 US Std Sieve

Refs: 1)Daniel(1902), 392 2)Mellor **11**(1931), 290-7 3)G.C. Hale, USP 1877127(1932) & CA **27**, 191(1933) 4)Thorpe **3**(1939), 11-13 4a)Davis(1943), 122 & 458 4b)D. Hart, PATR **1596**(1946) 5)O.G. Bennett & J. Dubin, USP 2457860(1949), & CA **43**, 2438(1949) 6)Kirk & Othmer **3**(1949), 947 7)Ibid **11**(1953), 323 8)Ullmann **5**(1953), 586 9)Shidlovskii(1954), 260 10)Sax(1957), 817 11)CondChemDict (1961), 658 12)Ellern, Pyrotechnics(1961), 139, 144, 147 & 283 13)Lange(1961), 262-3 14)Gmelin, Syst Nr **52**(1962ff) 15)US Joint Army-Navy Specification **JAN-L-488**

Lead Chromate, Analytical Procedures. Chromate group may be detected & detd using the methods listed under Chromate, Analytical

Procedures. These methods are described in Ref 1,pp 282-3 & 286-91; and Ref 2,pp 520-3. Lead ion may be detected and detd by one of the methods described in Ref 1,pp 500-527; and in Ref 2,pp 311, 362-3, 443, 482-6, 899-901 & 1039. Commercial analysis of lead chromate is described in Ref 1a(See also Ref 4)

Analysis of Lead Chromate, which is intended for use in US non-gaseous delay powders is described in Ref 3. The method includes the following tests:

A)*Moisture.* Heat a 5g sample in a tared dish at 100° for 2 hrs, cool in adesiccatorand weigh. Calc loss in wt as % moisture(Ref 3,p 2)

B)*Purity.* Dissolve ca 0.5g of sample,accurately weighed, by warming with 20ml of 20% NaOH in a glass stoppered 250ml bottle and dissolve in soln ca 2g of KI. Dilute with 100ml of w, add 15ml of concd HCl and, after allowing to stand for 5 mins while protected from light, titrate the liberated iodine with std 0.1N Na thiosulfate soln in the same manner as described in procedure B under Barium Chromate, Analytical Procedures

$$\%PbCrO_4 = (10.77 \times V \times N)/W,$$

where: V = ml of thiosulfate soln; N = its normality and W = wt of sample(Ref 3,p 2)

C)*Water soluble matter.* Add 100ml of boiling distd w to a 5g accurately weighed sample contained in a 250ml beaker and boil the mixt for 5 mins. Cool to RT, filter thru a tared sintered glass crucible and wash the residue with alc, followed by ether. After evapg the eth by suction, heat the crucible at 100° for 1 hr, cool in a desiccator and weigh. Calc the loss in wt as % soluble matter(Ref 3,p 2)

D)*Grit .* Stir a 50g sample with 200ml w in a beaker and pour the mixt thru a 3" tared No 170 US Std Sieve, both sides of which have been wetted with alc before using. Transfer any remaining insol matter from beaker to sieve and wash the residue on the sieve(while stirring it with a soft camel's hair brush) with stream of tap w until the w passing thru the sieve is clear and free from solid particles. Rinse the residue with distd w, followed by acetone, dry the sieve in a desiccator at 100° for one hr, cool and weigh. Transfer the residue to a smooth glass slide and rub the material with a steel spatula. The presence of grit is indicated by the persistence of a scratching sound and the appearance of scratches on the glass slide(Ref 3,p 3)

E) *Apparent Density.* Tare a 100ml graduated cylinder having graduations extending ca 7.5". Slowly pour 90 - 95 ml of the sample from a height of 12" from the base of the cylinder thru a funnel with a stem ca 1" long and 0.5" in diam, without jarring or shaking the cylinder. After levelling off the surface of material in the cylinder, take the reading and weigh the cylinder and contents. Calc the d by dividing the weight of $PbCrO_4$ in grams by its vol in mls (Ref 3,p 3)

This test may also be conducted by using the "Scott Volumeter" as mentioned in test F under Barium Chloride, Analytical Procedures

F)*Granulation.* Use US Std Sieve No 325 and procedure G described under Barium Chromate, Analytical Procedures(Ref 3,p 3)

Refs: 1)Scott & Furman(1939),pp 282-3, 286-91 & 500-27 1a)Snell & Biffen(1944), 529-32 2)Vogel, InorgAnalysis(1961),pp 311, 362-3, 482-6, 520-3, 899-901 & 1039 3)US Joint Army-Navy Spec **JAN-L-488** 4)StdMethods ChemAnalysis **1**(1962), 350-76 & 556-84

Magnesium Chromate, $MgCrO_4 \cdot 7H_2O$, mw 266.44; yel rhmb crysts; mp forms hexahydrate above 30°(Ref 2) and tetrahydrate ca 120° (Ref 4); d 1.761(Ref 2); d 1.695(Refs 3 & 4); very sol in w. Can be prepd either by crystn from a soln of magnesia in aq chromic acid (Ref 1) or by interaction of Mg sulfate with Ca chromate in w(Ref 2). Being an oxidizer it can react with reducing materials. Its fire hazard and toxicity are discussed by Sax (Ref 3)

As hydrated Mg chromates combine the props of both oxidizing and cooling agents, their use as possible components of permissible coal mining expls is suggested by the Authors

Refs: 1)Mellor **11**(1931), 274-5 2)Ullmann **5** (1953), 587 3)Sax(1957), 842 4)Lange(1961), 266-7 5)Gmelin Syst Nr **52**(1962ff)

Methylmercuric Chromate. See Methylmercuric Salts under M

Potassium Chromate(Potassium Chromate, Yellow or Tarapacaite), K_2CrO_4, mw 194.21; yel rhmb crysts(beta or ordinary form) transforms at 666-7° to hexagonal(alpha) form which melts at ca 997°(Ref 4), 984°(Ref 5) and 971°(Ref 6); density 2.715(Ref 4), 2.74(Ref 5), 2.732(Ref 6) and 2.70(Ref 8); soly in w at RT 39% and at 100° 45%(See also Ref 5). Can be prepd by

interaction of Ca chromate with K carbonate in w, by treating K dichromate with K carbonate or by roasting the powdered chromite with K carbonate & limestone(Refs 1,2,4,5 & 6a). Its toxicity and fire hazard are discussed in Ref 6. Its industrial uses are very limited because, in most cases, it can be replaced by the cheaper Na salt(Ref 4 & 5); has been used as an oxidizing agent in some expl compns, as for example Borlinetto Powder(qv)(See also Refs 3 & 4a)

Refs: 1)Mellor **11**(1931), 249 2)Thorpe **3**(1939) 110 3)Davis(1943), 166 4)Kirk & Othmer **3** (1949), 947 4a)W.H. Hartford, IEC **41**, 1993-7 (1949)(Props of K_2CrO_4) 5)Ullmann **5**(1953), 587 6)CondChemDict(1961), 928 6a)Mellor (1961), 870 & 872 7)Lange(1961), 290-1 8)Gmelin, Syst Nr **52**(1962ff)

Silver Chromate, Ag_2CrO_4, mw 331.77; dk red monocl crysts; mp decomp; d 5.625 at 25°; can be prepd by interaction of Ag nitrate with K chromate in w. Its toxicity and fire hazard are discussed in Ref 5

Use of Ag chromate in pyrotechnic compns is mentioned in Ref 4. Hale & Hart(Ref 2) patented delay powders contg insol chromates, such as Ag_2CrO_4, as oxidizers and Zn(or Zr) & S(or red P) as fuels

Refs: 1)Mellor **11**(1931), 263 2)G.C. Hale & D. Hart, USP 2468061(1949) & CA **43**, 589-90 (1949) 3)Kirk & Othmer **3**(1948), 948 4)Kirk & Othmer **11**(1953), 323 5)Sax(1957), 1104 6)CondChemDict(1961), 1023 7)Lange(1961), 302-3

Sodium Chromate, $Na_2CrO_4.10H_2O$, mw 342.16; yel translucent crysts, d 1.483, which form at 19.5-19.9° the *hexahydrate*, yel tubular triclinic crysts; at 25.9° the *tetrahydrate*, yel monocl crysts; and at 62.8° the *anhydrous* salt, mw 161.99, yel rhb crysts, d 2.72, which at 413° converts to hexagonal form. This latter form melts at 792° (Refs 1,2,3,4,5,7 & 9). All forms of Na chromate are very sol in w and sl sol in alc. Solubilities in w at various temps are given in Refs 3 & 4; and toxicity & fire hazard in Refs 6 & 7. The decahydrate can be prepd in the lab by treating Ca chromate with Na carbonate in w and crystallizing at a temp below 19.5°; industrially it can be prepd by roasting a finely ground chrome-iron ore mixed with soda ash and quicklime in an oxidizing atm using a reverberatory or rotary furnace. The roasted mass is treated with w to dissolve the resulting chromate, leaving ferric oxide as residue. Then the soln is concentrated and the decahydrate is crystallized at a temp below 19.5°. Anhydrous salt may be prepd by heating the hydrate to above 62.8°(Refs 1,2,4,7 & 8)

Industrial uses of Na chromates are discussed in Refs 4 & 7. The anhydrous salt can be used as an oxidizing agent in some commercial expls, but the dichromate is preferred,being less expensive per unit CrO_3(Ref 4). The decahydrate may be found useful(due to the presence of large amt of w of crystn) as a cooling agent in coal-mining expls

Refs: 1)Mellor **11**(1931), 244-8 2)Thorpe **3** (1939), 111 3)W.H. Hartford, IEC **41**, 1993-7 (1949) 4)Kirk & Othmer **3**(1949), 948-9 5)Ullmann **5**(1953), 586 6)Sax(1957), 1116 7)CondChemDict(1961), 1038 8)Mellor(1961), 870 & 872 9)Lange(1961), 306-7 10)Gmelin Syst Nr **52**(1962ff)

Sodium Chromate, Analytical Procedures. Chromate ion may be detected and detd using the methods listed under Chromates, Analytical Procedures. Sodium ion may be detected and detd using the methods described in Refs 1,2 & 3 US Federal requirements for Na chromate, anhydrous, technical grade and tests are described in Ref 4

Refs: 1)Scott & Furman(1939), 875-90 2)Vogel InorgAnalysis(1961), 557-60, 722 - 3 & 885 3)StdMethodsChemAnalysis **1**(1962), 11-19 4)US Federal Specification **O-S-558A**(Technical grade Na chromate)

Strontium Chromate, $SrCrO_4$, mw 203.64; yel monocl crysts, mp (?); d 3.895; v sl sol in w; sol in acids and Amm salts; can be prepd by treating a concd aq soln of Sr chloride with soln of K chromate, or by other methods(Ref 1). Its toxicity & fire hazard are in Ref 9 and industrial uses in Ref 11. Its uses in pyrotechnic items are discussed in Refs 2,3,4,5,6, 7,8 & 10

Refs: 1)Mellor **11**(1931), 270-1 2)G.J. Schladt, PATR **100**(1931)(Red pyro compns contg Sr Chromate) 3)G.J. Schladt, PATR **128**(1931) (Thermite-type pyro compns, contg Sr chromate, used as illuminants) 4)G.J. Schladt, PATR **470**(1934)(Red tracer compn contg Sr chromate) 5)G.J. Schladt, PATR **596**(1935)(Pyro compn for amber-colored signal, contg Sr chromate) 6)G.C. Hale, USP 245 0892(1949) & CA **43**,

408(1949)(Delay powders for fuzes. Eg: Sr Chromate 80, Mn 16 & S 4%) 7)G.C. Hale & D. Hart, USP 2468061(1961) & CA 43 589-90 (1949) [Gasless delay powders contg insol chromates(such as of Sr, Ba, Ca or Ag) as oxidizers and red phosphorus as fuel] 8)Kirk & Othmer **11**(1953), 323 9)Sax(1957), 1138 10)Ellern, Pyrotechnics(1961), 146 [Delay compns contg Ba or Sr chromate as oxidizers and Zr (or Mn) & P(or S) as fuels] 11)Cond ChemDict(1961), 1080 12)Lange(1961), 315-16 13)Gmelin, Syst Nr **52**(1962ff)

Strontium Chromate, Analytical Procedures. Chromate group may be detected and detd using the methods listed under Chromates, Analytical Procedures. Strontium ion may be detected and detd using the methods described in Refs 1,2 & 3. There is no US Military Specification for Sr Chromate

Refs: 1)Scott & Furman(1939), 899-902 2)Vogel, InorgAnalysis(1961), 552-3 3)StdMethodsChem Analysis **1**(1962), 993-1002

Zinc Chromate(*Zinc Chrome*), $ZnCrO_4$, mw 181.39; lt yel prisms; mp (?); d 5.3; v sl sol in w; sol in acids & bases. Its *heptahydrate*. $ZnSO_4.7H_2O$, lt yel crysts, can be prepd by the action of chromic acid on slurries of Zn oxide or hydroxide, or by other methods(Ref 1, 2,3,5 & 6). Toxicity and fire hazard are given in Ref 5a

Zn chromate is not important commercially, but its complex with K chromate, known as *Zinc Yellow*, is used as a corrosion inhibiting pigment(Ref 3) especially in aircraft construction (Refs 4 & 6)

Refs: 1)Mellor **11**(1931), 277-8 2)J.R.Callahan, ChemMetEngrg **50**, 111(June 1943)(Commercial manuf of Zn chromate) 3)Kirk & Othmer **3** (1949), 951 4)Thorpe **9** (1949), 635 5)Ullmann **5**(1953), 589 5a)Sax(1957), 1268 6)CondChem-Dict(1961), 1244 & 1250 7)Lange(1961), 328 -9

Zinc Chromate, Analytical Procedures. Chromate group and zinc ion can be detected and detd by the methods described in Refs 1,3 & 4. Analysis of commercial product used as a pigment is given in Ref 2

Refs: 1)Scott & Furman(1939), 282-304(Chromium) and 1054-92(Zinc) 2)Snell & Biffen(1944), 532-3 3)Vogel, Inorg Analysis(1961), 520-3 (Chromates) and 390, 401-2 433-4, 532-6, 615, 844 & 1045 4)Std MethodsChemAnalysis **1**(1962), 1228-65

DICHROMATES(or Bichromates) are salts of *dichromic acid*, $H_2Cr_2O_7$, which has never been isolated but is known in solns. Dichromates decomp on heating(and sometimes without heating, just on contact with combustible materials) giving off 3 oxygens per mole: $M_2Cr_2O_7 = Cr_2O_3 + M_2O + 3O$, where M is a monovalent metal. They are even stronger oxidizing agents than corresponding chromates. Some dichromates of organic compds are explosive and are listed separately under corresponding parent org compd

Methods of prepn, props and uses of inorg dichromates are discussed under individual compds

Refs: Same as under CHROMATES

List of Dichromates

Ammonium Dichromate or Bichromate, $(NH_4)_2Cr_2O_7$, mw 252.10, N 11.11%; red-orn monocl crysts; mp - starts to decomp ca 188° without melting, and the reaction: $(NH_4)_2Cr_2O_7 \rightarrow Cr_2O_3 + N_2 + 4H_2O$ becomes self-sustaining ca 225°(Ref 7)(See also Refs 2, 2a & 14); heated in flame it ignites and burns producing a flame which continues to burn after the flame is removed(Refs 2a & 5) d 2.160 at 25°/25°(Refs 2); 2.15(Ref 2a) & 2.155 at 25°/4°(Ref 7). Q_e 310kcal/kg(Ref 2a); Q_f 423kcal/mole(Ref 2a) and 430 ± 6kcal/mole (Ref 9a); max temp of expln 1180°; vol of gas on expln 444ml/g; specific energy(f) 2440kg/l; deton velocity - does not detonate; and impact sensitivity - some explns with 20kg wt falling 10-16cm; when heated while confined in an iron container with walls 1mm thick, it explodes, breaking the container and scattering the salt (Ref 2a); very sol in w(Refs 2,7 & 9); sol in alc, but insol in acet(Ref 14); may react explosively with some org compds(Ref 12). Amm chromate can be prepd by action of chromic acid on Amm hydroxide with subsequent crystn; or by other methods, such as heating an ammoniacal soln of K dichromate until the liq assumes a garnet-ed color, followed by evaporation and crystallization(Refs 3,4,6,9,11 & 13). Combustion of Amm dichromate is discussed in Ref 8a and toxicity & fire hazard in Refs 10 & 13

Uses: Being a strong oxidizing agent Amm dichromate found uses in some composite

expls, proplnts & pyrotechnic compns(Ref 3, 6 & 12). Daniel(Ref 1,p 418) stated that Majert of Berlin patented an expl contg Amm chromate, and that Cadoret patented an expl called Tribenite(Ref 1,p 773). Colver(Ref 1a) stated that an expl contg Amm dichromate and Amm or K picrate was patented in England in 1894. Taylor & Rinkenbach(Ref 1b) stated that Amm dichromate was little used in the manuf of expls. Daniel(Ref 1,pp 68 & 385) stated that Amm dichromate was used in sporting proplnt *Poudre J*, which contained NC 83 & dichromate 17%. Davis(Ref 5) and Izzo(Ref 8) lists a pyrotechnic mixt contg Amm dichromate 50, K nitrate 25 & dextrin 25%. Ellern(Ref 12,p 120) states that an extensive patent literature exists which refers to solid, gas-generating compns such as GuN & Amm dichromate. In the same Ref (p 279), the following compns are given as "high nitrogen type gas producers". a)Formula 51: AN 78.5, K nitrate 9.0, Amm oxalate(anhyd) 6.9 & Amm dichromate 5.6%, with 0.7 to 2.5 parts of china clay added b)Formula 52: NGu 56, GuN 28, Amm dichromate 8, dimethyldiphenylurea 4 & beech charcoal 4%

Refs: 1)Daniel(1902), 68-9, 418 & 773 1a)Colver (1919), 324 1b)C.A. Taylor & W.H. Rickenbach, US BurMinesBull **219**(1923), 49 2)E. Moles & F. Gonzalez, AnalesSocEspanFisQuim **21**, 204(1923) & CA **17**, 2999(1923) 2a)H. Kast, SS **22**, 7-8(1927) 3)Mellor **11**(1931), 323-5 4)Thorpe **3**(1939), 111 5)Davis(1943), 120 6)Kirk & Othmer **3**(1949), 951 7)W.H. Hartford, IEC **41**, 1993-7(1949) 8)Izzo, Pirotecnia(1950), 230 8a)A.A.Shidlovskii et al, ZhPriklKhmi **26**, 23-9 (1953) & CA **47**, 5226(1953) 9)Ullmann **5** (1953), 585-6 9a)A.F.Kapustinskii & A.A. Shidlovskii, CA **50**, 9849(1956) 10)Sax(1957), 274 11)Mellor(1961), 872 12)Ellern, Pyrotechnics(1961), 120 & 279 13)CondChemDict(1961), 72 14)Lange(1961), 218-19

Ammonium Dichromate, Analytical Procedures. Accdg to Taylor and Rickenbach(See Ref 1b), above, the methods of its analysis are identical with those of K dichromate, except that the total ammonia should be detd by the Kjeldahl method. See also under Ammonium Chromate and US Federal Specification O-A-498a

Barium Dichromate, $BaCr_2O_7$, mw 353.38; red, monocl crysts; mp (?); sol in hot concd H_2SO_4; decompd by w. Can be prepd by action of a soln of chromic acid on freshly pptd Ba chromate or by other methods(Ref 1). It is a powerful oxidizer. Its toxicity and fire hazard are given in Ref 4. Its *dihydrate,* $BaCr_2O_7.2H_2O$, yel or brn-red crysts loses $2H_2O$ at 120°. Uses not specified in Refs listed below

Refs: 1)Mellor **11**, 341 2)Kirk & Othmer **3** (1949) - not found 3)Ullmann **5**(1953) - not found 4)Sax(1957), 332 5)CondChemDict(1961), 126 6)Lange(1961), 224-5

Calcium Dichromate, $CaCr_2O_7.3H_2O$; brilliant red-orn crysts, existing above 42.1°, while below that temp the $CaCr_2O_7.4.5H_2O$ can by crystallized; both hydrates lose w of crystn at 100-105° and hydrolyze with formation of Ca chromate & Cr trioxide; heating to ca 400° causes slow evoln of oxygen, which at 500° becomes very rapid, until finally blk-violet crysts form at 1100-1200°(Ref 2,p 3355). Hydrates can be prepd by slowly treating a soln of Cr trioxide with pptd $CaCO_3$, or finely divided Ca oxide, or hydroxide as described in Ref 2 (See also Ref 1). Some props are given in Refs 3 & 4. No evidence was obtained of the existence of anhydrous $CaCr_2O_7$ as a definite crystalline phase(Ref 2,p 3355). As Ca dichromates are similar in their props to Ca chromate, they probably can be used for the same purposes

Refs: 1)Mellor **11**(1931), 340-1 1a)Kirk & Othmer **3**(1949) not found 2)W.H. Hartford et al, JACS **72**, 3353-6(1950) 3)Ullmann **5** (1953), 586 3a)Sax(1957) not found 4)Cond ChemDict(1961), 201

Copper(II) bis(ethylenediamine)Dichromate. See under COPPER AND ITS SALTS

Lead Dichromate, $PbCr_2O_7$, mw 423.23; brick red or brn-red, amorph or cryst pdr; mp(?); decomp by w; sol in alc or alkalies(Ref 5); can be prepd by treating Pb chromate with a concd soln of chromic acid or by other methods (Ref 1). It is a powerful oxidizer and reacts vigorously when heated with reducing materials; when heated to decompn, it emits highly toxic fumes of lead(Ref 4); can probably be used for the same purposes as $PbCrO_4$, namely in pyrotechnics

Refs: 1)Mellor **11**(1931), 342-3 2)Kirk & Othmer **3**(1949) - not found 3)Ullmann **5**(1953) not found 4)Sax(1957), 819-20 5)Lange(1961), 262-3 6)CondChemDict(1961) - not found

Potassium Dichromate(Potassium Bichromate or Red Potassium Chromate), $K_2Cr_2O_7$, mw 294.22; red tricl crysts; mp-when heated to ca

236°, the tricl or alpha form changes to monocl or beta form which melts at ca 398°, and decomposes at ca 500°(Refs 6,11 & 16); mp 396° (Ref 22); d 2.676 at 25°(Ref 23); 2.70(Ref 11); 2.723(Ref 12); 2.684(Ref 16) and 2.692(Ref 22); Q_f 488.5kcal/mol(Ref 16); it is nonhygroscopic and does not form any hydrates; insol in alc; its solubilities in w at different temps(% $K_2Cr_2O_7$) are: 4.3 at 0°, 11.7 at 20°, 20.9 at 40°, 31.3 at 60°, 42.0 at 80° and 50.2 at 100° (Refs 12 & 16). Can be prepd by adding concd sulfuric acid soln to K chromate or by other methods(Refs 8,8a,11,16 & 22). A detailed description of a lab method of prepn, by heating an aq soln of Na dichromate with K chloride, is given in Ref 16; and an industrial method of treating the cold satd soln coming from the granulators in the crystn of Na dichromate with the theoretical amt of KCl is described in Ref 11. Toxicity and fire hazard of K dichromate are discussed in Ref 19

It is a strong oxidizing agent

Following are US Federal Specification (Ref 24) requirements for technical grade material intended for general use: a)It shall be in cryst form and shall not be caked in container b)Purity as $K_2Cr_2O_7$ - not less than 99.0% c)Volatile Matter at 120 ± 5° - mat 0.2% d)Water - Insoluble Matter - max 0.1% e)Particle Size - 100% thru No 10 sieve & max 25% thru No 100 f)Sulfates(as SO_4) - max 0.1% and g) Chlorides(as Cl) - max 0.1%

Uses: K dichromate has been used in some explosive, proplnt and pyrotechnic compns. Its industrial uses(See Refs 4,11,16 & 22) are essentially the same as for Na dichromate, but as the latter is cheaper, it is used to a much greater extent than the K salt. In some cases, however, when an anhyd and nondeliquescent oxidizer is required(such as in safety match heads), the K salt is preferred(Ref 11,pp 951-2)

Following are examples of K dichromate's uses in expl, proplnt and pyrotechnic compns: *A)Explosive Compositions:* a)Ammonals(Ref 3,p 253) b)Beneké Explosive(Vol 2 of Encyclopedia,p B33-R) c)Borlinetto Powder(Vol 2,p B250-R; Ref 9,p 220 & Ref 20a,p 388) d)Boyd Powder(Vol 2,p B 259-L) e)Carbonites (Vol 2,p C 61-R); Ref 1a,pp 703, 714 & 716; Ref 7,p 401; Ref 9,p 352 & Ref 20,p Ger 26) f)Casthelaz & Désignolle(Vol 2,p C 84-L & Ref 9a,p 220) g)Dahmenites(Ref 1,p 790,under Von Dahmen; Ref 2,p 493; Ref 4,p 139; Ref 20,p Ger 33 & Ref 20a,p 375) h)Davey Powder (Ref 1,pp 79-80) i)Fractorite(Ref 1,pp 703, 705 & 712) j)Halsey & Savage of California patented in 1896-1899 several Amm picrate expl compns contg 20-25% K dichromate (Ref 1,p 367 & Ref 20a,p 389) k)Italian mining explosive listed by Belgrano(Ref 15,p 284): AN 84.5, DNN 10, K dichromate 4.5 & charcoal 1%; its props are: Power by Trauzl Test 400cc, Detonation Velocity 1800m/sec and Gap Test Value(Distanza colpo in Ital) 3cm l)Petroclastite(Ref 1,p 612 & Ref 20a,p 387) m)Pullwitz Safety Explosive(1895)(Ref 1,p 659 & Ref 20a,p 375) n)Pyroroxylite(1887)(Ref 1,p 665) o)VonBrank's Explosive contg K dichromate. See under Brank's Explosives in Vol 2,p B of this Encyclopedia p)Von Dahmen's Explosives contg K dichromate. See Dahmenite A and Ref 1,p 792) q)Ward & Gregory Powder(Ref 20a,p 400)

B)Propellants: a)A variety of Fr sporting proplnt, *Poudre J*, (listed under Ammonium Dichromate) contd 3% of K dichromate(Ref 4,pp 86-7) b) Fr sporting proplnt *Poudre Okell*(Ref 1,p 590) c)Older Amer proplnt patented in 1894 by the US Smokeless Powder Co: Amm picrate 55, K or Na picrate 25 & K dichromate 20%(Ref 1,p 780) d)Some bulk powders listed in Ref 9,p 289

C)Pyrotechnic Compositions: a)K dichromate 12, K chlorate 15, Zn dust 72, granulated charcoal 12 & dextrin 2 parts(Ref 9,p 88) b)Italian illuminating mixt: K dichromate 10, K chlorate 14, Zn dust 64, charcoal 10 & gum arabic or dextrin 2%(Ref 13) c)Safety matchhead compns(Ref 4,p 163; Ref 11,pp 951-2 and Ref 21,p 232) d)Thermite-type gasless pdrs contg Al & K dichromate(Ref 21,p 266)

Some other uses of K dichromate are described in Refs 10, 17 & 18

Refs: 1)Daniel(1902), 69, 79, 367 & 790-2 (K dichromate used in safety expls, acts not only as an oxidizer but also as a phlegmatizer contributing towards safety in regard to firedamp in gaseous coal mines) 1a)Gody(1907), 703, 705, 712, 714 & 716 2)Marshall 2(1917), 493 (Use in some expls) 3)Colver(1918), 253(Addn of K dichromate to an Ammonal increases its power w/o decreasing its safety) 4)Barnett (1919), 86-7, 139 & 163(Uses in proplnts, expls and pyrotechnic compns) 4a)Marshall, Dict (1920), 27-8 & 52 5)C.A. Taylor & W.H.

Rinkenbach, "Explosives", US BurMinesBull **219**(1923), 46-7(Prop of K dichromate; uses in certain classes of safety expls) 6)P.L. Robinson et al, JCS **127**, 547-9(1925)(Detn of melting and transition points) 7)Naoúm NG (1928), 401(Use in Carbonites) 8)Mellor **11** (1931), 328-38(Prepn & some props) 8a)Thorpe **3**(1939), 110-11 9)Davis(1943), 88, 289 & 353 (Uses in pyrotechnics, expls & proplnts) 9a)Pérez Ara(1945), 220 10)H. Aaronson, PATR **1562**(1945)(Prepn of TNB by oxidation of TNT with K or Na dichromate) 1)Kirk & Othmer **3**(1949), 951-2(Prepn, props & uses) 12)W.H. Hartford, IEC **41**, 1993-7(1949)(Some props, including soluby w at diff temps) 13)Izzo, Pirotecnia (1950), 217(An Ital pyro compn) 14) H. Flood & A. Muan, ActaChem Scand **4**, 365-9(1950) & CA **44**, 9781(1950) (Thermochemical props of dichromates) 15)Belgrano(1952), 284(A mining expl contg K dichromate) 16)Ullmann **5**(1953)(Prepn, props & uses) 17)D. Hart, USP 2696429(1954) & CA **49**, 5845(1955)(Use of dichromated Zr-Ni or Ti-Ni alloy pdrs in conjunction with Ba chlorate & K perchlorate in gasless delay compns) 18)J.E. Rainier & J.M. Swotinsky, PATR **1992** (1954)(Development of a rocket proplnt)(Conf) (Not used as a source of info) 19)Sax(1957), 1040(Some props) 20)PATR(1958),pp Ger 26 & 33 20a)Giua, Trattato **6**(1959), 375, 387-9 & 400 21)Ellern, Pyrotechnics(1961), 188, 232 & 266(Uses in pyro compns) 22)CondChemDict (1961), 929(Prepn, props & uses) 23)Lange (1961), 290-1(Some props) 24)US Federal Specification **O-P-559**(K dichromate, technical grade)

Potassium Dichromate, Analytical Procedures. Dichromate ion may be detected and detd by methods similar to those listed under Chromates. Potassium ion may be detected and detd as described in Refs 2,3 & 5

Method of analysis of commercial product suitable for use in mine expls, as practiced at US Bureau of Mines, Bruceton and Pittsburgh, Pa, is described in Ref 1. The method is not given here, because it does not seem to have any advantage over the method prescribed in US Federal Specification O-P-559(Ref 6)

This method consists of the following tests:

A)Purity as $K_2Cr_2O_7$. Weigh accurately a 4g sample, dissolve in distd w, transfer quantitatively to a 1000ml vol flask and dil to the mark. Transfer a 50.0ml aliquot to a 250ml glass stoppered Erlen flask, add 4-5g CP KI crysts, shake to dissolve, and add 10ml dil sulfuric acid(1:3). After allowing to stand for 10 mins, titrate the liberated iodine with 0.1N Na thiosulfate soln, until the yel color nearly disappears, Then add 5ml starch indicator and continue titration until the blue color just disappears. Correct for any thiosulfate consumed by the blank. One ml of 0.1N Na thiosulfate soln is equivalent to 0.00490 g $K_2Cr_2O_7$

B)Volatile Matter. Weigh accurately a 5g sample(quickly crushed to pass No 20 sieve just before weighing) into a shallow dish 6-8" in diam and 1-3" in depth. Heat in an oven at $120\pm5^{\circ}$ to const wt and calc the loss in wt as volatile matter

C)Water-Insoluble Matter. Dissolve a 10g, accurately weighed, sample in 100ml warm distd w and heat on a steam bath for 1hr. Filter thru a tared sintered glass crucible, wash the residue with warm distd w until the disappearance of yel color(ca 100ml in small portions) and dry in an oven to const wt

D)Particle Size. Shake(by hand, or mechanically) a 100g sample spread on a tared No 10 US Std sieve, which is placed on top of a tared No 100 sieve and determine the amt of material retained on each sieve. The method is described in detail in US Federal Specification **P-S-536**

E)Sulfates. Dissolve a 10g, accurately weighed sample, in 600ml w, contained in a 1000ml beaker. Add 75ml ethanol and 60ml concd HCl. When the reaction has ceased, bring to a boil and concentrate to a thick syrup. Dilute with w to ca 400ml, heat to boiling and, while boiling slowly, add 50ml hot 10% Ba chloride soln. Continue boiling for 5 mins, let stand overnight at 40-50° and filter thru a tared Gooch crucible. After washing the ppt with 300ml boiling w, dry the crucible at 150° for 30 mins, ignite it over a Meker burner for 3 mins, cool in a desiccator and weigh. Multiply the wt of $BaSO_4$ by 0.4115 to obtain the weight of SO_4

F)Chlorides. Dissolve a 10g, accurately weighed, sample in 50ml w contained in a 250ml beaker. Add a satd CP Na carbonate soln until the color changes to clear yel and titrate with 0.1N std Ag nitrate soln until the color changes to muddy brn. Multiply the mls of 0.1N Ag nitrate by 0.00355 to obtain the wt of Cl

Refs: 1)C.A. Taylor & W.H. Rinkenbach, "Explosives", USBurMine sBull **219**(1923), 47-8 2)Scott & Furman(1939), 861-74 3)Treadwell & Hall, Vols 1 & 2(1942) 4)Snell & Biffen(1944), 728(Prepn of std K dichromate soln) 5)Vogel, Inorg Analysis(1961), 304-13(Uses of K dichromate in volumetric analysis) 6)US Federal Specification O-P-559(K dichromate, technical grade) 7)StdMethodsChemAnalysis 1(1962), 20-26 & 350-76

Sodium Dichromate(Sodium Bichromate), $Na_2Cr_2O_7.2H_2O$; mw 298.03; red or red-orn deliq monocl crysts; mp loses w on prolonged heating at 105° forming anhydrous salt which melts at 320°and decomp at 400°; d of dihydrate 2.52(Ref 10); Kitk & Othmer(Ref 6) gives 84.6° as transition point from dihydrate to anhyd salt; the tech anhyd salt consists of red-orn, fine granular crysts, melting at 356° and starting to decomp ca 400°; Ullmann(Ref 8) gives 450° as decompn temp and d 2.52 for dihydrate; Lange(Ref 12), gives d 2.348 at 25° for dihydrate and loss of $2H_2O$ at 84.6°. Na dichromate is insol in alc and v sol in w: sol, % $Na_2Cr_2O_7$ by wt: 70.6 at 0° 73.18 at 20°, 77.09 at 40°, 82.04 at 60°, 88.39 at 80° & 91.43 at 100°(Refs 7 & 8). It can be prepd by one of the following methods: a)Action of H_2SO_4 on Na chromate or b)By roasting a mixt of finely ground chrome-iron ore with soda-ash and quicklime, in an oxidizing atm, in a reverberatory or rotary furnace. The roasted mass is then mixed with w to dissolve Na chromate thus separating it from insol ferric oxide. The crystallized chromate is treated with H_2SO_4 and dichromate is obtained(See Refs 1,6,7,8,10 & 11). Toxicity & fire hazard are discussed in Ref 9

US Federal Specification requirements(Ref 14) for technical grade Na dichromate intended for general use are as follows: a)It shall be $Na_2Cr_2O_7.2H_2O$, in granular form b)Purity–min 99.0% $Na_2Cr_2O_7$ of the ignited sample c)Volatile Matter at 120±5° - max 12.5% d)Water - Insoluble Matter - max 0.2% e)Sulfates (as SO_4)-max 0.1% f)Chlorides(as Cl) - max 0.2% f)Particle Size - not given, but in the old Spec O-S-595(1944) it was 100% thru No 8 US Std sieve and not over 5% thru No 100 sieve

US Military Specification requirements(Ref 15) for reagent grade Na dichromate are as follows: a)Material shall consist of red crysts b)Purity-min 99.0%,as $Na_2Cr_2O_7.2H_2O$ c)Chlorides (as Cl) - 0.005% d)Sulfates(as SO_4) - 0.03% e)Calcium(as Ca) 0.010%

Uses: Na dichromate, being an oxidizing agent less expensive than K dichromate, has been used very extensively in laboratories and for manufg purposes. It cannot be used, however, where its hygroscopicity is objectionable, as for example in some explosive, proplnt or pyrotechnic compns. Large quantities are used in labs for cleaning glassware. For this a "cleaning solution" (also called Chromic Mixture or Chromosulfuric Solution) is prepd by adding an excess of Na dichromate to concd sulfuric acid. Davis(Ref 3) and Aaronson (Ref 5) described the use of Na dichromate as the oxidizing agent in prepn of TNB from TNT. Hart(Ref 4) used concd aq soln of Na dichromate as a coating agent for powdered Mg or Mg-Al alloys employed in some pyrotechnic compns. He claimed that this treatment improved the resistance to moisture and eliminated the hazard which attended the previous practice of coating powdered metals with linseed oil. Industrial uses of Na dichromate are listed in Refs 6,8 & 10; and two expls *Nahsen* and *Wegel* contg Na dichromate are listed by Giua(Ref 9a)

Refs: 1)Mellor **11**(1931), 129-33(Extraction of Cr as chromate from chrom-iron ore); 325-8 (Other methods of prepn and props) 2)Thorpe **3**(1939), 111(Some props) 3)Davis(1943), 135 4)D.Hart, PATR **1403**(1944) 5)H. Aaronson, PATR **1562**(1945) 6)Kirk & Othmer **3**(1949), 952-3 7)W.H. Hartford, IEC **41**, 1993-7(1949) (Some props) 8)Ullmann **5**(1953), 575(Industrial prepn); 588(Props & uses) 9)Sax(1957), 1116 9a)Guia, Trattato **6**(1959), 385 & 388 10)Cond ChemDict(1961), 1040 11)Mellor(1961), 870 12)Lange(1961), 306-7 13)Ellern, Pyrotechnics (1961) - not listed 14)US Federal Specification O-S-595a 15)US Military Specification MIL-S-11161

Sodium Dichromate, Analytical Procedures. Dichromate ion and Na ion may be detected and detd by the method listed under Sodium Chromate, Analytical Procedures

Following are tests prescribed by US Federal Specification O-S-595a for technical grade Na dichromate:

A)Purity as $Na_2Cr_2O_7.2H_2O$. Use the same procedure as described in opn A under Potassium Dichromate, Analytical Procedures. One ml of

Na thiosulfate soln is equivalent to 0.00437g $Na_2Cr_2O_7$

B)Volatile Matter at 120° Same procedure as in opn B under Potassium Dichromate

C)Water-Insoluble Matter. Same procedure as in opn C under Potassium Dichromate

D)Particle Size - not found in new Specification

E)Sulfates. Same procedure as in opn E under Potassium Dichromate

F)Chlorides. Same procedure as in opn F under Potassium chromate

Following are tests prescribed by US Military Specification MIL-S-11161 for reagent grade Na dichromate:

a)Purity as $Na_2Cr_2O_7.2H_2O$. Weigh accurately a 0.2g sample(previously dried at 105 ± 5°) and dissolve it in 200ml of freshly-boiled and cooled distd w in a glass-stoppered flask. Add 3g of KI & 7ml of 37% HCl, allow to stand for 10 mins in the dark and titrate the liberated iodine with 0.1N Na thiosulfate soln, adding starch toward the end of titration

%Na dichromate = (AxBx100)/C,

where A=ml of 0.1N thiosulfate soln; B=0.004967 and C=wt of original sample

b)Chloride. Dissolve 1.000g sample in 20ml of w, add 10ml of 30% nitric acid, heat to ca 50°, add 5 drops of 2% aq Ag nitrate soln and allow to stand for 10min in the dark, and if any ppt forms, the material does not comply with Spec requirement

c)Sulfate. Dissolve 1.000g in 20ml of w, add 5ml of 37% HCl and 5ml of 12% aq Ba chloride soln. Allow to stand for 15mins and if any ppt forms, the material does not comply with Spec requirement

d)Calcium. Dissolve 2.000g in 30ml of w, add 5ml of 28% Amm hydroxide & 5ml of 4% aq Amm oxalate soln. Allow to stand for 5 mins and if any turbidity develops the sample does not comply with Spec requirement

Refs: See under Sodium Chromate

TRICHROMATES are derivs of hypothetical *trichromic acid,* $H_2Cr_3O_{10}$. They are strong oxidizing agents and for this reason are briefly described here

Ref: Mellor **11**, 349-51

List of Trichromates

Ammonium Trichromate, $(NH_4)_2Cr_3O_{10}$, mw 352.11; dk garnet-red, rhmb bipyramids; mp-darkens at 110-20°, decomp at 150-60° and explodes weakly at 190°; d 2.329 at 10°. Can be prepd by evapg a soln of Amm dichromate in concd soln of chromic acid(Refs 1 & 3). It can also be prepd by crystn of Amm dichromate from nitric acid d 1.39(Ref 2). Combustion of Amm trichromate is discussed in Ref 4. Its heat of formation is, accdg to Kast, 678.1kcal /mole(Ref 2) and 580 ± 6kcal/mole, accdg to Kapustinskii(Ref 5). Its uses are not described in the literature

Refs: 1)E.Jäger & G. Krüss, Ber **22**, 2034 (1889) 2)H.Kast SS **22**, 7(1927) 3)Mellor **11**(1931), 349-50 4)A.A. Shidlovskii & S.A. Oranzhereev, ZhPriklKhim **26**, 23-9(1953) & CA **47**, 5226(1953) 5)A.F.Kapustinskii & A.A. Shidlovskii, IzvestSektoraPlatiny i DrugBlagorMetalInstObshch i NeorgKhimAkadN **30**, 31-8 (1955) & CA **50**, 9849(1956)

Potassium Trichromate, $K_2Cr_3O_{10}$, mw 394.23, red prisms; mp - blackens and melts at 250° with decompn; d 2.667 at 10°. It was reported that the salt decrepitates a little when heated. Can be prepd by evapg, in cold over concd sulfuric acid, a soln of K dichromate in nitric acid, d 1.19. If nitric acid is more concd, some tetrachromate is formed(Refs 1 & 2). As it is a strong oxidizing agent, the possibility of its uses in expl compns is not excluded

Refs: 1)E. Jäger & G. Krüss, Ber **22**, 2039 (1889) 2)Mellor **11**(1931), 350

Sodium Trichromate, $Na_2Cr_3O_{10}.H_2O$, mw 380.02; dk-red deliquescent crysts; mp - not given; sol in w without decompn. Can be prepd by evapg, in cold over concd sulfuric acid, a soln of Na dichromate in chromic acid(Refs 1 & 2). Its uses are unknown to us

Refs: 1)A. Stanley, ChemNews **54**, 196(1886) 2)Mellor **11**(1931), 350

TETRACHROMATES are derivs of hypothetical *tetrachromic acid,* $H_2Cr_4O_{13}$. They are strong oxidizing agents and for this reason are briefly described here

Ref: Mellor **11**(1931), 351-53

List of Tetrachromates

Ammonium Tetrachromate, $(NH_4)_2Cr_4O_{13}$, mw 452.12; brn-red hygroscopic crysts, less stable than Amm trichromate; mp - blackens at 160°, melts at 170° and decomp spontaneously giving off nitrous fumes. Can be prepd by evapg, in cold over concd sulfuric acid, a soln of Amm trichromate in nitric acid, d 1.39(Refs 1 & 2).

Kapustinskii et al give 730± 10kcal-mole as heat of formation(Ref 3). Its uses are unkown to us

Refs: 1)E. Jäger & G. Krüss, Ber **22**, 2036 (1889) 2)Mellor **11**(1931), 352 3)A.F. Kapustinskii & A.A. Shidlovskii, CA **50**, 9849 (1956)

Potassium Tetrachromate, $K_2Cr_4O_{13}$, mw 494.24; brn-red or carmine-red deliquescent plates or monocl prisms; mp 215°, d 2.649 at 11°; decomp by w. Can be prepd by evapg slowly on a sand bath a soln of K trichromate in nitric acid d 1.4 to 1.5 or by other methods (Refs 1 & 2). Its uses are unknown to us

Refs: 1)E.Jäger & G. Krüss, Ber **22**, 2036 (1889) 2)Mellor **11**(1931), 352

Sodium Tetrachromate, $Na_2Cr_4O_{13}.H_2O$, mw 498.03; garnet-red deliquescent plts; mp 40-50° with decompn; sol in ww/o decompn. Can be prepd by evapg a soln of Na chromate in an excess of chromic acid(Refs 1 & 2). Its uses are unknown to us

Refs: 1)F. Mylius & R. Funk, Ber **33**, 3686 (1900) 2)Mellor **11**(1931), 352

Chromated Nitrocellulose(Coton-poudre chromé in Fr). Davey of England patented in 1877, the treatment of NC with a soln of chromate or dichromate together with or w/o K nitrate, glue or a hydrocarbon. This material was non-hygroscopic and suitable for use as a core in mining fuses, instead of BkPdr

Refs: 1)Cundill(1889) in MP **5**, 309 & 314 (1892) 2)Daniel(1902), 143 & 180

Chromathermography. See Item I, under CHROMATOGRAPHY.

CHROMATOGRAPHY

Chromatography may be defined as a physical method of separation in which the components to be separated are partitioned between two phases. One of these is stationary(solid or liquid), the other being mobile(liquid or gas), percolates thru stationary phase(Refs 58, 77 & 78)

Various branches of chromatography such as solid-liquid-, liquid-liquid-, paper- and gas-chromatographies, will be described below under "Current Methods of Chromatography"

Historical. Chromatography is based on the ability of some solid substances to *adsorb* (See Adsorption in Vol 1,p A105-L) some gases or liquids and on the ability of some liquids to *absorb* (See Absorption in Vol 1,p A5-R) other liquids or gases. The ability of untreated paper to separate various substances was known for centuries(Ref 78,p 7). For example, Pliny the Elder(AD 23-73) used papyrus impregnated with an extract of gall nuts for the detection of ferrous sulfate(Ref 54,p 3). The principle of absorption of a gas by a liquid was first observed in 1512 by the Strassburg physician H. Brunswig. He applied his discovery to the purification of aq ethyl alcohol as described in Ref 77,p 4. Accdg to Hersch(Ref 82a ,p 1), adsorption by solids was first discovered by Scheele in 1773 and by the Abbé Fontana in 1777. The ability of charcoal to remove color from solns was investigated by Lowitz in 1785 and systematic investigation of adsorption was carried out in 1814 by de Saussure. F. Runge [Dissertation, Univ of Berlin(1882)] and C. Schönbein in 1861(Ref 1), proposed to use filter paper as a selective adsorbent in analytical chemistry(See also Ref 77,p 5). The phenomenon of adsorption of liquids by solid materials such as Fuller's earth was described ca 1898 by the Amer geologist D.T. Day (1855-1925). He succeeded, during 1898-1903, to separate(using a column of Fuller's earth) petroleum into a light fraction(such as gasoline and kerosene), light and heavy oils(such as lubricating oils), and vaseline(Refs 2 & 78, pp 3-4). A few years later(1908-1913), Day induced Gilpin et al(Refs 4 & 78,p 5) to conduct more detailed experiments while using larger columns and liquids other than petroleum. Experiments of Day et al, were forgotten until Zechmeister referred to their work in a paper published in 1948(Ref 24)

Nearly simultaneously with Day and quite independently from him, a Rus botanist M.S. Tsvet(also spelled Tswett) [1872-1919(or 1920?)], devised a method for separating a plant leaf extract(such as mixts of chlorophyls and carotenoids) by passing the liquid thru a column of inulin, followed by washing("developing") it with pure solvent. He applied this method to analysis of many other substances (mostly pigments) and coined the terms "chromatographic adsorption analysis" or "chromatography", because he worked mostly with colored substances. (Greek word

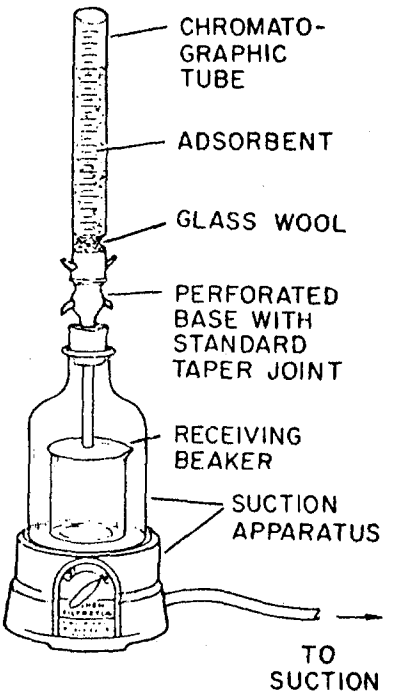
"chroma" means color and "graphein" means to write). As this method is used now also for analysis of colorless mixts, the term "chromatography" may be considered as a misnomer. Tsvet was the 1st to be aware of the great possibilities of this technique and he stated that "it still gives information where other analytical methods have failed"(Refs 3, 19, 58 & 78). Tsvet published in the period preceding WWI more than 50 papers on chromatography. As most of these papers were in Russian, the Tsvet technique was practically unknown in Europe or US until after his death. The 1st break was ca 1922' when Palmer conducted some chromatographic separations in the US(Ref 5). Nothing was done, however, in Europe until 1930's when papers of Kuhn & Lederer(Ref 6) and then of Karrer & Neilson (Ref 7) were published. From that time on, an avalanche of papers appeared throughout the world and reached by now probably more than 25,000

The original Tsvet's technique is still used, but some modifications and new techniques appeared since ca 1938. They are briefly described in pages which follow

Current Methods of Chromatography. Chromatography is presently understood to cover a group of related techniques for the separation, identification and quantitative detn of complex mixts of solids, liquids or gases. Included, in this group of techniques are: Solid-liquid adsorption chromatography; liquid-liquid partition chromatography; paper chromatography; gas chromatography(which includes gas-liquid and gas-solid chromatography); electrochromatography; ion-exchange chromatography; molecular sieves chromatography; thin-layer chromatography; chromathermography-and inverted chromatography

A. *Solid-liquid Adsorption Chromatography or Column Chromatography.* An apparatus for conducting this technique consists of the following parts(See Fig):

Vertical glass(seldom plastic or metal) tube 200-250mm long and ca 35mm in diam, provided at the bottom with a coarse porous porcelain disc or a loose wad of glass wool. This part of the tube is connected thru a std taper joint to a thistle-shaped funnel which is inserted(thru a hole in rubber stopper) into the neck of a bell jar suction filtrate assembly in the manner to have the tip of the funnel just over the receiving beaker



Chromatographic Adsorption Apparatus

Before starting the analysis, the tube is filled to about half with an appropriate adsorbent(such as alumina, Fuller's earth, Floridin, acid clay, mixt of Celite 545 with silica gel, mixt of Johns Manville's Hyper-Supercel with silicic acid, charcoal, etc). The resulting ensemble is known as a "chromatographic column". The columns may be packed either dry or wet. In the former case, the dry adsorbent is introduced into the tube in portions and each packed well with a tamping rod. This gives a more homogeneous column than adding adsorbent all at once. The column is then thoroughly rinsed with the solvent to be used for the chromatographic run until all the air is removed. For wet packing, a previously well mixed slurry of the adsorbent and of the solvent to be used in analysis is added gradually to the tube until the desired height is reached, taking care that some liquid always remains above the adsorption column. Then the column is rinsed with the solvent as in dry packing. To speed up formation of columns, both in dry and wet packing a slight suction is usually applied, carefully avoiding formation of cracks.

Wet packing is quicker and gives more homogeneous adsorbents. Various types of columns are described in Ref 58, pp 14-18 and in Ref 78,pp 56-8

Further operations consist of removal and separation of substances from the soln by means of the adsorption column

In many cases, it can be done by dissolving the sample in a nonpolar liquid(such as was used for washing the column) and introducing the soln in the column to form the so-called "starting zone". The vol of soln should be not larger than is required to occupy only a very small portion of the adsorbent. Then while using a slight vacuum, a series of progressively more polar solvents(such as formed by mixts of nonpolar with polar liquids) is gradually added to the top of the column until the weakly adsorbed substances would migrate rapidly to nearly the bottom of column, leaving the slower moving stronger adsorbed materials in the upper section of the column. As a result of this action, the adsorbed substances gradually separate from one another forming a series of bands, known as "zones"(some of which may be colored). The ensemble of zones is called "chromatogram" and the process of washing is known as "development of chromatogram". If it is desired to make colorless zones visible, the column may be extruded, without breaking it, from the tube and a narrow band is painted lengthwise using a small brush wetted with a reagent which would produce colored "streaks" with adsorbed substances(Ref 58,p 55). For example, in sepn of TNT from DNT and MNT, an alc KOH soln contg some acet, produces when streaked on a column, a red color with TNT zone, a bluish with DNT, and a yellowish with MNT(See also Ref 23)

In order to separate and identify the various substances in the column, each zone is cut off at the borders with a knife and treated with a "polar" solvent to dissolve the adsorbed compds. After sepg, by filtration, each soln from the material of column, the solvent is removed by evaporation and the residue is weighed and identified by chemical means. If, in chromatographing colorless compds, there is no satisfactory "streak" reagent available, a colored "tracing compound" may be used (Ref 58,p 54). For example, in the sepn of DMePh from other ingredients of a mixt, a small amt of p-MNA may be added to the soln before it is run thru the column. As p-MNA is yel in color and as its adsorption power is the same as for DMePh, both compds would move thru the column as one yel zone visible thru the tube, thus indicating progress of analysis. Other methods of revealing the position of colorless zones are listed in Ref 58,p 57

Sometimes(especially when only a few compds are in a mixt), it is more convenient not to extrude the column, but instead to subject it to a more prolonged washing directly in the tube. This treatment, called *elution,* will usually separate the components more efficiently into wider bands and these may then be removed(together with solvents) and collected separately in clean receivers as the percolation thru the column continues. Solvents used in washing the column are called *eluents or elution agents* and they may be alcs, eth, acet, dichloroethane, dichloromethane, chlf, et acetate or petr ether(Refs 58 & 78)

If in "elution analysis" several "eluents" are applied, one after the other starting with the liq of relatively low elution power and changing gradually to liqs of increasingly higher elution power, the technique is called "dilution with several eluents"(Ref 78,pp 65-6). It was introduced in 1938 by T. Reichstein et al, under the names of ' 'liquid chromatography" or "flowing chromatography" (Ref 58,p XX). Although this method permits achieving good separations, it has, to a large extent, been replaced by the "gradient dilution", in which mixts of solvents with gradually increasing elution power are used as eluents. It is described in Ref 58,pp 41-3 & Ref 78,pp 66-9

In a modification of "solid-liquid adsorption chromatography", known as *frontal analysis,* or *break-through*(introduced by A. Tiselius in 1940-43), the sample soln is continuously passed thru the column until the latter has become satd with the solutes, ie until the compn of the soln leaving the column would be identical with that of the soln entering the column (Ref 78,p 69). Concns of solns leaving the column may be approx detd by the use of an optical system registering changes in

refractive index based on the "Schlieren" method(Ref 58,p 4). If the solutes are adsorbed to a different extent by the adsorbent, they will require different periods of time in order to travel thru the column and during the course of analysis a number of zones will be formed in the column. The leading part of the zone is called its "front"; hence the name of the method(Ref 78,pp 69-70). In order to achieve a more satisfactory sepn of components, the modification known as "carrier frontal analysis" may be used(Ref 78,p 75)

In another modification of "solid-liquid adsorption chromatography", also devised by A. Tiselius in 1940-43(Ref 58,p XX) and known as *displacement analysis*, the soln of sample introduced in the upper section of the column is continuously rinsed with a soln of a substance which possesses a stronger adsorption power than any of the sample components. This substance, known as "displacer", replaces first the upper zone constituent and this action forces other zones to move ahead of the front produced by the displacer. Finally the zone of the constituent least adsorbed leaves the column and is followed immediately by stronger adsorbed substances including the displacer. The drawback of this method is that the zones cannot be sharply separated because they leave the column close together without being divided by layers of pure solvent. This difficulty has been overcome by using the modification known as "carrier displacement analysis". Here a soln of a number of so-called "carriers" is added to the sample soln. As these are substances of intermediate adsorption affinities betw the components of sample they would place themselves betw the zones of components in the chromatographic column. An important prerequisite of a carrier is that it should be easily separated(such as by chemical means) from the components of sample which are required to be isolated and identified(Ref 58,pp 5-8 & Ref 78,pp 70-5)(See also Refs 24, 31, 40, 48, 51, 59, 61, 70, 72, 74, 76, 83 & 86)

The chromatographic techniques outlined above are now widely used in analyses of expls and proplnts. It seems that Karrer & Nielsen were the first to use chromatography in this field(1934)(See Ref 7) and then no papers were published until after WWII(Refs 17, 20, 25, 27, 28, 29, 30, 32, 33, 35, 44, 64, 71 & 85). Considerable work was done during and after WWII for US Army & Navy by NDRC, PicArsn, CalTech, etc. Results of these investigations were discussed in various reports, such as OSRD, PATR, etc, all of them declassified only recently(See Refs 13, 14, 15, 16, 18, 22, 26, 37, 38, 49, 50 & 89). Detailed descriptions of chromatographic detns of some proplnt ingredients, as prescribed for US Ordnance labs, are given in Refs 105, 106 & 107. Combined chromatographic-spectrophotometric methods for analysesof proplnts are described in Refs 22, 26, 32, 37 & 107 and a combined chromatographic-polarographic method in Ref 79 Chromatographic analysis of some rocket fuels is discussed in Ref 79

B. *Partition Chromatography or Liquid-Liquid Chromatography.* This method is based on the following phenomenon. When a soln of a substance is shaken with an immiscible solvent, the solute would distribute itself betw two phases and when equilibrium is reached, the relation betw concn in the 1st solvent and concn in the 2nd solvent would be a constant, known as "partition coefficient". As various substances have different coefficients, it is possible to effect separation of components of a mixt by means of a solvent-solvent extraction, as was described in Ref 8,p 91 and Ref 35a. Martin & Synge(Ref 8,p 1358) found,however, that a more efficient fractional solvent-solvent extraction can be achieved by packing columns with silica-gel(contg ca 50% H_2O), placing the soln of sample on the column and "eluting" (developing) with water-immiscible solvents, such as chloroform contg small amts of butanol. The liquid held on the column(usually water or hydroxylated polar solvent) is termed "stationary phase", while the "eluent"(usually a non -polar solvent) is known as ' 'mobile phase". Substances other than silica-gel may be used for the column. These include cellulose, kieselguhr, starch or rubber(Ref 58,pp 103ff and Ref 78,pp 92-4 & 112ff). Eluent may also be a vapor(Ref 48,p 61)(See also Refs 51, 61 & 72)

C. *Paper Chromatography*(Papyrography or Partography). In this method, filter paper is used as an adsorbent in lieu of materials used in chromatographic columns. Although the principle of selective adsorption by paper (such as papyrus) was known for centuries and

although methods of analysis using filter paper were proposed and described by at least two investigators of the 19th century(See under Historical), no practical application of paper chromatography was made until about 1944, when the original method was modified by several investigators(Refs 11 & 12; Ref 77,p 5 and Ref 78,p 8)

Paper chromatography may be considered as a branch of "partition chromatography", because the sample on paper is a "stationary phase", while the solvent(eluent) is the "mobile phase"

The technique, in its simplest form, is briefly as follows: Using a micropipet, the soln of sample to test is applied as a spot (or as a streak) at a previously marked(with a pencil) place on a strip of filter paper, at least 3cm wide and 50cm long. After allowing the spot to nearly completely dry, the strip is suspended vertically so that its upper end hangs over the edge of the trough and dips into the solvent("eluent") contained in it. This causes the solvent to be adsorbed by the paper and to migrate down past the spot contg the sample. Its components are then drawn along by the solvent at different velocities(depending on the soly ratio) and thus move at different speeds downwards on the paper. When the solvent front has almost reached the lower end of the paper, the strip is removed and, after marking the position of the front, is allowed to dry. This technique is known as the "descending method", but if the paper strip is hung up with its lower end just dipping into the solvent in the trough, the technique is known as "ascending method". Here the solvent adsorbed by the paper moves upwards and carries with it the components of sample, as in the case of descending method

The resulting treated paper strips, known as "chromatograms", are then dried and sprayed with a reagent capable to produce color reactions with ingredients of sample (Ref 54a, pp 2ff)

There are several modifications of paper chromatography, including the "circular filter chromatography". For detailed description of various techniques, see Refs 31, 34, 42, 47, 48, 50a, 54, 58, 63, 78 & 94

Application of paper chromatography to analysis of explosives, such as substituted trinitrobenzenes, is described in Ref 98 and of some other expls in Ref 108

D. *Gas Chromatography* is a chromatographic separation process which found wide use in analysis of volatile mixtures. The technique consists of pass ing a sample, in the form of vapor or gas, thru a very narrow chromatographic column, serving as a "stationary phase". The column may be either an adsorbent(if it is a solid), or an absorbent (if it is a liquid). In the solid column the material may be silica gel, activated carbon, molecular sieves(such as zeolites) or Al oxide; while in the liquid column the material may be paraffin oil, silicone oil, polyethylene glycols, dinonylphthalate, etc, either distributed over an inert solid support(when in "packed columns"), or as coating on the inner wall(when in "capillary columns"). The solid support of packed column may consist of kieselguhr, Chromsorb, Celite, Teflon, glass powder, glass beads, etc. The technique of employing a solid stationary phase without liquid coating, known as *gas-solid chromatography*(GSC), is not used often because of the limiting number of types of adsorbents suitable for small diameter molecules, while the technique of employing a liquid as a stationary phase, known as *gas-liquid chromatography*(GLC), is used very extensively. The gas chromatography was first described in 1952 by James & Martin(Ref 43). It may be considered as a branch of partition chromatography. See also Refs 58, 77 & 78

Gas chromatographic separation may be conducted by either "frontal analysis", "displacement development" or "elution technique". In the latter technique, which is now predominantly used, a sample is injected into moving stream of inert gas(such as helium, nitrogen, hydrogen or CO_2) and the gas carries the components thru the column at rates dependent on their volatilities and interaction with the non-volatile liquid phase. This gas is called "carrier gas". As the " moving phase" proceeds down thru the ' 'stationary phase", the various molecules tend to dissolve in the liquid of column and some of them revaporize. The molecules of the component absorbed to the greater extent in the liquid of the column are retarded in their passage, while others leave the column earlier, together with the carrier gas. Then, gradually, all other

components follow. The effluent leaving the column passes thru a device capable of detection and characterization of components by measuring their chemical or physical properties. A detailed description of gas chromatography is given in Refs 58,77,78,92 & 93)(See also Refs 52,55,56,60,62,66,67,68,73,75,81,82,84, 85,87,88,89,90,91,95,99,100,102,103,104,109 & 110)

A branch of gas chromatography, known as *programmed temperature gas chromatography* (PTGC), allows one to vary the temp of the column during an analysis. The method is described in Refs 70a, 77 & 99(Compare with Item I)

Parsons et al(Ref 83a) applied gas chromatography to separation of MNT and DNT isomers from their mixts. Ettre & Váradi(Ref 85) used it in analysis of thermal degradation products of polymers, such as NC. Kuwada (Ref 89) detd w in hydrazine and de la Porte & Lightenberg (Ref 100) detd triacetin in NG propint. Wilhite(Ref 100a) designed a gas chromatograph intended to be soft-landed on the surface of the moon as part of the "Surveyor" spacecraft. It is assumed that the gas chromatograph would conduct, while on the lunar surface, an analysis of volatile constituents found on the moon

E. *Electrochromatography.* It is defined by Heftmann(Ref 78,p 14) as "a method of analysis in which direct current electrical potential promotes the separation of substances by differential migration from a narrow zone in a stabilized background electrolytic solution". For more info see Ref 58,pp 10-12 and Refs 65, 72 & 86)

F. *Ion Exchange Chromatography.* It is a process whereby a soln of ions is conducted thru a column consisting of an insoluble solid ion exchanger(such as ion exchange resin) saturated with a solvent. This action separates some of the constituents. This method was primarily designed for separation of fission products in connection with the work on atomic energy in the US(Refs 47a, 48a, 58, 78 & 86)

G. *Molecular Sieves Chromatography.* It is a method of separation using chromatographic columns packed with "molecular sieves"(Refs 58, 78 & 82a). Molecular sieves are solid materials possessing a very high sorption ability(for small molecules), because they contain a very large number of extremely small pores and openings. The most important sieves are cryst substances obtained by heating hydrated aluminosilicates(such as natural or synthetic "zeolites") until the water lodged in pores evaporates. Other substances, such as xerogels and starch, may also be used as molecular sieves(Refs 54a, 58, 76a, 78 & 82a). One of the applications of molecular sieves is the dehydration of jet fuels(Ref 82a,p 106)

H. *Thin-Layer(or Thin-Film) Chromatography.* In this method thin layers of adsorbents(such as silica gel, Al oxide, etc) are bound to glass plates(usually 20x20cm or 20x5cm) by a suitable binding agent(such as starch adhesive). These adsorption plates are often called *chromatoplates* or *chromatostrips.* By means of a micropipet, a small drop of sample soln is placed ca 20mm from side and bottom of the plate with other drops placed 10mm apart. Then the plate is placed nearly vertically in a chamber filled with the developing solvent to a height of ca 5mm and the chamber closed to prevent the evapn of solvent. After allowing the liquid to rise to a height of ca 10cm(which takes 20-60 mins), the plate is removed and dried(at ca 50°). If resulting chromatogram is colorless or nonfluorescent, the plate is sprayed with a reagent which produces colors with component of sample, but does not dissolve or react with the adsorbent(Ref 78,pp 87-90 & 113)(See also Ref 58,p 12, under Miscellaneous; Ref 86,p 7R and Ref 96). Hansson(Ref 97) describes application of thin layer method to sepn of the following expls: PETN, DINA, RDX, HMX, Tetryl, TNB, TNT, HNDPhA and AN(See also Refs 81a & 111)

I. *Chromathermography.* The term coined by Zhukhovitskii et al(Ref 41) for a chromatographic analysis in which a stream of air is applied while the furnace(which heats consecutive sections of the adsorbing column, and causes desorption) is moved down the column. The air stream thus distributes the components at different spots of the temperature field, and keeps them separated. The method, first proposed in 1951, was discontinuous(Ref 41 & 45), but later(Ref 46), the continuous modification, called "thermal dynamic method" was devised (Compare with "programmed temperature gas chromatography" listed under item D)

J. *"Inverted" Chromatography.* In this modi-

fication of chromatography proposed by Lowman (Ref 8a), the adsorbent is poured or dropped thru a column of the soln of substances to be adsorbed(See also Ref 58,p 13). The term "inverted chromatography" is not listed by Heftmann(Ref 78)

This section on chromatography was reviewed by Delbert J. Cragle of Picatinny Arsenal

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Chrome Alum or Potassium-Chrome Alum. See Vol 1, p A156-R and pA156-)L for Ammonium-Chrom Alum

Chrome-Ammonites Ger pre-WWI coal-mine expls: a) AN 63.25, (nitrate 17.5, CC 9.25, K chrome alum 9.5 &vaselin or paraffin 0.5% (Ref 3) b) AN 70.0, K nitrate 10.0, TNT 12.5, K chrome alum 7.0 & vaselin or paraffin 0.5% (Refs 3 & 4) c) AN 63.25, K nitrate 17.5, CC 9.25, Amm chrome Alum 9.5 & vaselin 0.5% (Ref 2) d) AN 70.0, K nitrate 10.0, TNT 12.5, Amm chrome alum 7.0 & vaselin 0.5% (Refs 1 & 5). The last expl was called *Reinforced Chrome-Ammonite* (Verstärktes Chromamonit in Ger)
Refs: 1)Colver (1919), 248 & 250 2)Marshall, Dict (1920), 22 3)Thorpe, Suppl **1** (1934), p 456 4)Thorpe, Vol 4 (1940), 554 4a)PATR **2510** (1958), p Ger 28 (Chromammonite) 5) Giua, Trattato **6** (1959), 372

Chrome Ammonium Alum. See Ammonium -Chrome Alum in Vol 1, p A156-L

Chrome Greens. Paint pigments which are mixts of ppts of chrome yellow (PbCrO4) and iron blues, the latter obtained by pptg a ferrous ferrocyanide from potassium ferrocy-anide and ferrous sulfate (Ref 1). US Govt requirements and tests are given in Ref 2
Refs: 1)CondChemDict (1961), 271 & 615 2)US Specification **TT-C-235(3)** (Chrome-green, pure, paint pigment)

Chrome Iron Ore. see Chromite

Chromel. Trade name for a series of Cr-Ni alloys
Ref: CondChemDict (1961), 271

Chrome Orange. See Chrome Red

Chrome Plating of Gun Barrels in Germany. See PATR **2510** (1958), p Ger 28-R

Chrome Potash Alum. See Potassium-Chrome Alum, Vol 1, p156-R

Chrome Red or Chrome Orange. (American Vermilion). A paint pigment consisting of basic Pb chromate which approximately corresponds to the formula $PbCrO_4$. PbO, with some variation in the proportion of the PbO and $PbCrO_4$ (Ref 1). US Govt requirements and tests are given in Ref 2
Refs: 1)CondChemDict (1960), 272 2)US Specification **TT-C-290(3)** (Chrome yellow and chrome orange dry paint pigments)

Chrome Yellows. Yel paint pigments contg 95% or more $PbCrO_4$ (Ref 1) (See also Lead Chromate). US Govt requirements and tests are given in Ref 2
Refs: 1)CondChemDict (1961), 272 2)US Specification **TT-C-290(3)** (Chrome Yellow and Chrome Orange paint pigments)

Chromic Acetylacetonate. Same as Chromium Acetylacetonate

Chromic Acid. (Chromic Anhydride or Chromium Trioxide), [Chrom (VI)-oxyd in Ger]. CrO_3, mw 100.01 (The name "chromic acid" is in common use, although the true acid H_2CrO_4, exists only in soln). Dark purplish-red deliq crysts, mp 197° (dec), d 2.70 at 20°; v sol in w; sol in alc & ether. Solubilities in w at different temps are given in Ref 2a, p1996. Can be prepd by treating Na dichromate with concd

sulfuric acid and crystallizing the resulting CrO_3, or by other methods (Refs 1, 2, 2a, 3, 5 & 6). Its toxicity and fire hazard are discussed by Sax (Ref 4). Davis (Ref 1a) described the use of chromic acid for prepn of TNB from TNT. Chromic acid is a parent compd of chromates and dichromates. It is a powerful oxidizing agent and violent explns or fires can be produced on intimate contact with strong reducing agents. US Govt requirements and tests for chromic acid are given in Ref 7

Refs: 1)Mellor **11** (1939), 211-40 1a)Davis (1943), 135 2)Kirk & Othmer **3** (1949), 945-6 2a)W.H.Hartford, IEC **41**, 1994-6 (1949) 3)Ullmann **5** (1954), 584-5 4)Sax (1957), 484 5)CondChemDict (1961), 272-3 6)Lange (1961), 242-3 7)US Specifications MS-36039 &O-C-3038 8)Anon "Properties of Materials Used in Pyrotechnic Compositions", US Materiel Command Pamphlet, **AMCP 706-187**, Washington, DC, 20315 (1963), pp96-8

Addnl Ref: a)G.A.Tuey, Chem & Ind **1948**, 766 & CA **43**, 3199 (1949) (A violent deflagration might take place on dissolving chromic acid in acetic anhydride as occurred in 1948 in GtBritain) b)D.A.Peak, Chem & Ind **1949**, 14-15 & CA **45**, 7791 (1951) (In order to avoid a spontaneous deflagration which might occur in prepn of an oxidizing reagent from chromic acid and acetic anhydride, the temp should be kept below 30°, while adding chromic acid in small portions, with const stirring of cooled acetic anhydride) c)T.Urbański, BullAcadPolonSci, SerSci **8**, 13-14 (1960) (in Eng) & CA **54**, 12023-4 (1960) (Use of chromic acid in nitration of toluene)

Chromic Anhydride. See Chromic Acid

Chromic Azide. See Chromium Triazide in Vol 1, p A530

Chromic Azide Complexes. See Chromium Azide Complexes in Vol 1, pA530-R; and also Azidopentammine-chromium (III) Perchlorate, Chloropentamminechromium (III) Azide and Hexamminechromium Azide on pA277 under Ammines

Chromic Chloride. See under Chlorides

Chromic Hydroxide. See under Hydroxides

Chromic Nitrate. See under Nitrates

Chromic Oxide. (Chromium Oxide, Chromium Sesquioxide, Chromium Hemitrioxide or Green Cinnabar) [Chrom (III)-oxyd in Ger], Cr_2O_3, mw 152.02, dk-grn amorphous pdr or hex crysts, mp 1990°, d5.21; insol in w, s l sol in alc; can be prepd by heating Amm dichromate or Na dichromate with sulfur and washing out the Na sulfate (Refs 1, 2, 3&5). An industrial method is briefly described in Ref 2, p945. Chromic oxide has been used in metallurgy, in manuf of soluble Cr salts, as a green pigment in manuf of glasses and ceramics and as a catalyst. Cr_2O_3-CuO catalysts are widely used, such as in manuf of methanol and in cyclizations of hydrocarbons (Refs 2 & 5). Its use in smoke-producing pyrotechnic compns is discussed under Chromium Compounds for Smoke Production. Chromic oxide, as well as Cr hydroxide, are parent compds of Chromites

Refs: 1)Mellor **11** (1931), 176-85 2)Kirk & Othmer **3** (1949), 944-5 3)Ullmann **5** (1954), 590-1 4)Sax (1957) - not found 5)CondChemDict (1961), 273 6)Lange (1961), 240-1 7)Gmelin, Syst Nr **52** (1962-) 8)Anon "Properties of Materials Used in Pyrotechnic Compositions", US Materiel Command Pamphlet, **AMCP 706-187**, Washington, DC 20315 (1963), pp99-102

Chromihydrazoic Acid, Silver Complex. See Vol 1, pA531-L, under Chromium Azide Complexes

Chromite or Chrome Iron Ore. (Chrome Iron Stone) (Chromerz, Chromeisenstein, Chromit or Ferrochrom in Ger), $Cr_2O_3.FeO$ or $Fe_2(CrO_2)_2$. This ore, which is the most important commercial source of chromium and and its compds, is not found in nature in pure form but mixed with oxides of Al and/or Mg. Its color varies from iron-blk to brn-blk or streak dr brn. There are several grades: metallurgical, refractory, or chemical; av d 4.6 and hardness 5.5. Several methods for production of chromium from chromite are known, such as silicothermic, aluminothermic & electrolytical

Refs: 1)Mellor **11**, 123-5 2)Thorpe **3** (1939), 94-6 3)Kirk & Othmer **3** (1949), 944-5 4) Ullmann **5** (1954), 571-82 5)CondChemDict (1961), 271 6)Gmelin, Syst Nr **52**, Teil A, Lieferung 1 (1962), 196-213

Chromites. Salts of general formula $MCrO_2$, where M is a monovalent metal. They may be regarded as *metachromites* derived from hypothetical metachromous acid, $HCrO_2$, which is known only in solns or in the form of its oxide, Cr_2O_3. The most common chromites, $NaCrO_2$ and $KCrO_2$, can be obtained by the action of chromic oxide, Cr_2O_3 on Na or K hydroxide (Ref 1, p197). Although these and other chromites are oxidizers, they do not contain as much oxygen per unit wt as do chromates or dichromates There is no open literature info at our disposal that chlorites have been used in expls, proplnts, or pyrotechnic compns
Refs: 1)Mellor **11** (1931), 196-206 2)Partington (1950), 887 3)Gmelin, Syst Nr **52**, TlA, Lfg 1 (1962), 285ff

CHROMIUM AND ITS COMPOUNDS

Chromium. (Chrom in Ger, Chrom in Fr, Cromo in Ital & Span and Khrom in Rus), Cr, at wt 52.01; steel-gray, hard cryst metal, mp 1903 + 10°pp 2642,° d 7.14 at 29°, vap pressure 1mm at 1616°; insol in w & in nitric acid; sol in HCl, strong alkalies and dil sulfuric acid. Can be prepd by reducing chromic oxide, Cr_2O_3, by the thermite process using powdered Al or by other methods (Refs1,2,3,4,5,6,8&10). The usual starting material for industrial prepn of Cr metal is chromite (qv) and the methods of its treatment are: a)Silicothermic Process, which is based on the reaction $2Cr_2O_3+3Si=4Cr+3SiO_2$ b)Aluminothermic Process, which is based on the reaction $Cr_2O_3+2Al=2Cr+Al_2O_3$ and c)Electrolytic Process, which is used for electroplating thin films of Cr on other metals and to produce powdered or flaked Cr for use in powder metallurgy (Ref 3, p938). Cr does not react with Na picrate to form Cr picrate (Ref 3a). Cr metal is practically non-toxic and the fire hazard of its dust is moderate. It has been used in many alloys, stainless steel, for chromium -plating, high temperature research and in nuclear energy research (Ref 8)
Refs: 1)Mellor **11** (1931), 122-70 2)Thorpe **3** (1939), 96-7 3)Kirk & Othmer **3** (1949), 935-38 3a)O.E.Sheffield, PATR **1783** (1950) 4)Partington (1950), 887 4a)A. Sully, "Chromium", Academic Press, NY (1954) 5)Ullmann **5** (1954), 556-98 6)M.J. Udy, "Chromium", Reinhold, NY (1956), ACS Monograph, No 132 (Vols 1 & 2) 7)Sax (1957), 485 8)CondChemDict (1961), 274 9)Lange (1961), 272-73 10)Gmelin, Syst Nr **52** (1962)

Chromium, Analytical Procedures. See Vogel, InorgAnalysis (1961) 311, 520-22, 791 & 953 and StdMethodsChemAnalysis1 (1962), 350-76.Analytical procedure for detn of Cr content in dichromated aluminum is described by W.J.Huff in PicArsnGenLabRept **59-H1-289** (1959)

List of Chromium Compounds

Chromium Acetylacetonate or Chromylacetylacetone. See Vol 1, pA53-R
Addnl Ref: F.Bellinger et al, IEC **40**, 1330 (1948) & CA **42**, 6115 (1948) [It was found that Cr acetylacetonate added in quantities of about 2% (together with 4% of gasoline) to nitromethane, prevented its detonation when used a rocket proplnt such as for launching a buzz bomb]

Chromium Acetylide. See Chromium Carbide in Vol 1, pA72-R

Chromium (III) ammine Complexes. These include Triazido-triamminechromium(III), Aquopentamminechromium(III) Perchlorate, Azidopentamminechromium (III) Perchlorate, Chloropentamminechromium (III) Azide, Hexamminechromium (III) Azide, Hexamminechromium (III) Nitrate and Hexamminechromium (III) Perchromate described in Vol 1, pA277
[See also W.R.Tomlinson, Jr. PATR **1364** (1943) & PATR **1632** (1946) (Expl props of chromium- and cobalt-complexes]

Chromium Ammonium Alum or Chromium Ammonium Sulfate. See Ammonium-Chrome Alum in Vol 1, pA156-L

Chromium (III) azidopentammine Perchlorate. See Vol 1, pA277

Chromium Borides. At least three cmpds are known: CrB (crysts, mp 1550°, d 6.21 & Moh's hardness 8.5); CBr_2 (crysts, mp 1850°, d 5.15, hardness 2010 Knoop; resists oxidation up to 1100°); and CBr_3 (crysts, d 6.1 & Moh's hardness 9+). Because of their high mp's, high hardness and corrosion resistance, they were proposed for use in jet and rocket engines (See also Borides in Vol 2,pB249-R) *Ref:*CondChem Dict (1961), 274

Chromium Carbide. See Vol 1, pA72-R, under Acetylides

Chromium Chlorate. See Chromous Chlorate in Vol 2, pC187-R

Chromium Chloride. See under Chlorides in this Vol

Chromium (III) chloropentammine Azide. See Vol 1, pA277

Chromium Compounds for Smoke Production. J.DeMent, USP 2995526 (1961), p 8, proposed to use the following mixts for smoke-producing pyrotechnic items: a)Cr stearate 1.0 to 3.0, K chlorate 4.0 to 12.0 & Amm acid fluoride 2.0 to 8.0 parts. This compn, when ignited by a flame, produced on burning a heavy copious, lavender-gray smoke and sublimate b)When using mono- or tri-chloroacetic acid in previous mixt, instead of fluoride, the color of smoke produced was grayish-black to blackish c)Equal parts of chromic oxide, K chlorate, K bromide, K bromate, sulfur & K acid sulfate gave a very fast burning admixture which emitted a heavy grayish smoke d)Upon the addn of chromic oxide 1.0 to the admixt of K bromate 1.0, sulfur 1.0 & K bromide 1.0 part, the deflagration started spontaneously, proceeding very quickly and emitting a whitish smoke e)Equal parts of chromic oxide, K chlorate, K iodide, K iodate & sulfur deflagrated on contact with a flame with evolution of a heavy, copious deep violet smoke changing to purple-gray

Chromium Dioxychloride. See Chromyl Chloride under Chlorides in this Vol

Chromiumethylenediamine Complex. See Chromium (III) tris (ethylenediamine) Peroxysulfate in this section

Chromium Hemitrioxide. See Chromic Oxide

Chromium(III) hexammine Complexes. See Vol 1, p 277

Chromium (III) hexaurea Complexes, also called *Chromium Hexacarbamides,* of general formula $[Cr(H_2N.CO.NH_2)_6]X_3$, where X stands for chlorate, chromate, nitrate, nitrite, perchlorate, etc, are briefly described in Mellor (Ref 1). They can be prepd by crystg from solns of urea and Cr salt. Tomlinson et al (Ref 2) detd the following expl props of *chromium (III) hexaurea nitrate,* $[Cr(H_2N.CO.NH_2)_6](NO_3)_3$:
Explosive Temperature (5 sec) 265°; *Impact Sensitiveness* (2 kg wt) 50cm; and *Sand Test* 3.0g vs 42.0g for TNT *Refs:*1)Mellor **11**(1931), 401 2)W.R.Tomlinson Jr, K.G.Ottoson & L.F.Audrieth, JACS **71**, 376 (1949)

Chromium Nitrate or Chromic Nitrate, $Cr(NO_3)_3 9H_2O$; mw 400.18; purple, rhombic crysts; mp 36.5°, decomp ca 100°; sol in w & alc. Can be prepd by the action of nitric acid on Cr hydroxide (Refs 1,2&4). Used as a mordant in textile dyeing. An anhydrous salt, $Cr(NO_3)_3$, was reported to be obtained on evapg a nitric acid soln of chromic oxide, dissolving the resulting dk brn mass in w and crystg. The anhydrous salt takes up moisture from the air to form the trihydrate (Ref 1). There exists also the hydrate, $Cr(NO_3)_3 . 7.5H_2O$ (Ref 3) *Refs:* 1)Mellor**11** (1931), 474 2)Kirk & Othmer **3** (1949), 944 3)Sax (1957), 485 4)CondChemDict (1961), 273

Chromium Oxide. See Chromic Oxide

Chromium Oxychloride. See Chromyl Chloride in this Vol, under Chlorides

Chromium Picrate, Basic, $[C_6H_2(NO_2)_3.O]Cr$, $2C_6H_2(NO_2)_3 .O.CrO, 27H_2O$; grn crysts, N ca 11.38%; mp-expl at 330° (Ref 4 for salt confg $13H_2O$); can be prepd by mixing equivalent quantites of Ba picrate & Cr sulfate

and evapg the filtrate in vacuum over sulfuric acid. On heating the above hydrate to 80°, the ennea ($9H_2O$) hydrate was obtained and on heating to 150° the anhydrous salt formed (Refs 1&2). A monohydrate was also reported and its sensitivity to impact was detd by Hopper as 8" with 2kg wt, using PicArsn apparatus (Ref 3)
Refs: 1)Beil **6**, 278 2)O.Silberrad & H.A. Phillips, JCS **93** 486 (1908) 3)J.D.Hopper, PATR **764** (1936) 4)Davis (1943), 165

Chromium Potassium Alum or Chromium Potassium Sulfate. See Vol 1, pA156-R

Chromium Sesquioxide. Same as Chromic Oxide

Chromium Stearate, $Cr(C_{18}H_{35}O_2)_3$; blue-grn pdr, mp-softens ca 75°(Ref 4). Was prepd by Koenig (Ref 3) by pouring (with some stirring) a warm aq soln of Na stearate into an excess of a 1% soln of Cr chloride or sulfate. Lawrence (Ref 4, pp667-70) used a more complicated method of prepn. Solubilities in different solvents are given in Ref 3, pp354-55. Stearates of Cr, Co, Ca, Li & Zn were investigated at PicArsn (Ref 5) and found to be suitable as additives to semi-plastic expls, such as RDX Compositions A-3 and A-4 in order to eliminate the adherence of expl to dies during continued pelleting operations. It was also found that the above stearates facilitated press-loading operns. Following are examples of mixts tried at PicArsn: a)Comp A-3 97.5%, stearate 2.5% with 0.5% graphite added b)Comp A-4 98.0% & stearate 2.0%. Stearates can be replaced partly or completely by talcum powder. Graphite added to the mixt during coating of Comp A-3, was found to improve the ease of coating by reducing the electrostatic chge of powdered material
Refs: 1)Beil **2**, 379, (172) & [350] - not found 2)Beil **2**, {1010} 3)A.E.Koenig, JACS **36**, 952-56 (1914) 4)A.S.C.Lawrence, TrFaradSoc **34**, 665 & 771 (1938) 5)A.L.Forchielli, PATR **1787** (1950)

Chromium Triamminotetroxide, $CrO_4.3NH_3$; brn crysts; mp-detonates and becomes incandescent forming chromic oxide; d 1.964 at 15° can be prepd by method of Riesenfeld from chromic acid, ammonia & hydrogen peroxide as described in Refs 1&2
Refs: 1)E.H.Riesenfeld, ZAnorgChem **74**, 48 (1912) 2)Mellor **11** (1931) 358

Chromium Trioxide. See Chromic Acid

Chromium (III)tris(ethylenediamine) Peroxysulfate, $[Cr(en)_3]_2(S_2O_8)_3$. It was reported by Beacom that this compd can be ignited by flame and exploded by exposing it to ultraviolet radiation (3650A°), followed by heating to 115°. Its method of prepn was not described by Beacom; and we could not find this compd listed in Beil or in the alphabetical indexes of CA's (except the listing indicated in Ref 1)
Note: en is abbreviation for ethylenediamine
Refs: 1)S.E.Beacom, Nature **183**, 38-9 (1959) & CA **53**, 11087 (1959) 2)Anon, C&EN **37** No 15, p70 (April 15, 1959)

"Chromosorb". Trademark for a line of closely graded (screened) calcined or flux-calcined diatomic aggregates, manufd by Johns-Manville, New York 16, NY, for use in vapor-phase gas chromatography
Ref: CondChemDict (1961) 275

Chromous Chlorate. See Vol 2, pC187, under Chlorates

Chromylacetylacetone. See Chromium Acetylacetonate in Vol 1, pA53, under Acetylacetone and Derivatives

Chromyl Chloride. See under Chlorides in this Vol.

Chronoamperometry, Chronopotentiometry, Chronopolarography and Oscillographic Polarography. A "steady state" is said to be reached when the current-potential curve of a soln examined by the usual electrochemical methods (such as potentiometric titration, voltammetry, etc) becomes independent of time (at least for the period of time needed to carry out the measurements). However there are electrolitic phenomena which change with time, resulting in the existence of an unsteady

state at the time of measurement. At constant concn, these phenomena can be described by a function of three variables: current, potential and time. In practice the following methods or techniques have been used:

A. *Chronoamperometry.* There are two types of technique:

a) *Chronoamperometry at Constant Potential.* An electrochemical technique in which the electrode potential is kept constant at such a value that the electrochem reaction in question occurs at a measurable rate (current). The soln is not stirred and the electrodes are fixed while the variation of the electrolysis current, with time, is measured. This technique while used for studying electrochem reactions has rarely been used for analytical purposes(Refs 9,15&31)

b) *Chronoamperometry with Linearly Varying Potential* (Linear Chronoamperometry). A technique in which the electrode potential is varied linearly with time, with the initial potential chosen so that the electrochem reaction in question occurs at a negligible rate. If the potential sweep is allowed to occur once at a low rate, the method is called "single sweep" and if many periodic sweeps occur, the method is known as "multi-sweep" (See also under item D). In the case of the "single sweep" if the steady state did not have time to establish itself, the recorded current-voltage (or time) curve results, showing maximum (or minimum) peak. The peak current is proportional to the concn and increases with the rate of potential chge. This method can be used in electrochem analysis employing either solid or hanging mercury drop electrodes. The sensitivity is much higher than that of conventional polarography and has been used to det concns down to 5×10^{-7}M (Refs 3, 11 & 31)

B. *Chronopotentiometry* (Formerly called Voltammetry at Constant Current). These terms were applied by Delahay et al (Refs 4&5) to measurements in which the course of polarization of an electrode (immersed in an unstirred soln) under forced constant current was followed potentiometrically as a function of time. The potential-time curve recorded in the presence of a depolarizer is characterized by a transition time, during which the rate of change of potential is relatively small. This time interval is of primary analytical importance because of its dependence on concn. The sensitivity of this technique is excellent and its accuracy is greater than that of conventional polarography. This method has found wide application in analytical chemistry. For addnl info on this subject see Refs 6,7,8,10,12,14,16,17,18,19,20,21,22,24,25,26 28,29,30,31 & 32

C. *Chronopolarography.* The term proposed by Barredo (Ref 1a) for polarographic method of investigating the variation of current with time (recorded oscillographically) of phenomena that occur at electrode-solution interface (Compare with item D)

D. *Oscillographic Polarography.* Accdg to Charlot et al (Ref 31, p308), this term includes all electrochemical techniques where the voltage or current is varied periodically ("multi-sweep" method) and the cathode-ray oscilloscope is used as the measuring instrument. An example is "Oscillographic Polarography with Controlled Potential." In this technique the potential applied to the dropping mercury electrode is varied linearly with time. The curves obtained on the cathode-ray screen have the same form as those for linear chronoamperometry. This technique has greater resolving power of halfwave potentials and greater speed for analysis than conventional polarography. For addnl info on this subject, see Refs 1,2,13 &27

This section on Chronoamperometry, etc, was written in collaboration with Charles Ribaudo of Picatinny Arsenal

Refs: 1)P.Delahay, JPhysColloidChem **53** 1279-1301 (1949); Ibid **54**, 402-11 & 630-39 (1950 (Oscillographic polarography) 1a)J.G. Barredo, JChemPhys **19**, 1065-66 (1951), (Chronopolarography) 2)T.Berzins & P. Delahay, JACS **75**, 555-59 (1953) (Oscillographic polarography with controlled potential) 3)C.A.Streuli & W.D.Cooke, AnalChem **26**, 963-70 (1953) (Chronoamperometry with linearly varied potential using polarized Hg pool cathode) 4)P.Delahay, "New Instrumental Methods in Electrochemistry", Interscience, NY (1954), Chapters 3,4,5,6,7 & 8 5)P. Delahay & G.Mamantov, AnalChem **27**, 478-83 (1955) (Voltammetry at constant current; theoretical principles. The term "chronopotentiometry is recommended as less cumbersome

than the expression "voltammetry at constant current") 6)C.N.Reilley, AnalChem **27**, 483-91 (1955) (Voltammetry at const current-experimental evaluation) 7)C.N.Reilley & W. G.Scribner, AnalChem **27**, 1210-15 (1955) (Chronopotentiometric titrations) 8)L. Gierst & P.Mechelynck, AnalChimActa **12**, 79-91 (1955) (Chronopotentiometry at const current and in non-agitated soln) 9)H. Gerischer & W.Vielstich, ZPhysChem (Frankfurt a/M), **3**, 16 (1955); Ibid **4**, 12 (1955) (Chronoamperometry at const potential) 10)T Tsukamoto, Polarography(Japan) **4**, 3-11 (1956) & CA **50**, 11852 (1956) (Chronopotentiometry at const current; a review) 11)J.W. Ross et al, AnalChem **28**, 1768 (1956) (Chronoamperometry with linearly varied potential using hanging Hg drop stationary, electrode) 12)L.Gierst, AnalChimActa **15**, 262-70 (1956) (Chronopotentiometry, with const current, of non-agitated solns employing the device called in Fr "transitomètre de précision") 13)G.W.C.Milner, "The Principles and Applications of Polarography and Other Electroanalytical Processes", Longmans, Green & Co, London (1957), 119-34 14)F.C.Anson & J.J.Lingane, JACS **79**, 1015-20 (1957) (Anodic chronopotentiometry with Pt & Au electrodes) 15)W.Vielstich & P. Delahay, JACS **79**, 1874-76 (1957) (Chronoamperometry at const potential) 16)Y. Takemori et al, JPhysChem **61**, 968-69 (1957) (Alternating current chronopotentiometry) 17)T.Kambara & I.Tachi, JPhysChem **61**, 1405-07 (1957) (Chronopotentiometry-stepwise potential-time curves for arbitrary number of reducible species) 18)R.H.Adams et al, AnalChem **30**, 471-75 (1958) (Chronopotentiometric studies with solid electrodes) 19)P.J.Elving & A.F.Krivis, AnalChem **30**, 1648-52 (1958) (Anodic chronopotentiometry with a graphite electrode; analytical applications) 20)J.D.Voorhies & N.H.Furman, AnalChem **30**, 1656-59 (1958) (Quantitative anodic chronopotentiometry with Pt electrode) 21)D.E.Dieball, UnivMicrofilms (Ann Arbor, Mich), Publ No **24553**, 118pp; Dissertation Abstr **18**, 50-1 (1958) & CA **52**, 6051 (1958) (Evaluation of chronopotentiometric method of analysis) 22)Y.Takemori et al, ZPhysChem (Leipzig), Sonderheft, July **1958**, 89-96 & CA **53**, 5952 (1959) (Chronopotentiometry. A graphic method for simultaneous analysis of several components) 23)K.J.Martin & I. Shain, AnalChem **30**, 1808-10 (1958) (Differential voltammetry using the hanging Hg drop electrode) 24)N.Tanaka & T.Murayama, ZPhysChem (Frankfurt a/M) **14**, 370-74 (1958) (Analytical chronopotentiometry) 25)J.J. Lingane, "Electroanalytical Chemistry", Interscience, NY (1958), Chap 22 (Chronopotentiometry) 26)G.Charlot, "Modern Electroanalytical Methods", Elsevier, Amsterdam (1959) 27)J.Heyrovsky, "Oszillographische Polarographie mit Weckselstrom", AkadVerlag, Berlin (1959) 28)J.D.Voorhies, UnivMicrofilms (Ann Arbor, Mich), LC Card No **Mic 59-5237**, 222pp; Dissertation Abst **20**, 2596-7 (1960) & CA **54**, 9551 (1960) (Voltammetry at controlled current) 29)W.H.Reinmuth, Anal Chem **32**, 1514-17 (1960) (Chronopotentiometric potential-time curves and their interpretation) 30)P.Delahay, "Advances in Electrochemistry and Electrochemical Engineering", Interscience, NY (1961) 31)G.Charlot, J.Badoz-Lambling & B. Trémillon, "Electrochemical Reactions. The Electrochemical Methods of Analysis", Elsevier; Amsterdam (1962), 309-10 (Chronoamperometry at const potential); 310-13 (Chronoamperometry with varying potential); 314-18 (Chronopotentiometry); 318-24 (Oscillographic polarography) 32)D.N.Hume, Anal Chem **34**, 178R-179R (April 1962) (Chronopotentiometry; a review)

CHRONOGRAPHS,

Chronoscopes, Chronometers and Other Devices Used in Measuring Velocities of Projectiles in Flight and of Detonation Velocities of Explosives. Accdg to definition given in Ref 46, a *chronograph* is an instrument for measuring and recording time, while a *chronoscope* is an instrument for the precise measurement of small time intervals. A *chronometer* is a very precise clock or watch and, if constructed so that it can be started or stopped (for precise timing) at will, it is known as a *stopclock* or *stopwatch*. It does not seem that any distinction betw the terms "chronoscope" and "chronograph" is made when instruments for time measurements are used in exterior ballistics (such as for detn of velocities of projs), or in detn of

some props of expls (such as their deton vels). For example in the work of 1873 (Ref 1) the instruments of Schultze, Le Boulengé, Vignotti and Benton were called chronoscopes, while in later works the same instruments were called chronographs

Historical on Chronographs and Other Devices Used in Measuring Velocities of Projectiles. The earliest known device was conceived in 1707 by Cassini, but the first practical instrument based on his idea was not constructed until 1740 by Robins of England. This device, known as "Ballistic Pendulum Chronograph" should not be confused with "Ballistic Pendulum" which is used for detn of power of expls (See Vol 1, ppVII-VIII of Encyclopedia). In the Robins instrument, projectiles of known wt, fired by different chgs of propInt, were made to hit the lower part of the pendulum, to which a heavy wt was attached. The velocity of projectile would be a function of length of pendulum, its wt and size of swing and could be calcd from formulas given in Ref 10 pp 39-51. This instrument underwent in later years many improvements, such as that by Hutton (1775), Didion-Morin-Piombert (1836), Piombert (1860) and Minarelli-Fitzgerald (1900-01) (See also Ref 42, pp 262-3)

Other older methods of detn of vel of projs were based on different ideas. For example, Bernoulli (1738) calcd muzzle velocity from the time, detd by a chronometer, required for a proj of a known wt to fly straight upwards, and then return to the ground, while Lombard calcd it from the distance and time required for a proj to fly at a known angle (Ref 10, p36). In the device of Mathey (1773), two hollow, concentric cardboard cylinders, one stationary and the other rotating around its vertical axis, were used. A bullet was shot thru both cylinders perpendicular to their axes The two holes made by the bullet in the stationary cylinder would form an angle of 180°, while the angle in the rotating cylinder would be somewhat smaller. The difference betn these two angles would be a function of the velocity of the bullet as well as of the speed of the rotation of the cylinder (Ref10, p36). This device was improved in 1907 by Hartmann & Braun Co of Frankfurt a/M (See Ref 10, p37)

The device of Grobert (1801, modified in 1804), called Rotating Disks Chronograph (Drehscheiben-Chronograph in Ger) consists of two cardboard disks, divided into radial degrees and mounted vertically 4m apart on a common horizontal axis. A bullet shot in a trajectory perpendicular to both disks while they were stationary would make holes located at the same angle from 0°; but if the disks were rotated at a high, uniform speed, the hole in the disk located farthest from the weapon's muzzle would be farther from 0° than the hole in the 1st disk. The smaller the difference betw these two angles, the higher would be the vel of bullet, provided the speed of rotation of the disks and the distance betw them were the same in all tests (Ref 10, p37 & Ref 42, p262)

The above listed Ballistic Pendulum Chronograph, and devices of Mathey and of Grobert are purely mechanical. To this type belong also the chronograph of Debooz, invented in 1834 in France. It consisted of a metal target plate suspended from a string or wire at a distance of 50m from the muzzle of the weapon to test. The string rested on two pulleys, one of which was near the person firing the weapon. At the instant of firing, the end of the string nearer the weapon was released, thus allowing the plate to fall by gravity. The distance, (h), the plate had fallen betw the time the string was released and the instant of impact of proj on the plate, was measured, and the time required for the proj to travel the distance of 50m was calcd from the formula $t=\sqrt{2h/g}$ sec. This gives vel of proj equal to $50/t$ m/sec (Ref 10, pp 38-9)

In a recent Ger review on chronographs, Rumpff (Ref 42) subdivided the past and present instruments into the following four classes:

1) *Mechanical.* This includes the chronographs described under Historical

2) *Mechanical-Electrical.* This included: a)Spark Chronograph of Siemens (1845) (Ref 10, p87 & Ref 42, p266) b)Pendulum Chronograph of Navez (1853) (Ref 10, p79) and c)Falling Rod Chronograph of Le Boulengé (1864) (Ref 10, p66 & Ref 42, p263). This latter apparatus will be described here on acct of its frequent use in the past. It was

listed, but not described, in Vol 2 of this Encyclopedia under Boulengé (Le) Chronograph, pB258-L
3) *Mechanical-Optical.* This included: a)Neesen's Chronograph (ca 1900) (Ref 10, p315 & Ref 42, p1) b)Kampé de Fériet's (1920) (Ref 6; Ref 10, p338 & Ref 42, p1) c)Rumpff's(1923) (Ref 10, p340 & Ref 42, p2) d)Duda's (ca 1923) (Ref 10, p329 & Ref 42, p2) and e)Paul's (ca 1925) (Ref 7; Ref 10, p335 & Ref 42, p2) (See also Refs 8,14,15,23, 27,33,35&36)
4) *Electrical.* This included: a)Drum Chronographs of Siemens (1845) and of Wetzer (1950) (Called Bandchronographen in Ger) (Ref 42, p3) b)Oscillographs of Blondel (1896) (Ref 10, p83 & Ref 42, p3), of Rumpff (1927) (Ref 42, pp3-4) and of Thilo (1935) (Ref 42, pp3-4) c)Cathode-Ray Oscillographs of Rogowski (1930), of Bray & Siekmann (1952) and of Rumpff (1954) (Ref 42, pp3&5-6) d)Kerr-Cell Chronographs of Schmidt (1935) and of Tawil (1952) (Ref 42, pp3&6-8) (See also under CAMERAS, HIGH-SPEED PHOTOGRAPHIC in Vol 2, pC15) e)Current-Impulse Chronographs (Stromimpuls Chronographen in Ger) of Pouillet (1845) and of Roth-Rumpff (1935) (Ref 42, pp3&8) f)Condenser (Charge) Chronographs (Ladungschronographen in Ger) of Radakovitch (1900), of Jones (1930), of Rumpff (1934) and of Weibel (1950) g)Frequency Chronographs (Frequenz-Chronographen in Ger) of Eccles-Jordan (1919), Eccles (1940) Electronic Counter Chronographs (Zählgeräte or Zähler in Ger) (since 1944 in US & 1954 in Europe (Ref 42, pp3&9-11) (See also Ref 34) h)Radar Chronographs of Barlow (1949) and of Koch (1952) (Ref 42, pp3& 11-13) Other electrical chronographs are described in Refs 5,21,32,35,37,40,41 & 43 (See also under CAMERAS, HIGH SPEED PHOTOGRAPHIC in Vol 2)

Chronographs and Detectors Currently Used for Velocity Measurements of Projectiles. The measurement of proj velocity is based on timing the flight of a proj over a precisely measured distance, known as a *base line.* The devices used to mark the beginning and end of the base line are called *detectors.* They are usually screens which on passage of projs thru them (or in their vicinity) operate *chronographs,* which serve to record these passages on a time scale. The distances betw two (or more) screens, and also betw the muzzle of the weapon and the screen nearest to it are measured (in US to the nearest 0.01ft) by means of a steel tape. This gives info necessary for detg the *average velocity* and also for calcg the *muzzle velocity*, either by formulas, such as given in Ref 30a, pp52-6 or by using special tables or charts (See also Refs 44,47,48,&49)
Following is a brief description of screens:
a. *Boulengé (Le) Screen.* A flat frame on which is strung a continuous wire making vertical passes separated by small intervals. The ends of the wire are connected to, and are part of, the chronographic circuit. Passage of the proj thru separated screens breaks the wire and interrupts an electrical circuit in each case. This and the Aberdeen Screen (See below) are *contact* screens, because they function only on contact with proj (Ref 9, p113; Ref 28, p68; Ref 49, p3) (See also Refs 44 or 47)
b. *Aberdeen Screen.* A flat frame on which are mounted two thin sheets of electrically conducting material (such as tin foil) separated by sheets of dielectric material (such as paraffined paper) or by an air gap. Each foil is connected to one of the opposite sides of the recording circuit of a timing device (usually the Aberdeen Chronograph). Then the screens are placed at desired distances from the gun in such a manner that the frames are perpendicular to the line of proj's flight. As the proj strikes the 1st screen, contact from one foil to another is completed thru the proj body and the circuit of chronograph is closed. The same happens when the proj strikes the 2nd screen, etc. Usually two chronographs, connected in parallel, are used in order to check the results (Ref 9, p116; Ref 30a, p52; Ref 49, p3) (See also Refs 44 or 47)
c. *Solenoid Coil.* It consists of an octagonal or round wooden frame with a number of turns of wire wound around the outer periphery. The opening of the frame should be large enough to permit free passage of the proj thru the center. At least two coils are used, located at known distances from the weapon. Effective use of solenoids requires that the

proj be made of a magnetic material and that it be magnetized prior to firing. The passage of a magnetized projectile thru the coil induces an elec impulse and this is transmitted to a chronograph, such as Camera Chronograph, also known as Recording Oscillograph (Ref 9, p117 & Ref 49, p1) (See also Refs 44 or 47). The solenoid coils used for detn of vel of AA projs are briefly described in Ref 9, pp120-21 & Ref 28, p89. Methods of magnetization of projs are briefly described in Ref 49, p10.

d. *Photoelectric Screen.* It is a combined optical and electronic instrument, the function of which is to detect the passage of a proj thru a limited field of view. To accomplish this, the device utilizes a photocell (phototube). At least two screens are placed on the ground at predetermined distances from the weapon. Each screen is so oriented that its optical axis intersects the trajectory of proj at a right angle. The passage of the proj over each screen, between the light source and the photocell, casts a shadow on the cell, thus producing a change in current. The change in current is transformed into a pulse which is fed into a chronograph, such as a Counter Chronograph. When the proj intersects the narrow field of the 1st screen, the chronograph beginsto measure the time and measuring is discontinued when the proj passes over the 2nd screen. This gives the time interval (t) and, if the distance betw screens is (D), the average velocity of proj is D/t.

There are several types of photo-electric screens of which the following are the more important:

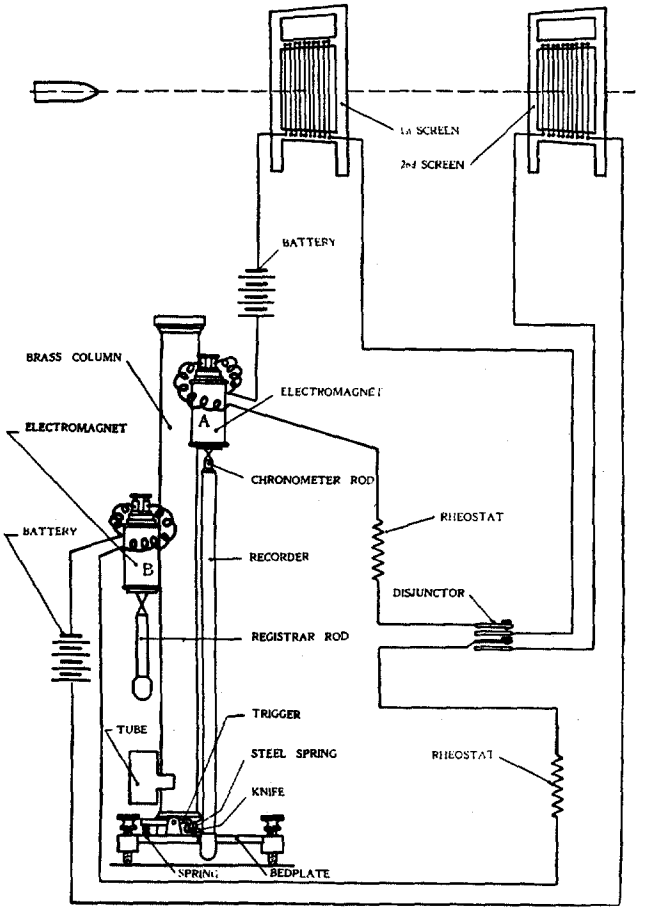
1) *Sky Screen or Field Sky Screen.* A photoelectric screen which utilizes natural light (sky light) for activating the photocell and is intended for outdoor firings. It cannot be used for night firings, or during rain or snowfall, or when the line of sight of the screen falls on or near the sun (Ref 48, p3 & Ref 49, pp2-3) (See also Refs 44 or 47)

2) *Lumiline Screen.* A photoelectric screen which utilizes artificial light for activating the photocell and is designed, primarily, for indoor or night outdoor firings of small arms. It can also be used for daylight firings, but for this the photocell should be shielded from natural light (Ref 30a, p50 & Ref 49, p3) (See also Refs 44 or 47)

A photoelectric screen developed in Canada, and known as *Ottawa Lumiline Screen,* is briefly described in Ref 30a, p51

Following is the list of principal chronographs used in conjunction with the above screens:

A. *Boulengé (Le) Chronograph.* This instrument, listed (but not described) in Vol 2, pB258-L, was developed in France in 1866. Because of its simplicity and low cost, it is still used, especially in testing small arms. It is fairly accurate when short intervals of time are involved. A schematic presentation of apparatus and equipment such as has been used in some US Ordnance installations is given in Fig



Electrical Connections of Le Boulengé Chronograph

The apparatus consists of a vertical brass column to which are clamped two electromagnets, one (A), located higher than the other (B), but on opposite sides of the column.

The magnet A is electrically connected to the 1st Le Boulengé Screen (See item a), while the magnet B is connected to the 2nd screen. When the circuits are closed (before the test), the magnet A would hold a vertically suspended long iron rod (called "recorder"), which is covered with a thin Cu or Zn tube. At the same time, the magnet B would hold a short iron rod, known as "registrar". Passage of proj thru the 1st screen causes breaks in the wires, thus causing the interruption of current in the magnet A and fall of the "recorder" as a free body. Passage of proj thru the 2nd screen breaks the circuit of magnet B, thus causing the "registrar" to fall and to strike a special device which would push a horizontal knife against the "recorder" which is in the state of falling. The indention produced by the knife would indicate the height of the fall. For locating the "zero" point, the knife is made to strike the recorder, while the rods remain stationary

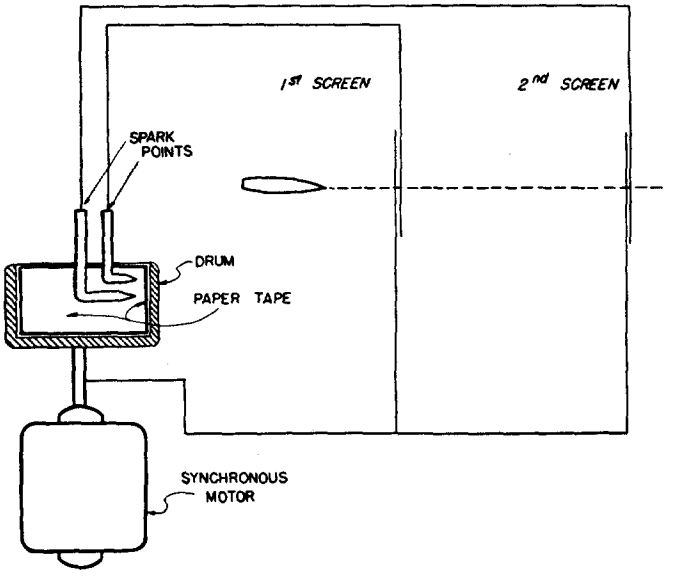
If the length of fall is h, and g is acceleration due to gravity, the time of fall, t, may be approx calcd from the formula: $t=\sqrt{2h/g}$; and an average velocity, V, of proj, from the formula D/t, where D is the distance betw two screens

For more precise methods of calcn see Ref 28 p87 & Ref 30a, pp19-20 (Refs 3,5,9,12,27,28, 29,30,36,43,44&47)

As long as h in the above calcn is small, a slight experimental error does not affect t to any great extent, but when h becomes comparatively large, experimental error affecting t cannot be disregarded. To overcome this difficulty, the instrument known as *Clepsydra* or *Klepsydra* (water clock in Greek) was developed by Le Boulengé. Clepsydra consists of a T-shaped vessel, provided with a needle valve (located at the foot of the T) and filled with mercury. The valve is actuated electrically - breaking of wires of the 1st screen opens it, while breaking of the 2nd screen closes it. By weighing the Hg which ran out thru the open valve into a tared vessel located underneath, and comparing the wt with that obtained in calibration of device, it is possible to calc, fairly accurately, the time t (Ref 5, p204; Ref 10, p124 & Fig 37 in Ref 10)

An instrument invented in England ca 1866 by *Bashforth,* was probably more accurate than the Boulengé. For its description, see Ref 5, p204 & Ref 10, pp95-6

B. *Aberdeen Chronograph.* This instrument developed during 1918-19 at Aberdeen Proving Ground, Md by Maj A. L. Loomis & Dr P. E. Klopsteg is schematically presented in Fig

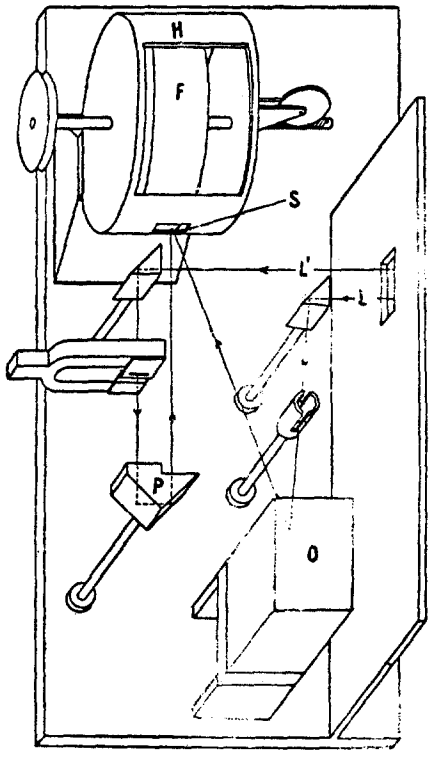


Schematic Diagram of Aberdeen Chronograph

Essentially it consists of a drum rotated at const speed by a synchronous motor. A recording strip of a paraffin-coated paper, equal in length to the inner circumference of the drum, is held against it, during rotation, by centrifugal force. Mounted in the vicinity of the drum and in circuit with it and with two (or more) Aberdeen Screens (See item b, above) are needle points. As a proj passes successively thru the screens, sparks jump from corresponding needle points to the drums, thus puncturing the recording strip of paper. By measuring the distance betw the perforations (D, in mm), and knowing the rate of rotation of the drum (R, in rpm) and its inner circumference (C, in mm), it is possible to calc the time of flight (T, in secs), from the formula: T=D/(RxC). The velocity of proj (V, in ft/sec) is calcd from the formula S/T, where S is the distance betw two screens, in ft (Refs 5a & 9; Ref 10, p95; Ref 28, p88 & Ref 30a, p43) (See also Refs 38,44 or 47)

C. *Camera Chronograph,* formerly called *Solenoid Chronograph* because it was used with the solenoid coil (See item c, above).

The original apparatus, formerly used at Aberdeen PG, was developed betw 1919 & 1922 by Dr. E. A. Eckhardt & collabs (Ref 9, p118 & Ref 28, p90). A schematic arrangement of app is shown in Fig



CAMERA CHRONOGRAPH

The app consisted of a camera H contg a photographic film F, mounted on a drum which rotates and is translated axially at the same time. Rays of light L from an arc lamp are deflected by the 1st prism and pass thru a slit, to exclude the outer rays, to the mirror of the galvanometer O. Here they are reflected and pass thru the shutter S to the film F. Rays L^1 from the same light pass to a 2nd prism where they are deflected thru slits in two plates attached to the prongs of the tuning fork; the two slits are in coincidence, permitting passage of the light. If a 500-cycle/sec fork is used, the time interval betw the lines photographed on the film will be 0.001 sec. When there is no current to deflect mirror, the film shows a straight band, but the current generated in each solenoid coil (See item c) by the passage of the projectile deflects the mirror thus causing a break in the band. As the distance betw solenoid coils and the time required for passage of a proj betw them are known, the velocity is calcd by dividing the distance by time. An accuracy of ca 99.98% could be obtained with this type of app (Ref 28, pp90-1)

It should be noted that techniques employing purely mechanical, optical-mechanical, or electro-mechanical chronographs, although fairly accurate in some cases, are considered now of inadequate accuracy for measuring very high speeds, when extremely short intervals of time are involved. The instruments for this purpose are purely electronic in nature because they eliminate the need for recording system components which inevitably possess inertia. Such instruments can also be used for detg deton velocities of expls. Oscillographs using electronic devices, such as cathode-ray tubes are briefly discussed in this section under item 1 (See also Ref 42, p5 and Vol 2, under CAMERAS, etc. ppC13-R, C15-R and C17-L)

Following are brief descriptions of electronic instruments for detg velocities of projectiles:

D. *Counter Chronograph.* An apparatus using a high-frequency oscillator of known characteristics (such as producing a frequency of 100000 cycles per sec) and a neon light panel for counting and recording the number of cycles during the interval it takes a proj to pass betw two (or more) detecting devices (such as photoelectric screens, briefly described as item d). The number of oscillations occurring during the flight of proj from the 1st to the terminal screen is made to appear upon the neon light panel and this gives the time of flight, in either microseconds or hundredths of milliseconds, depending upon the frequency of the oscillator with which the chronograph is equipped (Ref 30a, p28); Refs 43,44 or 47 & Ref 49, pp4-5)

A counter chronograph may be considered an "electronic stop watch". Its principal advantage is the speed with which the velocity can be reported; and the disadvantages (as compared to the camera chronograph), are a lack of automatic permanent record and the difficulty in operating over noisy lines (Ref 49, p5). Some of the newest types minimize these disadvantages.

One of the models used by US Ordnance Corps

for testing small arms is described in Ref 30a p28. A model used by US OrdnCorps in conjunction with Sky Screen is described in detail (including instructions for its operation) in Ref 48, pp5ff. It measures elapsed time for durations of 0.00001 to 0.09999 sec. A model, called Counter Chronograph with Shaping Adapter" is briefly described in Ref 49, pp4-5

One of the most popular instruments used in the US is the *Potter Counter Chronographs*, such as *Model 460*. This and other models are manufd by the Potter Instrument Co, Inc, Plainview, LI, NY (See Ref 30a, pp35-9; Ref 34 and pamphlets which can be obtained from the firm). An apparatus developed during WWII by the National Research Council of Canada under the name of *Ottawa Chronograph* was also used in US. Its description is given in Ref 30a, pp28-34

In addn to Potter Co, the following US firms are engaged in manuf of counter chronographs: a) Berkeley Division of Beckman Instruments, Inc, Richmond, Calif b) Hewlett-Packard Co, Palo Alto, Calif c) Computer Measurements Co, San Fernando, Calif d) The Detectron Corp, North Hollywood, Calif and e) Lavoie Laboratories, Inc, Morganville, NJ There are probably other firms, but we have no info on their products

E. *Machine Gun Chronograph,* also called *Printing or Recording Chronograph.* It operates in the same manner as counter chronograph, except that it can record (on electrosensitive papers) time intervals of machine gun and automatic cannon fire. It can reset itself, and can be ready for the next round at rates up to 9000 rounds per minute. In addn to recording velocities of each round fired, the instrument records rate of fire. (Refs 44 or 47 and Ref 49, p6)

Accdg to Ref 49, p6, in all velocity work with cannon, conducted now for US Ordn Corps either the camera chronograph or counter chronograph is taken as the standard. When both instruments are employed, the velocity reported is the mean of the two values

F. *Field Chronograph.* This includes any self-contained detection and time measuring device designed for field use. One of such instruments, the *Remington Chronoscope,* is completely portable. Its principles of operation consist of charging a condenser at constant current, during the interval of time to be determined, and measuring the resulting voltage on the condenser in terms of time. A more detailed description of app is given in Ref 30a, pp39-43. Another type of field chronograph uses a wave of known frequency, which is transmitted along the trajectory from a point in back of a weapon. The wave reflected from the moving proj causes a Doppler-Effect and the resulting electronic phenomena are transmitted to an oscilloscope, giving data from which the proj velocity can be computed (Refs 44 or 47). A field chronograph of Rumpff is described in Ref 20 (See Ref 43 Doppler-Effect Chronograph)

G. *Rotating Disc Chronograph.* This apparatus was developed at Picatinny Arsenal to be used for detn of duration of flames from priming compns. A photograph of apparatus, its detailed description, as well as description of testing method are given in Ref 24a. The apparatus consists of electric motor (capable of being set to rotate betw 3000 and 18000rpm), rotating disc and firing mechanism. Duration of flames for standard priming compns was found to be betw $4x10^{-4}$ and $56x10^{-4}$ seconds

Chronographs and Other Devices Used for Measuring Detonation Velocities of Explosives

Historical. Accdg to Taylor (Ref 36a, pp24-9), the earliest work on measuring deton velocities was conducted in 1880's by M. Berthelot, P. Vieille, E. Mallard & H. Le Chatelier. One of the simplest methods (which is still used to a considerable degree) was devised by H. Dautriche [CR **143**, 641 (1906)] but previous to this, H. Mettegang [Intern-Kongr für angew Chemie, Berlin **2**, 322 (1903)] described a spark chronograph, which gradually became a standard equipment of explosives laboratories competing with Dautriche method. Mettegang app is, accdg to Marshall (Ref 4, p477), based on the idea of Noble's Chronoscope devised in 1871 for measuring velocities of projectiles.

As the Dautriche and Mettegang methods are not sufficiently accurate for measuring vels involving short time intervals (for example the Mettegang app is accurate to only 10μ

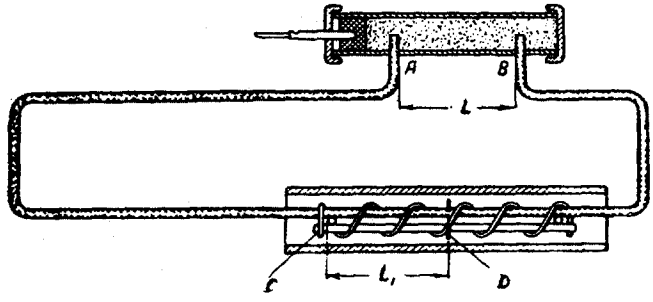
sec or more), attempts have been made to apply the "condenser discharge" method first described by R. Sabine in PhilMag [5] **1**, 337 (1876), to such measurements. This principle was first used by M. Radakovic [BerAkadWissWien **109**, 941 (1900)] to measure vels of projs, but later investigators extended it to measuring deton velocities. The instruments designed up to about 1946 were not sufficiently accurate due partly to the generation of electrostatic charges in the expln and partly to the leakage of condenser chge thru the ionized expln products. This difficulty has been overcome by the use of trigger devices which allow the integrating circuit to be isolated from extraneous electrical influences. Electronic triggering methods are particularly advantageous since functioning delays are reduced almost to nil. The first successful "electronic condenser chronoscope" was described in 1946 by Niesewanger & Brown (Ref 32). The app was capable of measuring time intervals of 10^{-5} sec with an accuracy of ±5%. A portable instrument of Brimley (Ref 34a), called "Micro-Timer", especially designed for field use was claimed to measure time intervals as low as 10^{-7} sec. Among other electronic devices for measuring short time intervals, one of the most notable was the "Crystal-Controlled Chronoscope", such as designed by Fitch (Ref 33b). This instrument, accdg to Taylor (Ref 36a, p28), is equal or even better in precision than condenser chronoscopes (See also an electronic device known as "Counter Chronograph", described here as item D)

The first photographic instrument for detg deton vels was, accdg to Taylor (Ref 36a, pp28-9), the "Rotating Drum Camera", devised in 1881 by M. Berthelot & P. Vieille. They used the camera first in the study of deton of expl gases and then, in 1885, applied it to deton vels of condensed expls, as described in AnnChimPhys **6**, 556 (1885). The photographic methods possess the advantage of giving a continuous record of the deton process, but their limitation lies in their being able to view only the luminescence of the deton at the charge boundaries. As these can, under certain conditions be distorted (such as by jetting, for example), the film assessment might produce erroneous deton rate values. Furthermore, confinement of the chge in metal or other opaque materials, precludes viewing of the deton optically, except possibly by high-speed X-ray techniques, such as item JJ described under CAMERAS, etc. in Vol 2 of Encyclopedia, pC17

Chronographic Methods Used in US Before, During and After World War II. Most labs where deton vels of expls were measured, used before WWII either Dautriche Method (described here as item H) or Mettegang Chronograph (described here as item I). As both of these methods proved to be not very accurate for measuring short time intervals, other methods, such as high-speed photographic techniques and electronic chronographs, began to replace them. The more important among photographic techniques are methods using Drum Cameras, or Smear & Streak Cameras (See ppC14 &C16 in Vol 2 of Encyclopedia), and among electronic devices the Counter Chronograph, described here as item D

As all of the above methods have some limitations, other techniques, such as "Pin Chronograph" and "Micro-wave Technique" were developed during and after WWII (See items J & K)

H. *Dautriche Method.* In this test, (schematically represented in Fig), the detonation velocity of an explosive is measured by comparison with a known "standard".

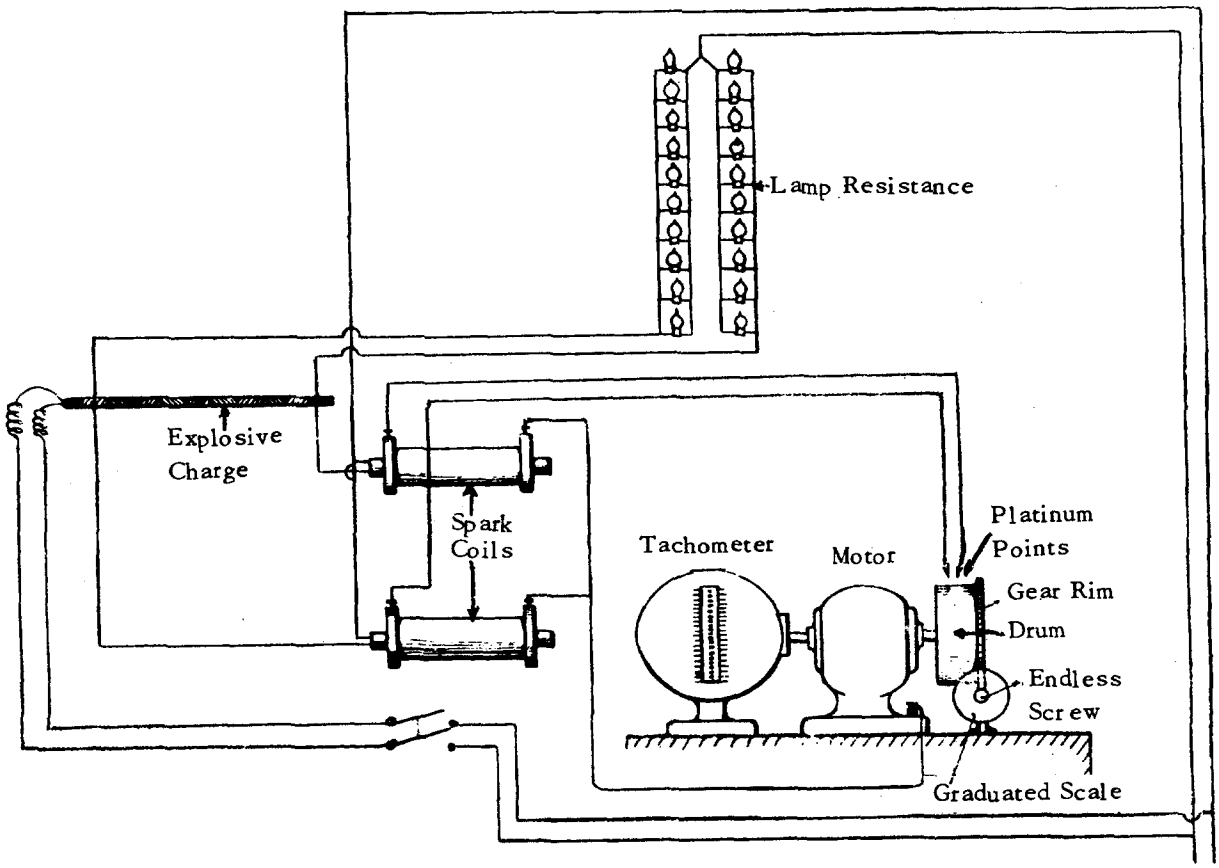


DAUTRICHE METHOD

A length of cordeau (detonating fuse) of previously accurate detd velocity (such as 7200m/sec for PETN fuse), using the Mettegang or other type of chronograph, serves as a standard. Although the Dautriche test,

itself, does not use any chronographs, it is described here for the sake of comparison with other methods for detg deton vels. This method is very simple, does not require expensive equipment and is fairly accurate. With the exception of US, it is still widely used in many countries

Procedure. The explosive to test is packed very uniformly into a cartridge (metallic, plastic or cardboard tube 20-30mm ID) closed at one end and provided on the side with two openings A & B (located at a known distance apart, such as L=15-30 cm) in which the two ends of the cordeau (of known length, such as 2 meters) are inserted. At the open end of the cartridge a detonator is inserted into the expl charge and this end is closed with a cap provided with a perforation thru which a length of safety fuse (or elec wires) is inserted. The middle section of cordeau is attached (by means of a string) to a narrow lead plate, ca 3mm thick, in such a manner that the mid-point of cordeau is exactly against the reference mark, C, on the plate and the remaining length of the plate is in close contact with the section of cordeau leading to point B. The plate and the cordeau attached to it may be placed inside an iron tube for protection of operator. When the expl is fired and the detonation wave reaches point A, a secondary deton wave is initiated and this will travel thru the cordeau towards the point C. Meanwhile the wave in the cartridge continues to move towards B and, on reaching it, another secondary wave is initiated. This wave will travel from B thru cordeau in the direction opposite to the wave travelling from A and when they meet a sharp impression will be produced on the lead plate at the point of their meeting, D. Since the waves at the cordeau are approaching at equal velocities, the time for the wave in the cartridge to pass the distance L is equal to the time for the wave in the cordeau to pass twice the distance L_1. If deton vel of cordeau is v and of expl to test is V, then $2VL_1=vL$, which gives



METTEGANG APPARATUS

$V=vL/2L_1$. (Refs 4, p479; 22, p57; 27, p183 29, p68; 30, p98; 36a, p25 & 41a, p75)

I. *Mettegang Chronograph or Mettegang Recorder.* A schematic view of equipment is given in Fig on p C312

The model used for many years in the lab of the US Bur Mines at Bruceton, Pa is described in Ref 17. The primary components of this apparatus are: a soot-covered drum, 50cm in circumference, connected to a 220v, DC elec motor provided with a rheostat for controlling its speed; a vibration tachometer for measuring rpm of the drum (which are usually 120rpm); induction coils, the primaries of which receive their current from 220v, AC, service line, while secondaries are in circuit with Pt terminals placed ca 0.25 mm from surface of the drum; and a microscope with spider line for observing the surface of the drum

Procedure: The explosive to test, usually in paper cartridges, is placed in iron tubes 42" long and of diam sl larger than diam of expl charges. Previously the tube was perforated transversely and centrally thru its sides by two small holes, the 1st being 2" from the end of the tube in which the elec detonator is inserted in the expl chge, while the 2nd hole is exactly 1 meter from the 1st hole, and then each hole is covered with a piece of tape. After inserting the chge of expl, two bare Cu wires (B&S, gage No 35, each 4" long) are run thru the holes and pulled taut. A No 6 elec detonator is imbedded centrally in the column of expl near the 1st hole and the charged tube is suspended on two wires in a bombproof firing chamber. The Cu wires protruding from the tube's holes are connected to wires leading to primary circuits of induction coils of the Mettegang app which is located in another bldg at a safe distance from the expl chge. After this, just before firing, the legs of elec detonator are connected to the magneto blasting unit and the chge is fired. The resulting expl wave hits the 1st Cu wire and ruptures it, thus interrupting the primary elec current in the 1st induction coil. This would induce the current in corresponding secondary circuit (one wire of which is in permanent contact with the drum, while the other terminates in a Pt point facing the drum) and cause a spark to jump towards the drum, thus producing a bright spot on the sooty surface. A 2nd spot would be produced on rupturing the 2nd Cu wire. Then the distance betw two spots is accurately measured (by means of microscope) and the number of revolutions of the drum to travel betw two spots, is read on a tachometer dial. If the circumference of the drum is known, the time (t) required for the expl wave to travel betw two spots, can easily be calcd and the deton velocity would be equal to l/t, where l is the length of expl chge (Refs 2a,4,17,22,29,30 & 31)

J. *Pin Chronograph (or Pin Machine) Technique.* This technique first came into use ca 1944 at Los Alamos Scientific Laboratory, New Mexico (Refs 40a & 42c). Since 1944 several successful variations have been developed as described in Refs 39a, 39b & 40d

The pin machine technique makes use of electronic circuitry which may conveniently be subdivided into the following components:

a) *Ionization-Operated or Shock-Operated Switches, called Pins.* They are distributed along the explosive charge, which is in the form of a "stick", at known exact distances

b) *Oscillograph or Cathode-Ray Chronograph.* It is an apparatus which records signals photographically on a calibrated time base of a cathode -tube. If these signals are not recorded but appear temporarily as a visible wave form on a fluorescent screen, the device is known as an *oscilloscope*

Note: The term "signal" may be defined, when used in electronic communications or impulses, as an independent variable or quantity detectable, such as a voltage, current or magnetic field having a wave form or coding which represents the intelligence transmitted. The motion on the cathode -ray screen resulting from one such variable is called the *sweep.* The process of examining (tracing) the pattern obtained on the screen in response to all variables, is known as *scanning*

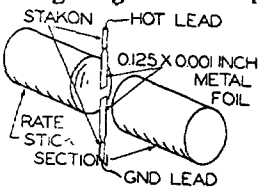
c) *Signal Mixer Circuit.* This component of pin chronograph is a network of capacitors and resistors which sends to the oscillograph (or oscilloscope), the electrical pulses which mark the passage of the detonation wave thru the explosive charge

In measuring detonation velocities of explosives, a variety of techniques for time base presentations on the cathode-ray screen have been developed. These include single sweep (and its multichannel modification), raster sweep, zigzag sweep and spiral sweep

The modifications of pin chronograph techniques include also variations in types of switches. For example, in the technique developed by Camp-

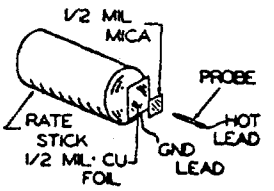
bell et al (Ref 40a), for testing cast or highly pressed expls, two types of pin switches are used: ionization and mechanical. The first type depends on ionization for closure, while in the second type (used only when detonation wave proceeds without sufficient ionization to operate the 1st type switch), the closure is effected by the motion of a thin metal foil which is accelerated by the pressure of the deton wave.

In the ionization pin switch, two extremely thin strips of foil (usually of silver), are placed between the increments of the explosive to test with the foil ends being separated by a gap, many times that of the largest grains of explosive (See fig)



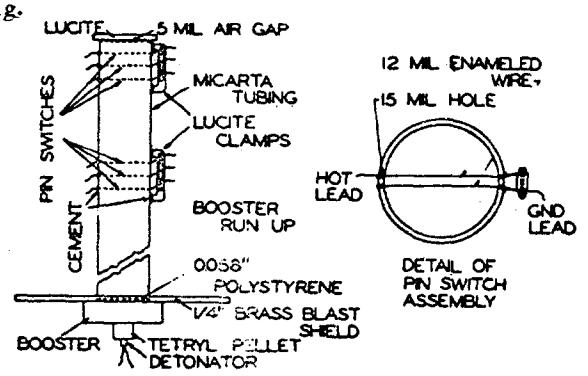
IONIZATION PIN SWITCH

In the mechanical switch (See Fig) a thin metal foil in the shape of a square is placed against the "rate stick" and this is followed by a thin piece of insulating material (such as mica, Mylar or Nylon)



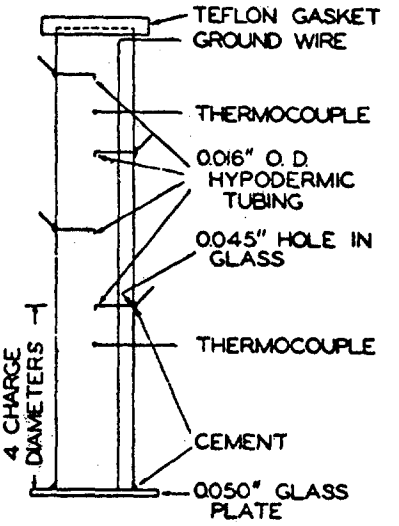
MECHANICAL PIN SWITCH

In the ionization switch method (which is most commonly used), the "rate sticks" are assembled (with the foils betw them) either by means of taping or clamping. For granular expls at low loading densities the arrangement used is shown in Fig.



PIN SWITCH FOR GRANULAR EXPLOSIVES

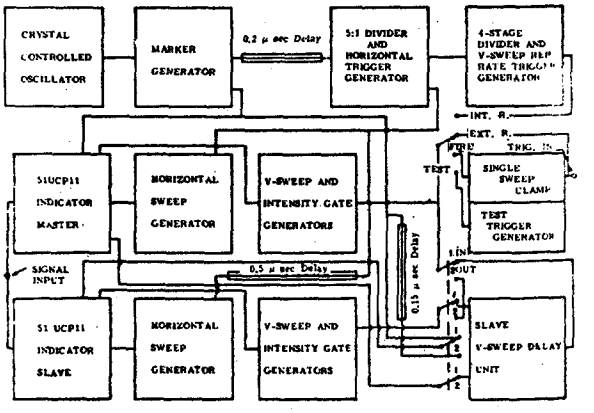
Each switch is composed of a pair of fine Cu wires stretched tightly by means of a clamp on the outside of containing tube. The spacing betw wires is regulated to a few multiples of the maximum grain size. In order to avoid premature operation of the switch by means of photo-ionization, enameled wires are used. In case of liquid expls, the arrangement represented in Fig



PIN SWITCH FOR LIQUID EXPLOSIVES

is used. By means of the common ground, the detonation wave is maintained at ground potential. The discharge of the pin mixer capacitors takes place via the spaced probes and the detonation wave to ground (Ref 40a, pp22,23 &27) (See also Ref 40d)

Block diagram of raster-generating circuitry of pin machine described by Campbell et al is given in Fig

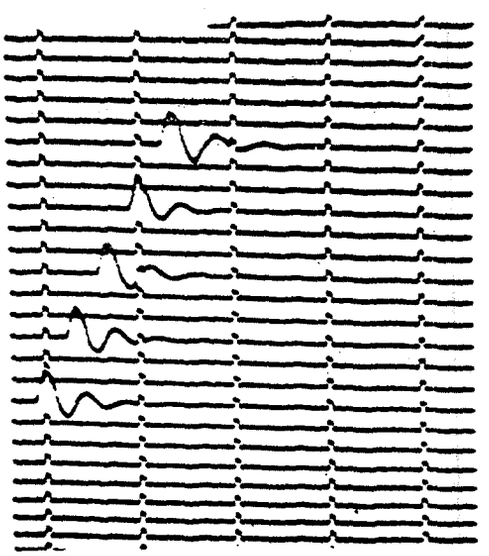


BLOCK DIAGRAM OF RASTER-GENERATING CIRCUITRY OF PIN MACHINE

Procedure. The expl chge (stick) is initiated (by means of a detonator and booster) thus causing a detonation. As the ionized shock front of deton wave travels thru the chge and hits the switches (pins or foils), they are shorted thus allowing the capacitors to be discharged thru the switches. The pulses produced by this action proceed thru mixer circuits to an oscillograph where they are displayed on a cathode-ray oscilloscope screen along with time calibration markers. This display is photographed and detonation rates are computed from expl increment lengths, and measurements of time displacements are read on a film reader

For obtaining time resolutions of 0.1% or better, it is necessary to produce sweeps (or traces) of considerably greater lengths than those produced by *single sweep* (single straight line) on a typical 5" cathode-ray oscilloscope screen (see also *multichannel sweep* described by Gibson in Ref 39a). Therefore, Campbell et al (Refs 40a & 40d) used the *raster type oscillograph*, while other investigators used "folding type" sweeps, such as zigzag and spiral sweeps

The raster type apparatus produces on an oscilloscope screen, a "raster type pattern", which is a set of parallel lines traced in sequence on the screen, such as represented on Fig given on p20 of Ref 40a (See also Ref 40d, p569 and Ref 45a, pp383-84)



SAMPLE CHRONOGRAPH RECORD

Pin switch signals are equally spaced at intervals of 7.33μ secs

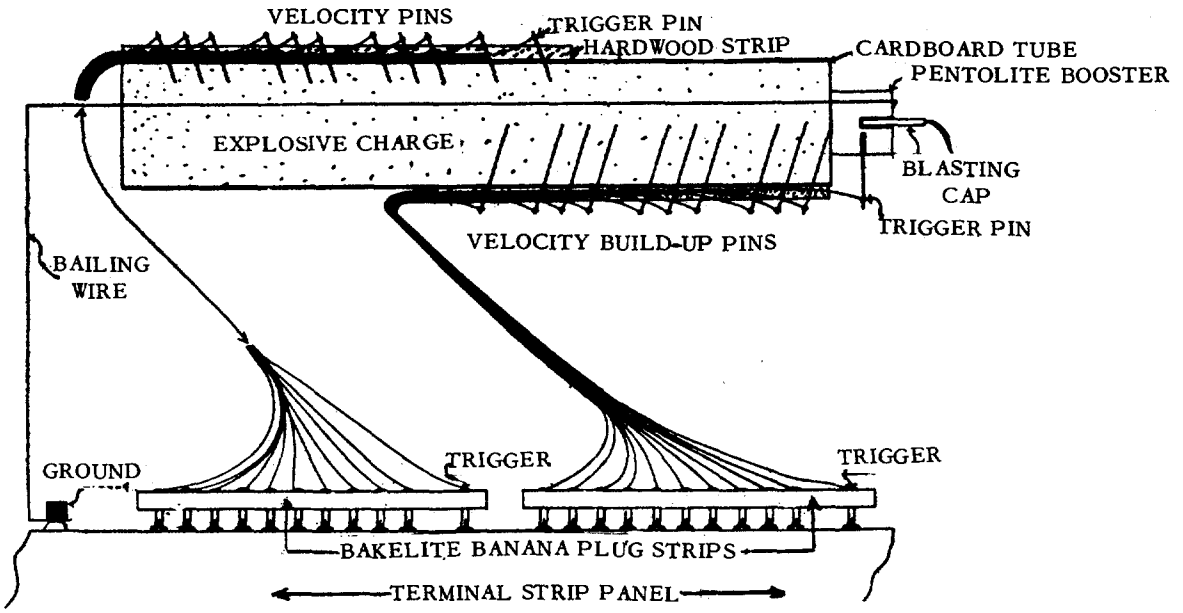
The technique using the raster type apparatus is now generally accepted in US as being preferable to other techniques, when precise measurements of detonation velocities are required. The reasons for preference are, besides precision (which permits 0.01μ sec resolution in up to 150μ sec time interval): time coverage (ample for almost any length of expl sample) and ease of maintenance (Refs 40a & 40d)

In the pinset method of Cook et al, schematically represented in Fig, the system employs a modified raster-type sweep using a triangular wave form for the horizontal deflection and single vertical sweep which gave a *zigzag type sweep*, as represented in Fig 27, p30 of Ref 42c (See also Ref 40c). Although the technique of using zigzag type oscillograph is not as accurate as the "raster-type" technique, the equipment is less expensive, because a readily available high speed oscilloscope, the "Tektronix Model 517^{A}" may easily be modified to produce a zigzag sweep (See also Refs 42a, 42c & 44a)

In the so-called *spiral sweep oscillograph*, spiral images are produced on an oscilloscope screen. This app is seldom used because it is not as accurate as other types of oscillographs

K. *Chronographic Method Employing Micro-wave Technique.* This method is more often applied to non-steady state studies where deton velocity varies considerably with time. It is not recommended when pin techniques can be employed, because it is less accurate (1-2% vs as low as 0.1% for pin technique), and greater complication in instrumentation, prepn of the expl chge, and interpretation of the records. Measurement of deton rates by microwave technique is limited to solid expls having low dielectric losses (See Refs 42b & 53)

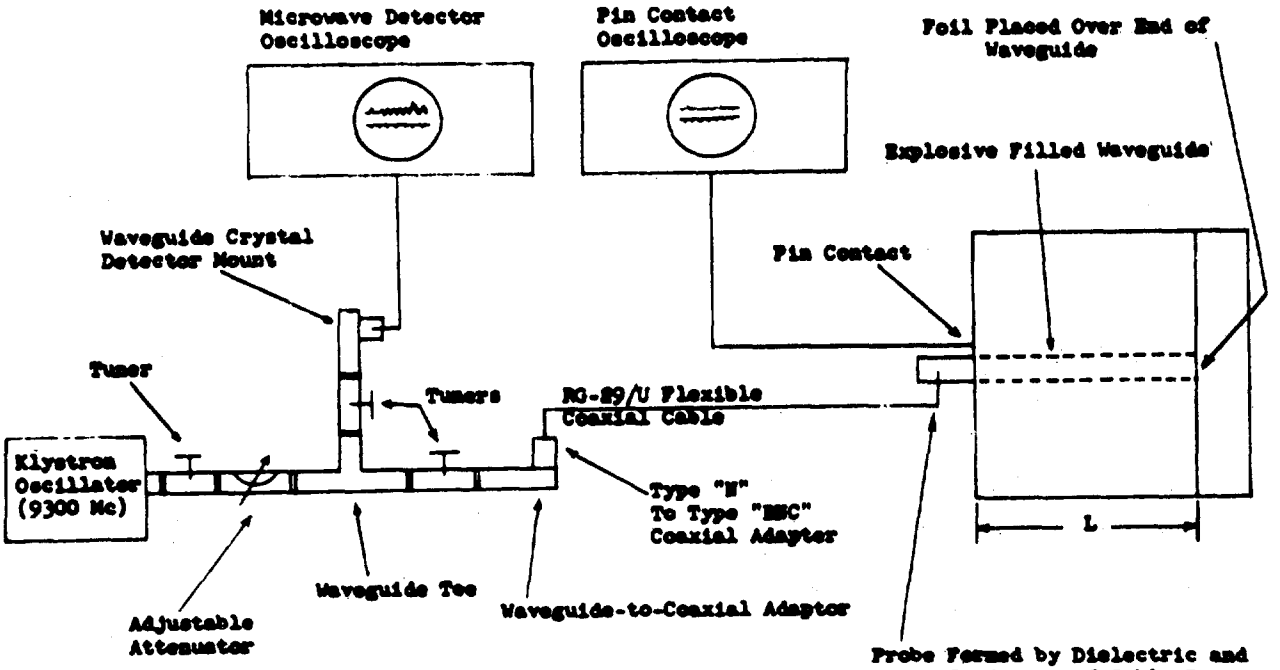
The microwave technique is, accdg to Boyd & Fagen (Ref 40b) and other investigators, based on the reflection of microwaves (in the 10^{10} cycles/sec region) from the ionized deton front in an expl-filled resonant waveguide section. As the front advances, the impedance of the resonant system goes thru approx one cycle for each one-half wavelength of the waveguide. Two oscilloscope records (each with precise time markers and a common reference pulse) are produced for each shot. One displays a pin switch signal showing the arrival of the deton front at the end of the expl being tested. The other displays, by means of a microwave detector, a trace



PINSET DIAGRAM OF COOK'S METHOD

(line) showing the times at which the waveguide becomes resonant at the oscillator frequency. Hence, one obtains the distance (in ½ wavelengths) and the time (between resonant peaks) which would permit calculation of deton rate.

A schematic arrangement of technique used by Boyd & Fagan at Los Alamos Scientific Laboratory is given in Fig



BLOCK DIAGRAM OF MICROWAVE TECHNIQUE

Some discussion on microwave technique is given by Cook (Ref 42c, pp31-2)

The section on CHRONOGRAPHS, etc was written in collaboration with E. E. Walbrecht & A. Mackenzie of Picatinny Arsenal

Refs: 1)G.D.Benton, "A Description of the Thread Velocimeter for Fire Arms", National Armory, Springfield, Mass (1873) (One of the earliest Amer instruments for detn of velocities of projectiles) 2)F.J.Zapata, "Chronograph", Naval Experimental Battery, Annapolis, Md (1875) (An electrical, rather complicated, but satisfactory instrument, used in Spanish Artillery for detn of velocity and time of flight of projs) 2a)C.E.Bichel, "New Methods of Testing Explosives", Griffin & Co. London (1905) (Mettegang Chronograph) 2b)C.E. Bichel, SS **3**, 403-05 (1908) (Mettegang Chronograph) 3)H. Kast, SS **8**, 90-1 (1913) (Siemens Spark Chronograph) 4)Marshall **2** (1917), 477-80 (Mettegang Chronograph and Dautriche Method for detn of deton velocities of expls 5)Barnett(1919) (Le Boulengé, Bashforth, Schultze-Marcel-Deprez and Mahieu Chronographs) 5a)D.A.Millar, ArOrdn **4**, 219-22 (1924) (Aberdeen Chronograph) 6)J. Kampé de Fériet, MAF **4**, 289-304 (1925) (A chronograph for detg vel of projs) 7)M. Paul, MAF **4**, 305-58 (1925) (Some new measuring devices in exterior ballistics) 8)L. Thompson & N. Riffolt, JOptSocAmer **10**, 704-05 (1925) (Optical Chronograph) 9)A.W.Ford, ArOrdn **7**, 113-24 (1926) & MAF **5**, 1293 (1926) (Muzzle-velocity measurements at Aberdeen Proving Ground by LeBoulengé Bréger Chronograph and Aberdeen Solenoid Chronograph. Also the Joly Chronograph, developed in France) 10)Cranz, **3** (1927), 35ff (Many types of chronographs employed for ballistic purposes are described) 11)E.Jones, SS **22**, 326 (1927) (A chronograph for detn of detonation velocities of short-length samples) 12)H. Kast & H.Selle, SS **23**, 217-19 (1928) (Detn of detonation velocities with a Spark Chronograph) 13)H. Rumpff, SS **24**,13-17 (1929) (Condenser Chronograph, which is a modification of Jones Chronograph, described in Ref 11) 14)H.Rumpff, SS **24**, 55-7 & 99-100 (1929) (The Optical Chronograph) 15)A.Haid et al, JahresberChem-Techn-Reichanstalt **8**, 121-24 (1930) (Measurements of deton velocity by means of an Optical Chronograph) 16)E.M.Shinkle, ArnOrdn **11**, 94 (1930) & MAF **10**, 544-5 (1931) (Chronographs used at Aberdeen PG to obtain an automatic record of the time of flight of AA projectile) 17)C.E.Monroe & J.E. Tiffany, "Physical Testing of Explosives," USBurMines Bull **346** (1931), 60-2 (Detailed description of Mettegang Chronograph) 18)W. Friederich, SS **26**, 221-22(1931) (Rotating Drum Chronograph for detg deton vels of expls) 18a)Marshall **3**, (1932) 143-46 (Detn of deton vels of expls by Condenser Sound Wave and Photographic methods) 19)T. Aoki & M.Haginara MAF **12**, 1021-36 (1933) (A new Rotating Drum Chronograph and its application to study of acceleration of rifle bullets) 20)H. Rumpff, MAF **12**, 1037-51 (1933) (Ballistic Chronograph for field measurements of muzzle vels of artillery projs) 21)J.F.Roth, SS **28**, 42-6 (1933) (Electron-Tube Chronograph for lab method of detg deton vels) 22)Stettbacher (1933), 54-7 (Mettegang Chronograph); 57-9 (Dautriche Method) 23)J.Taylor & R.Wark, MAF **12**, 1073-82 (1933) (Optical Chronograph for measuring vels of rifle bullets and lead shots of sporting guns) 24)C. Cranz et al, MAF **12**, 1083-91 (1933) (Ballistic Chronograph utilizing Kerr-Effect) 24a)J.B. Nichols, PATR **580** (1935) (Rotating disc chronograph) 25)Cranz, Ergänzungsband (1936), 163-71 (Description of several chronographs used in ballistics) 26)A.Chambionnat, MAF **15**, 1063-1131 (1936) (Photographic Chronographs for study of ballistic problems) 27)H.Selle, SS **32**, 179-83 (1937) [Detn of deton vels of expls by chronographs of Le Boulengé, Condenser, Pouillet, Spark Optical and Friederich (See Ref 18); and comparison of results with those obtained by the Dautriche Method] 27a)H.Schardin, "Beiträge zur Ballistik und Technischen Physik", Barth, Leipzig (1938), 103-27 (Reproduced in 1944 by Edwards Bros, Ann Arbor, Mich) 28)Hayes (1938) 85-92 (Le Boulengé, Aberdeen and Solenoid Chronographs used for detg vels of projs) 29)Vivas, Feigenspan & Ladreda **4** (1944), 62-70 (Le Boulengé & Mettegang Chronographs and Dautriche Method) 30)Pérez Ara (1945), 93-101 (Le Boulengé & Mettegang Chronographs and Dautriche Method) 30a)Anon, "Manual of Test Methods for Small Arms Ammunition," **ORD-M608-PM**, Frankford Arsenal, Philadelphia, Pa (1945), OPM 7-13 (Ballistic measurements by means of a chronograph) 31)G.J.Mueller, PATR **1465** (1945) [Comparison of method of detg deton vel of expls by the Queen Gray Chronograph (Mettegang type) with that of the Rotating Drum Camera designed and constructed at PicArsn, showed that the latter is the more accurate] 32)C.R.Niesewanger & F. W. Brown USBurMines **RI 3879** (1946) (Electronic,

Condenser Chronoscope for measuring deton vels of expls) 33)Stettbacher (1948), 11-12 (Optical Chronograph for detg deton vels of expls) 33a)T. Soler, Edit, "Cathode Ray Tube Displays", McGraw-Hill, NY (1948) 299 (Definition of "circular sweep" used in some chromatographic techniques) 33b)V.Fitch, RevSciInstr **20**, 942-44 (1949) (Crystal-controlled Chronoscope) 34)P.B. Sharpe, Ordn **34**, 434-36 (1950) (An electronic apparatus "Counter Chronograph" of Potter Instrument Co, Great Neck, NY, employed for measuring bullet vels) 34a)K.J.Brimley, TransSocInstrTechnology (London) **2**, 2 (1950) (Electronic Condenser Chronoscope) 34b)SACMS, "Internal Ballistics" Philosophical Library, NY (1951), 148-59 (Devices used in GtBritain for proj velocity detns) 35)R. Sartorius, MAF **25**, 387-419 (1951) (Piezo-Optical Chronograph of Tawil and its application to calibration of detonating fuses) 36)Stettbacher, Pólvoras (1952), 13-14 (Optical, Spark and Rotating Mirror Chronographs) 36a)J. Taylor, "Detonation in Condensed Explosives",Clarendon Press, Oxford (1952) 24-32 (Measurements of detonation velocity) 37)P.Fayolle & P.Naslin, MAF **26**, 767-817 (1952) and **27**, 7-75 & 294-346 (1953) (Description of various devices used in France for measuring short time intervals; included are Electromechanical and Electronic Chronographs) 38)Anon, "Fundamentals of Small Arms" TM **9-2205** (1952), 219-21 (Aberdeen and Le Boulengé Chronographs) 39)C.L. Pataky & L.A. Ouellette PATR **1932** (1953) (Conf) (A chronograph for measuring short intervals of time) (Not used as source of info) 39a)F.C.Gibson, RevSci Instr **25**, 226-31 (1954) (Multichannel single-sweep oscillographic method for detg deton vels) 39b) E.F. Pound & M.A. Cook, "Pin-Oscillograph for Measuring of Detonation Velocity", ERG Tech Rept **33**, Univ of Utah, SaltLake City (1954) (Contract No 7-onr-45107; Project No 357239) 39c)M.A.Cook et al, JApplPhys **26**, 426-28 (1955) (Measurement of deton vel by Doppler-Effect at 3-cm wavelength) 40)H.Crenn, MAF **29**, 817-29 (1955) (Electronic Chronograph) 40a)A. W.Campbell et al, "Technique for the Measurement of Detonation Velocity", pp 18-32 in the "Second ONR (Office of Naval Research) Symposium on Detonation", Washington, DC (1955) (Pin technique) 40b)T.J.Boyd,Jr & P.Fagan, "A Microwave Technique for Measuring Detonation Velocity", pp 33-38 in the "Second ONR Symposium on Detonation" (1955) 40c)E.F.Pound & M.A.Cook, "The Measurement of Detonation Velocity by Microwave Resonator Techniques", ERG TechRept **44**, Univ of Utah, Salt Lake City, (1955) (Contract No N7-orn-45107; Proj No 357239) 40d)A.W.Campbell et al, RevSciInstr **27**, 567-74 (1956) (Precision measurements of deton vels of liquid and solid expls) 41)P. Fayolle, "Chronograph for Multiple-Flash High -Speed Camera" pp190-93 in "High-Speed Photography", by R.B.Collins, Edit, Butterworths, London (1957) 41a)Gorst (1957), 75-6 (Dautriche Method) 42)H. Rumpff, Explosivst **1957**, 261-67 and **1958**, 1-13 (Chronographs for measuring short time intervals in ballistics; a review) 42a)E.G.Leger & K.Park, "A Zig-Zag Oscilloscope Presentation for Detonation Velocity Measurement in Explosives", CARDE TechMem **170/58** (1958) 42b)C.F.Cawsey et al, PrRoySoc **248A**, 499-521 (1958) (Microwave Interferometer in measuring deton vels) 42c)Cook (1958), 29-32 (Pin and Microwave Methods) 43)R.E.Kutterer, "Ballistik", Vieweg, Braunschweig (1959), 90-104 (Le Boulengé Kerr-Effect, Thilo, Spark, Condenser, Doppler-Effect and Counter Chronographs) 43a)J.F.Rider & S.D.Uslan, "Encyclopedia of Cathode-Ray Oscilloscopes and Their Uses", J.F.Rider Publisher, Inc, NY (1959), **7** - 7 & **7** - 77 (Definitions of "sawtooth sweep" and "spiral sweep" signals) 44)Glossary of Ord (1959), 70-1 (Chronographs) 44a)E.G.Leger et al, "A Zig-Zag Oscilloscope Presentation of Millimicrosecond Accuracy for Microsecond Time Intervals", CARDE TechMem 233/59 (1960) 45) K.Schriever, Explosivst **1961**, 55-60 (Modern Photochronograph) 45a)N.M.Cooke & J.Marcus, "Electronics and Nucleonics Dictionary", McGraw-Hill NY (1960), 75 (Definition of chronograph); 383-84 (Definition of term "raster") 46)Merriam-Webster's (1961) 402 (Chronograph) 47)OrdTechTerm (1962), 70-1 (Chronograph) 48)Anon, "Operation and Organization Maintenance. Field Sky Screenand Counter Chronograph", TM **9-1860-7** (1956) 49)Anon, "Ordnance Proof Manual", Vol 4, OPM **80-15** (1957), Aberdeen Proving Ground, Md (Velocity measurement of projectile) 50)Ibid, OPM **80-20** (1957) (Translation of instrumental velocity to Muzzle or striking velocity) 51)Ibid, OPM **80-25** (1957) (Time of projs flight measurements) 52)Ibid, OPM **80-80** (1957) (Photographic methods for velocity detns) 52a)J.Berger & J.Viard, "Physique des Explosifs Solides", Dunod, Paris (1962), 133-37 (Chronographe a oscillograph

cathodique); 137-38 (Chronoscope à charge de condensateur) 53)E.E.Walbrecht, "Dielectric Properties of Some Common High Explosives", PicArsnTechMemo **1170** (1963)
(See also Refs under CAMERAS, HIGH-SPEED, PHOTOGRAPHIC in Vol 2, ppC13ff)

Chronophotography or Photochronography (Chronophotographie in Ger) or *Optical -Photographic Method for Measuring Average Velocity of Projectiles.* In this method a series of instantaneous photographs of a moving projectile is taken using continuous lighting and instantaneous shutter. One of the 1st devices of this kind was built in 1873 by Janssen (photographic revolver, allowing the taking of 70 successive photos). Muybridge of US used 12 to 30 photographic cameras placed in a row, and took as many photographs in succession. Marey of France used only one camera and succeeded in making 110 photos per second. Athanasius of Greece increased this number to 140. Further improvements were made by Anshütz, Duda (Ballistograph of Duda), Paul, Reynaud (1878), Uchatius, Edison, Lumiere Lendelfeld (1903), Neuiger (1904), Clees, Lehmann (1916), Kampé de Fériet (1925), Rumpff and others

Another way for taking a series of photographs is to use discontinuous lighting such as electric sparks. Pioneers in this field were Marey & Wood (ca 1890), Bull (1904), Kranzfelder & Schwinning (1905), Bensberg (1907), Cranz (1909) Schatte (1912), Cranz & Glatzel (1912) and others. (See also under CAMERAS, HIGH-SPEED PHOTOGRAPHIC, Vol 2, ppC13ff)
Refs: 1)Cranz **3** (1927), 53 & 327-62 2)Cranz, Ergänzungsband (1936), 165 3)W.D.Chesterman, "Photographic Study of Rapid Events", Clarendon Press, Oxford, England (1951) 4)K.Schriever, Explosivst **1961**, 55-60 (Modern Photochronography

Chronopolarography. See under Chronoamperometry, etc

Chronopotentiometry. See under Chronoamperometry, etc

Chronospectrography. A method of spectral analysis in which the speed of the reaction (when a reagent is added to sample at const rate) can be followed by measuring the change in intensity of transmitted light of the proper wave length.
Ref: J.M.González Barredo, AnalesRealSocEspañ Fis y Quím **44B**, 29-40 (1948) & **45B**, 1171-82 (1949); CA **42**, 8107 (1948) & **46**, 344 (1952)

Chrysammic Acid. See 2,4,5,7 - Tetranitro - 1,8 -dihydroxyanthraquinone under Dihydroxyanthraquinone

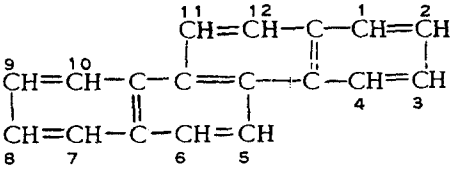
Chrysammidic Acid. See 2,4,5,7 -Tetranitro - 8 - amino 1 - hydroxy-anthraquinone under Aminohydroxyanthraquinones and Derivatives, Vol 1, pA217-L

Chrysazine. Same as 1,8 - Dihydroxyanthraquinone

Chrysazine, Tetranitro - Same as Chrysammic Acid

Chrysene and Derivatives

Chrysene or Benzophenanthrene (called Chrysen or 1,2 - Benzo-phenanthren in Ger),



(Present CA system of numbering); mw 162.26, H 11.18%; col pltlts(from benz or AcOH), giving a violet-red fluorescent color in soln, mp 250°, bp 448°, d 1.274; sol in hot benz or hot AcOH; diffc sol in cold eth, CS_2, AcOH or benz; derived from coal-tar fractions boiling above 360°; also produced by the cracking of petroleum fractions at 630-80° using catalysts, or by heating indene at 500-700° with catalysts, and by passing naphthalene & coumarone thru a red-hot tube (Refs 1 to 4). Other props & methods of prepn are given in the Refs
Refs: 1)Beil **5**, 718, (355) & [629] 2)Ullmann **3** (1929), 434 3)Thorpe **3** (1939), 118 4)Kirk & Othmer **7** (1951), 615 & 647

Mononitrochrysene, $C_{18}H_{11}NO_2$; mw 273.28, N 5.13%. Only one isomer is formed on direct nitration:
6-Nitrochrysene, orn-yel to bright yel ndls (from pyridine), mp 213.7-14.6°; sol in hot nitrobenz; sl sol in benz or AcOH; v sl sol in cold alc, eth or CS_2; was prepd by nitration of coml chrysene in glacial AcOH, using HNO_3 + H_2SO_4 at 40° (Ref 2)

See also Refs 1 & 4. Compare with Ref 3.
Refs: 1)Beil **5**, 719 2)M.S.Newman & J.A. Cathcart, JOC **5**, 620 (1940) & CA **35**, 1049 (1941) 3)M.S.Newman & A.I.Kosak, JOC **14**, 379 (1949) & CA **44**, 594 (1950) 4)K.Fukui et al, JChemPhys **22**, 1440 (1954) & CA **48**, 13404 (1954)

Dinitrochrysene, $C_{18}H_{10}N_2O_4$; mw 318.28, N8.80%. Only one isomer is known:
6,12-Dinitrochrysene [same isomer is called 2,8 -Dinitrocrysene in Ref 2], fine bright-yel ndls (from pyridine), mp 380.5-82.5°; sl sol in alc, eth or benz; v sl sol in hot AcOH; was prepd by addg fuming HNO_3 to a stirred suspension of chrysene in AcOH contg concd H_2SO_4 (Ref 3). See also Refs 1 & 2. Compare with Ref 4
Refs: 1)Beil **5**, 270 2)IGFarbenind, FrP 794534 (1936) & CA **30**, 5238 (1936) 3)M.S.Newman & J. A.Cathcart, JOC **5**, 620 (1940) & CA **35**, 1094 (1941) 4)M.S.Newman & A.I.Kosak, JOC **14**, 379 (1949) & CA **44**, 594 (1950)

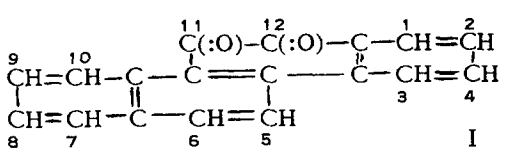
Trinitrochrysene, $C_{18}H_9N_3O_6$, not found in Beil or in CA thru 1956

Tetranitrochrysene, $C_{18}H_8N_4O_8$; mw 408.28, N13.72%. One isomer of unspecified positions of the nitro groups is known: yel ndls (from AcOH), mp above 300°, explodes violently on heating rapidly; sl sol in hot AcOH; insol in common solvs was prepd by dissolving chrysene in fuming HNO_3 (Refs 1,2,3 & 4)
Refs: 1)Beil **5**, 720 2)C.Liebermann, Ann **158**, 307 (1871) 3)E.Schmidt, JPrakt Chem **9**, 283 (1874) 4)L.Schmid & M.Zentner, Monatsh **49**, 92 (1928) & CA **22**, 3415 (1928)

Note: Higher nitrated derivs of Chrysene were not found in Beil or in CA thru 1956

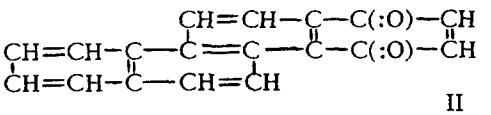
Chrysenedione and Derivatives

Chrysenedione, Chrysenequinone or Chrysoquinone (Called Chrysenchinon, Chrysochinon, Dioxo -dihydrochrysen or Benzo-phenanthrenchinon in Ger),

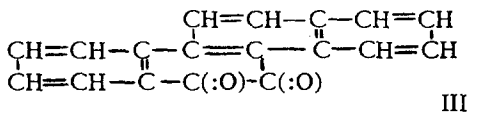


I

(Present CA system of numbering)

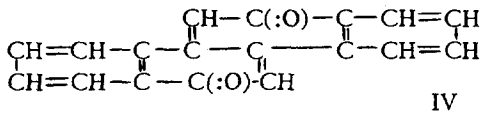


II



III

(View Formula III inverted and compare with Formula I, above)



IV

The above isomers are known and described in the literature (mw 258.26, O 12.39%):
Formula I. 11,12-Chrysenedione [called 1,2 -Benzo-phenanthrenchinon or Chrysenchinon-(1.2) in Ger], orn-red ndls (from toluene or benz), lfts or pltlts (from hot AcOH), mp 235-239.5°; mod sol in hot alc or benz; v sl sol in eth or cold CS_2. Its **Perchlorate salt**, $C_{18}H_{10}O_2{}^+HClO_4$, dk violet prisms (from tetrachloroethane), explodes ca 190°; and is decompd into its constituents by water. Other props & methods of prepn are given in Refs 1, 2, & 3
Formula II. 1,4-Chrysenedione [called 1,4-Chrys-oquinone by Bachmann & Deno(Ref 6)], glistening golden-bronze flakes (from AcOH), mp 206.5-207.5°; was prepd by reacting 1-vinylnaphthalene & p-benzoquinone in AcOH for 5 hrs at 100° (Ref 6)
Formula III. 5,6-Chrysenedione, a compd listed as such in CA Subject Index 31-40 (1937-46), p4958 and in 41-50 (1947-56), p3156s; but appears to be identical in props with that of the 11,12 -Chrysenedione described above. For example, see Refs 5 & 7
Formula IV. 6,12-Chrysenedione [called Chryso-chinon - (2.8) or amphi-Chrysenchinon in Ger], reddish-yel ndls (from AcOH), mp 288-90° (dec); v sl sol in most solvs, except AcOH & benz; was prepd by oxidn of 6,12-dihydroxychrysene with PbO_2 in hot AcOH. Its *Sodium bisulfite compd*, $C_{18}H_{10}O_2{}^+NaHSO_3{}^+2H_2O$, yel ndls, was very unstable [Ref 1, p(441) & Ref 4]
Refs: 1)Beil **7**, 827, (441) & [760] 2)K.A. Hofmann et al, Ber **43**, 2629 (1910) & CA **5**, 290 (1911) 3)E.Beschke, Ann **384**, 143 (1911) & CA **6**, 341 (1912) 4)E.Beschke & F.Diehm, Ann **384**,

174 & 182-83 (1911) & CA **6**, 342 (1912) 5)L.F. Fieser & E.M.Dietz, JACS **53**, 1131 (1931) & CA **25**, 1818 (1931) 6)W.E.Bachmann & N.C.Deno, JACS **71**, 3068 (1949) & CA **44**, 8346 (1950) 7)E. D.Bergmann et al, JACS **75**, 3232 (1953) & CA **47**, 11841 (1953)

Mononitrochrysenedione, $O:C_{18}H_9(NO_2):O$; mw 303.26, N 4.62%. Two isomers are known: *x-Nitro-11,12-chrysenedione* (called x-Nitro-5,6 chrysenequinone in the literature), red ndls (from alc), mp 252° (Refs 1,2 & 3), or yel crysts (from nitrobenz), mp 256-57° (Ref 4); was prepd by treating acetylaminochrysenedione with dil HNO_3 (Ref 3), or by chromic acid oxidn of 6-nitro-chrysene (Ref 2), or by nitration of 11,12-chrysene dione with concd HNO_3 at RT (Ref 4). See also Ref 6 for a clarification of the confusion which exists in the literature
6-Nitro-11,12-chrysenedione (called 12-Nitro-5,6 -chrysene-quinone in Refs), silky, bright red, sublimable ndls (from AcOH), mp 302-3°; was prepd by oxidn of 6-nitro-chrysene by Na dichromate in AcOH, or by similarly refluxing for 12 hrs a mixt of nitro-6-n-valeryl-aminochrysene Na dichromate & AcOH (Ref 6). Newman & Cathcart (Ref 5) also reported to have prepd this compd but could not isolate it in a pure state
Refs: 1)Beil **7**, 828 2)E.Bamberger & C.Burg-dorf, Ber **23**, 2444 (1890) & JCS **58II**, 1312 (1890) 3)R.Abegg, Ber **24**, 953 (1891) & JCS **60I**, 731 (1891) 4)K.M.P.Singh & S.Dutt, ProcIndAcadSci **8A**, 187-93 (1938) & CA **33**, 1943 (1939) 5)M.S. Newman & J.A.Cathcart, JOC **5**, 618-22 (1940) & CA **35**, 1049 (1941) 6)Ng.Ph.Buu-Hoi, JOC **19**, 1396-98 (1954) & CA **50**, 279 (1956)

Dinitrochrysenedione, $O:C_{18}H_8(NO_2)_2:O$; mw 348.26, N 8.04%; red ndls, mp 230° (Refs 1 & 2), or yel crysts (from nitrobenz), mp 235°(Ref 3); mod sol in hot alc or AcOH; sl sol in benz or eth; was prepd by refluxing chrysenedione with concd HNO_3 for 3 hrs. Its expl props were not detd
Refs: 1)Beil **7**, 828 2)A.Adler, Ber **12**, 1893 (1879) 3)K.M.P.Singh & S.Dutt, ProcIndAcadSci **8A**, 187-93 (1938) & CA **33**, 1943 (1939)

Trinitrochrysenedione, $O:C_{18}H_7(NO_2)_3:O$, not found in Beil or in CA thru 1956

Tetranitrochrysenedione, $O:C_{18}H_6(NO_2)_4:O$; mw 438.26, N 12.79%; orn crysts, mp >300°; insol in common org solvs; was prepd by refluxing chrysenedione with fuming HNO_3 for 8 hrs (Refs 1,2 & 3). Its expl props were not detd
Refs: 1)Beil **7**, 828 2)C.Liebermann, Ann **158**, 314 (1871) 3)K.M.P.Singh & S.Dutt, ProcIndAcad Sci **8A**, 187-93 (1938) & CA **33**, 1943 (1939)

Note: Higher nitro derivs of Chrysenedione were not found in Beil or in CA thru 1956

Chrysenequinone. Same as Chrysenedione

Chrysoidine (Diaminoazobenzene or 4-Phenylazo -m-phenylene diamine Hydrochloride) [called 2,4 -Diamino-azobenzol; 4-Benzolazo-phenylenediamin -(1.3); or Base des Chrysoidins in Ger], $C_6H_5.N:N.C_6H_3(NH_2)_2$; mw 212.25, N 26.40%; yel ndls (from hot w), mp 117.5°; sol in alc, eth, chlf, benz or aniline; sl sol in hot w. It forms numerous salts which are cryst comds. The *Hydrochloride* (also called Chrysoidine Y or Chrysoidine Extra), $C_{12}H_{12}N_4{\cdot}HCl$, red-brn powd or large, black shiny crysts having a green luster, is the commercial product. The *Dihydrochloride*, col crysts (from alc), dec at 222°. Other props & methods of prepn are given in Ref 1

The use of Chrysoidine in a WWI Amer airplane yel smoke-signal grenade compn: $KClO_3$ 33, lactose 24, auramine 34 & chrysoidine 9%, is listed by Davis (Ref 3). Izzo (Ref 4a) lists, anong others, the following yel color smoke compns: a)$KClO_3$ 35, lactose 25, auramine 10 & chrysoidine 30% and b)Lactose 25, chrysoidine 25 & p-nitroaniline yellow 50%. He also lists an orange smoke: $KClO_3$ 25, lactose 25, chrysoidine 45 & fossil meal 5%
Refs: 1)Beil **16**, 383, (336) & [203] 2)Colour Index, Editor, F.M.Rowe, The Society of Dyers and Colourists, Yorkshire, GtBrit (1924), p5 (System No 20) 3)Davis (1943), 123 4)Hackh's (1944), 201 4a)Izzo, Pirotecnia (1950), 235

Chrysophanic Acid. See 4,5-Dihydroxy-2-methyl -anthraquinone under Dihydroxymethylanthraquinone

Chrysoquinone. Same as Chrysenedione

Chuffing. See under Burning, Unstable (in Rocket Motors), Vol **2**, p B361-L

Chugging. See under Burning, Unstable (in Rocket Motors), Vol **2**, p B361-R

CI. Abbr for "Colour Index" (Brit), "Color Index" (Amer), which is the semiofficial description of dyes arranged by numbers assigned to chemical classes. The 1st edition (1924) is now superseded by the 2nd (Ref 1) which uses a different set of numbers from that of the 1st edition.
Refs: 1)Society of Dyers and Colourists (and American Association of Textile Chemists and Colorists), "Colour Index", Dean House, London, vols 1,2,3 & 4 (1955-59) 2)CondChemDict (1961), 277

Cibalite. A smokeless proplnt patented in 1892 by Kalliwoda von Falkenstein of Austria, which consisted of specially prepd NC and some K dichromate. For prepn of NC, the cotton was treated with dil K permanganate soln, followed by washing, drying and nitrating with nitric acid (d1.3) at temp of 70°
Ref: Daniel (1902), 143

CIA. See Vol 1, p Abbr 10-R

C.I.A. (Brit). Chief Inspector of Armaments (Issue Branch, Red Barracks, Woolwich, SE 18)

Cible (Fr). Target

CIC. Combat Information Center

CIC. Counter Intelligence Coprs

Cicene. See Cycene

Cigarette Burning. A type of burning induced in a solid grain (such as in rocket proplnts, BkPdr, gasless delay elements & pyrotechnic candles) by permitting burning on one end only, so that the burning progresses in the direction of the longitudinal axis
Refs: 1)Rocket Encycl (1959), 89 2)OrdTechTerm (1962), 71

Cigarette Fuze (U). Described in classified report TR-734, Diamond Ordnance Fuze Laboratories, by H.P.Kalmus

Cilferite or Cilpherite. A silver-grey comp, used by the French during WWI: AN 78, Al 3, ferrosilicon 14 & wood meal 5% (Ref 1). Littleton (Ref 2) gives AN 78.50, ferrosilicon 13.75, Al (fine) 2.75 & wood meal 5.00%. Its props: insensitive to rifle bullet, Fr Trauzl test 85 (PA 100) and deton velocity 4200m/sec at d 1.208
Refs: 1)M.M.Kostevich, "HE & P Burning Ground," Imp d' Art Voltaire, Paris 9 (1927), 40 2)Dr L.R.Littleton; private file, Washington, DC (1958)

Cinchonamine and Derivatives
Cinchonamine, $C_{19}H_{24}N_2O$, wh, rhmb ndls (from alc), mp 184-94°. An alkaloid which may be obtd, by extraction, from the bark of Remijia Purdieanna. Being a strong diacidic base, it forms well defined salts, such as *nitrate*, (which is practically insol in w) (Refs 1 & 2), *nitrite* and *picrate* (Ref 1)

Dinitrocinchonamine, $C_{19}H_{22}N_4O_5$, mw 386.40, N 14.50%; yel, amor pdr which is explosive; can be prepd by nitrating cinchonamine with nitric acid (Ref 2)
Refs: 1)Beil **22**, [358-9] 2)Thorpe **3**, (1939), 152

Cinchonino-tetraazido-copper, $[(N_3)_2Cu.C_{19}H_{22}N_2O.Cu(N_3)_2]$; N(azide) 28.52%, greenish-brn solid, mp-explodes mildly at 154-55°; burns in flame but is insensitive to impact; was prepd by treating a mixt of cinchonine, $C_{19}H_{22}N_2O$ [an alkaloid derived from various species of cinchona bark (Ref 1)] and Cu nitrate (both dissolved in methanol) with an aq soln of Na azide (Ref 2)
Refs: 1)Beil **23**, 424, (132) & [369] 2)A. Cirulis & M.Straumanis, JPraktChem **162**, 322-23 (1943) & CA **38**, 1970 (1944)

Cine Chromatography and Radiography. See Vol 2, pp C14, C15 & C17

Cinematography; Application in Ordnance of. See under CAMERAS, HIGH-SPEED PHOTOGRAPHIC, Vol 2, pp C13ff

Cinnabar. A native red mercuric sulfide, HgS. It was used in some pyrotechnic compns. See Davis (1943), 61 and under Sulfides

Cinnamic Acid and Derivatives.
Cinnamic Acid or β-Phenylacrylic Acid, (Zimtsäure in Ger), $C_6H_5CH{:}CH.COOH$; mw 148.15 col monocl prisms, mp 133°, bp 300°, d 1.245; can be prepd by heating benzaldehyde with Na acetate in presence of dehydrating agents, or by

other methods. It forms salts and can be nitrated and ozonized
Refs: 1)Beil **9**, 572, (224) & [377] 2)Kirk & Othmer **4** (1949), 1-4 3)CondChemDict (1961), 298

Cinnamic Acid Azide or Cinnamoyl Azide. (Formerly called Cinnamoylazoimide), C_6H_5.CH:CH.CO.N_3; mw 173.17, N 24.27%; col plates (from petr eth), mp 86° with evoln of nitrogen; easily sol in benz, et acet, acetone, chlf & cold alc; can be prepd by shaking cinnamoyl chloride with Na azide in ether. Its expl props were not detd (Refs 1,2 & 3). It was patented for use as a blowing agent in prepn of high-strength resinous foams, contg glycidyl polyethers of dihydric phenols (Ref 4)
Refs: 1)Beil **9**, 591 & [393] 2)M.O.Forster, JCS **95**, 437 (1909) 3)L.W.Jones & J.P.Mason, JACS **49**, 2531-2 (1927) 4)H.L.Parry & B.O. Blackburn, USP 2739134 (1956) & CA **50**, 9787 (1956)

Cinnamic Acid Diazonium Nitrate or Cinnamoyl-3-diazonium Nitrate (Zimtsäure-m-diazoniumnitrat in Ger), HOOC.CH:CH.C_6H_4.N(:N).O.NO_2; mw 237.17, N 17.72%; cd ndls, mp explodes violently on heating or on impact; easily sol in w; was prepd by diazotizing 3-aminocinnamic acid with K nitrite in dil HCl, followed by pouring the resulting soln into concd nitric acid
Refs: 1)Beil **16**, 551 2)S.Gabriel, Ber **15**, 2296-7 (1882)

Cinnamic Acid Ozonide or Cinnamoyl Ozonide C_6H_5.CH:CH.COOH.O_3. It was claimed to be prepd in methanolic soln by ozonization of cinnamic acid. Attempt to isolate it by adding w resulted in decompn into benzoic and glyoxalic acids.
Refs: 1)Beil-not found 2)E.Briner & A.Gelbert, Helv **22**, 1484 (1939) & CA **34**, 1640 (1940)

Cinnamic Acid Peroxide or Cinnamoyl Peroxide. See Dicinnamoyl Peroxide

Mononitrocinnamic Acid, $C_9H_7NO_4$, mw 193.15, N 7.25%. All three isomers: o-, m- and p- are known, but none of them is explosive.
Refs: 1)Beil **9**, 604-07, (245-47) & [402-04] 2)OrgSynth **5**, 83-4 (1925) 3)H.W.Underwood Jr & E.L.Kochmann, JACS **48**, 254-7 (1926) 4)P. Ruggli et al, Helv **28**, 335 (1945) 5)W.Davey & J.R.Gwilt, JCS **1950**, 206

Dinitrocinnamic Acid, $C_9H_6N_2O_6$, mw 238.15, N 11.76%. The following isomers are described in the literature: *2,4-Dinitrocinnamic Acid,* $(O_2N)_2C_6H_3$.CH:CH.COOH, lt yel ndls (from w), mp 179° (Refs 1,2,5 & 6); *2,6-Dinitrocinnamic Acid,* col ndls (from w), mp 181° (Refs 2 & 4); *4,a-Dinitrocinnamic Acid,* $O_2N.C_6H_4$.CH:C(NO_2).COOH, col lflts; unstable (Refs 1 & 3)
Refs: 1)Beil **9**, 609 2)Beil **9**, (251) 3)P. Friedländer & J.Mähly Ann **229**, 224 (1885) 4)S. Reich, Ber **45**, 808 (1912) 5)K.C.Pandya et al, JUnivBombay, **10**, Pt 3, 78-82 (1941) & CA **36**, 3794 (1942) 6)J.Rai & K.B.L.Mathur, JIndian ChemSoc **24**, 383-8 (1947) & CA **42**, 5881 (1948)

Nitrocinnamic Acid Azides or Nitrocinnamoyl Azides, $C_9H_6N_4O_3$, mw 218.17, N 25.68%. The following isomers are described in the literature: *o-Nitrocinnamoyl Azide,* $O_2N.C_6H_4$.CH:CH.CO.N_3 lt yel crysts (from benz), mp 89° (dec); was prepd by treating o-nitrocinnamoyl chloride with Na azide, previously activated by mixing with a small amt of hydrazine hydrate as described in Ref 4. Its expl props were not detd
m-Nitrocinnamoyl Azide, col rhmb crysts (from ether), mp 117-18° (dec); puffs off at higher temp; insol in cold w or alc, sol in chlf or ether; was prepd by treating m-nitrocinnamoyl hydrazide with Na nitrite + HCl in water covered with ether (Refs 1 & 2)
p-Nitrocinnamoyl Azide, lt yel crysts, mp 123°; was prepd by treating β-hydrazino-p-nitrohydrocinnamic acid (obtained on reacting p-nitrocinnamic acid with 2 mols of hydrazine) with Na nitrite, as described in Ref 3
Refs: 1)Beil **9**, [404] 2)T.Curtius & E.Kenngott, JPraktChem **107**, 100-01 (1924) & CA **18**, 1657 (1924) 3)M.Freri & A.Solza, AttiAccad LinceiClasseSciFisMatNat **29**, 691-93 (1939) & CA **34**, 4730-31 (1940) 4)P.Ruggli et al, Helv **28**, 333 (1945) & CA **40**, 867 (1946)

CIT-4. One of the rocket proplnts developed at California Institute of Technology. It contained: NC (10.92%N) 50.6, NG 37.0, DNEtB 9.0, EtCentr 1.0, C black 0.1, hydrocellulose 1.5, (K nitrate) 0.6 & MgO 0.2%. On the basis of tests conducted at PicArsn, it was concluded that CIT-4 did not have the advantageous burning character-

istics reported by CALTECH; nor did it appear to be markedly superior to other cool proplnts currently available.
Ref: V.Lindner, "Evaluation of the CIT-4 Propellant", PATR **1744** (1949)

Citraconic Acid and Derivatives
Citraconic Acid (Methylmaleic Acid), $CH_3.C(COOH):CH(COOH)$; mw 130.10, col ndls, mp 92-3°, d 1.617; can be prepd by carefully heating citric acid, or by other methods
Refs: 1)Beil **2**, 768, (309) & [652] 2)OrgSynth **11** (1931), 28 3)CondChemDict (1961), 280

Citraconic Acid Azide or Citraconyl Azide, $CH_3.C(CO.N_3):CH(CO.N_3)$; mw 180.13, N 46.66% crysts with irritating odor, mp 114° (dec), explodes mildly at higher temps; unstable; was prepd by treating citraconyl hydrazide in dil soln (0.4%) with aq Na nitrite and normal HCl at 0°
Refs: 1)Beil - not found 2)M.Freri, AttiAccad-Lincei **22**, 264 (1935) & CA **30**, 6375 (1936)

Citraconic Acid, Action of Nitric Acid on. Baup (Ref 1), was the first to study the action of nitric acid on citraconic acid. He claimed to separate two main products which he named Eulyt($C_6H_6N_4O_7$) & Dyslyt ($C_8H_6N_4O_6$). Some later investigators, such as Ciamician & Zatti (Ref 3) and Angeli (Ref 4) could not obtain Dyslyt (called in Engl *dislite*) together with Eulyt (called in Engl *eulite*); but others, such as Bassett (Ref 2) and Fusco et al (Ref 7 & 8) obtained both products. Eulite is an expl
Refs: 1)S.Baup, Jahresberichte für Chemie **1851**, 405; AnnChimPhys **33**, 192 (1851) & Ann **81**, 102 (1852) 2)H.Bassett, ChemNews **24**, 631 (1871) & ChemZtr **43**, 157 (1872) 3)G.Ciamician & C. Zatti, Gazz **19**, 263 (1889) & ChemZnt **60**, 118 (1889) 4)A.Angeli, Ber **24**, 1303 (1891) 5)A. Quilico, Gazz **65**, 1203 (1935) & CA **30**, 5220 (1936) 6)A.Quilico & R.Fusco, Gazz **66**, 278-99 (1936) & CA **31**, 1805 (1937) 7)R.Fusco, Gazz **68**, 380-6 (1938) & CA **32**, 9066 (1938) 8)R.Fusco & S.Zumin, Gazz **76**, 223-38 (1946) & CA **41**, 2038 (1947)

Citraconic Acid, Ozonization of. Among the attempts to prep and isolate an ozonide of citraconic acid, may be cited the work of Briner & Frank, who conducted ozonization in methanol at 60°. They obtained a soln of a product which exhibited some reactions of an ozonide. The product was, however, unstable and could not be separated from the solvent
Ref: E.Briner & D.Frank, Helv **21**, 1306-08 (1938) & CA **33**, 5354 (1939)

Citric Acid and Derivatives
Citric Acid (2-Hydroxy-1,2,3-propane-tricarboxylic Acid) (Citronensäure or 3-Oxy-propan-tricarbonsäure in Ger), $C_6H_8O_7$, mw 192.12; col crysts, mp 153°, bp decomp, d 1.542 at 20/4°; sol in w or alc; sl sol in eth; can be obtained from lemon, lime or pineapple juice. It has been used in foods, drinks, medicines, NC lacquers, alkyd resins, etc (Refs 1,3 & 5). Davis (Ref 2) describes its use in prepn of HMTPDA; and Zenftman & Forlin (Ref 4) patented its use in fuse compns
Refs: 1)Beil **3**, 556, (194) & [359] 2)Davis (1943), 451 3)Kirk & Othmer **4**, (1949), 8-23 (A review with 45 refs) 4)H.Zenftman & J.Forlin, USP 2590060 (1952) & CA **48**, 6700 (1954) [Fuse compn using thermostatic binder can be continuously extruded, provided ca 0.4% of a special antihardening agent, such as citric acid or $KHC_2O_4.H_2O$ is included in formulation consisting of 79.6% fuse pdr (mixt of Pb_3O_4 41, KNO_3 33 & Si 26%) and 20% colloided NC (mixt of NC 50, DBuPh 48 & DPhA 2%)] 5)CondChemDict (1961) 280

Citric Acid Azide or Citric Acid Triazide, $N_3.OC-.CH_2.C(OH)(CO.N_3).CH_2.CON_3$; mw 267.17, N47.19%; ndls (from eth); mp melts & explodes on warming slightly; explodes violently in flame, on impact or friction; sl sol in eth & more diffc in alc; was prepd by Curtius (Ref 2) from hydrochloride of citric acid hydrazide and Na nitrite
Refs: 1)Beil **3**, (198) 2)T.Curtius & F.Sauvin, JPraktChem **95**, 248-49 (1917); JCS **112** I, 639 (1917) & CA **12**, 901 (1918)

Citric Acid Nitrate, $HOOC.CH_2.C(ONO_2)(COOH).CH_2.COOH$; mw 237.12, N 5.91%; crysts; sol in w or alc; insol in eth; was prepd by treating anhyd citric acid with 1p fuming nitric acid and 2ps concd sulfuric acid.
Refs: 1)Beil **3**, 566 2)J.P.Champion & H. Pellet, BullFr [2], **24**, 448 (1875) & ChemZtr **47**, 37 (1876)

Citronellic Acid Ozonide and Ozoneperoxide. Two slightly expl compds, $C_{10}H_{18}O_7$ and

$C_{10}H_{18}O_6$, were obtained by Harries & Himmel (Ref 2) on treating citronellic acid, $C_{10}H_{18}O_2$ (Ref 1), dissolved in CCl_4, with ozone. On evapn of CCl_4 a col syrup was obtained, which decompd on contact with w
Refs: 1)Beil **2**, 445-6, (194-5) & [418-19] 2)C.Harries & A.Himmel, Ber **41**, 2189 (1908) & CA **2**, 2807 (1908)

Civil Engineering, Uses of Explosives in. The use of expls in civil engineering includes: road-making, excavating for railroads, quarrying, blasting, tunneling, demolition, breaking up mass concrete foundations, etc.
Ref: J.Lorimer, JInstCivilEngrs (London) **26**, No 5, 50-112 (1946) & CA **41**, 285 (1947)

Civil (or Civilian) Explosives, also called **Commercial (or Industrial) Explosives.** See under COMMERCIAL or INDUSTRIAL EXPLOSIVES in Vol 3 of this Encyclopedia; BLASTING EXPLOSIVES in Vol 2 and Agricultural and Forestry Uses of Explosives in Vol 1

CK. U.S. military designation for Chemical Warfare Agent,Cyanogen Chloride. See Vol 2, pC167-L

C-Kampfstoffe (Chemische Kampfstoffe). Ger for Chemical Warfare Agent. A paper on defense against chemical warfare was recently published by E.Plotze in Explosivst **1963**, 115-20ff

CL. A smokeless proplnt consisting of NC alone. It was used in England until 1900, when it was replaced by proplnt Cooppal (qv), originated in Belgium, but also manufd in England
Refs: 1)Cundill (1889) in MP **5**, 309 (1892) 2)Daniel (1902), 143 & 155

CL. US military designation for chlorine when used as a Chemical Warfare Agent. See Vol 2, pC167-L

Claessen's Detonator. A detonator consisting of a tube open at both ends to allow the chge to be ignited at both ends instead of at one end only, so that two waves of deton will meet at about the midpoint of the tube, thus causing more effective deton of the main chge in which the detonator tube is inserted
Ref: C.Claessen, BritP 21344 (1911) & CA **7**, 1100 (1913)

Claessen's Explosives. Following expls, not including those used in primers or detonators, were patented by C.Claessen: a)GerP 179789 (1904) &CA **1**, 2203 (1907) (NG expls are rendered low-freezing by incorporating nitrated derivs of glycol) b)FrP 355695 (1905) & CA **1**, 1344 (1907) (Use of Tetryl or Ethyl Tetryl as components of expl compns in shells, torpedoes or detonators) c)FrP 355844 (1905) & CA **1**, 1344 (1907) (NG expls are rendered low-freezing by incorporating 5-20% dinitromonochlorhydrin) d)BritP 24240 (1906) & CA **1**, 2646 (1907) (AN expls made safe for use in coal mines contg firedamp by incorporating some chrome alum or Amm chromate) e)FrP 364803 (1906) & CA **1**, 2959 (1907) (NG expls rendered low-freezing by incorporating dinitrodiglycerol) f)BritP 2509 (1908) & CA **3**, 377 (1909) (Weather-proof blasting expls are obtd by intimately mixing ammoniacal K nitrate with aniline-metallic compds) g)USP 957307 (1910) & CA **4**, 2378 (1910) [A waterproof blasting expl: AN 70-95, K nitrate 0-20 & 5% of a comp of aniline with a salt (such as Al nitrate, Cu sulfate, etc), with or w/o sugar] h)FrP 463714 (1913) & CA **8**, 3238 (1914) [Expls based on Hexanitroethane (HNEt)] i)BritP 24839 (1913) & CA **9**, 1249 (1915) [Expls contg HNEt, such as 1)HNEt 64 & TNT 36%-suitable for blasting purposes and 2)HNEt 45 & TNT 55%-suitable for filling shells] j)SwedP 40981 (1916) & CA **10**, 2800 (1916) (TeNAns & Tetranitrophenetole as replacements for TNT in expl mixts)

Claessen's Primer and Detonator Mixtures. The following compns were patented by C. Claessen: a)FrP 451925 (1912) & CA **7**, 3843 (1913); GerP 265025 (1912) & CA **8**, 251 (1914) BritP 29901 (1912) & CA **8**, 2062 (1914) (In prepn of primer compns in which a portion of the fulminate is replaced by PA or TNT, the ease of firing and power can be further increased by the replacement of these latter compds with PETN) b)FrP 455369 (1913) & CA **8**, 1672 (1914); BritP 6057 (1913) & CA **8**, 2949 (1914) (Compns for small-arms primers having shorter flame than those prepd with Na sulfide & MF, consist of N_4S_4 alone or in combination with oxidizing agents, such as K chlorate or perchlorate, Ba nitrate, Pb dioxide, etc) c)FrP 459979 (1913) & CA **8**, 3238 (1914); BritP 13086 (1913) & CA **8**,

3860 (1914) (Use of LA in conjunction with MF or MF+$KClO_3$ in detonators contg a nitro compd as a base chge) d)BritP 16456 (1913) & CA **9**, 153 (1915) (Use of LA in detonators in conjunction with a small quantity of BkPdr, guncotton, picrates or perchlorates and a nitro compd as a base chge) e)GerP 281497 (1913) & CA **9**, 1996 (1915); GerP 284400 (1913) & CA **10**, 970 (1916) [Chges in detonators to replace MF or its mixts with KNO_3. Eg: Tetryl (base) 0.85g, LA 0.15g & K picrate 0.05g] f)GerP 288655 (1913) & CA **10**, 3162 (1916) (Use of 2,3,5,6-TeNAns as a base chge in detonators) g)GerP 289446 (1914) & CA **10**, 2800 (1916) (Use of 2,3,4,6-TeNAns as a base chge in detonators instead of 2,3,5,6-TeNAns) h)SwedP 40297 (1916) & CA **10**, 1597 (1916) (A priming compn consisting of LA together with a chge of MF or MF+$KClO_3$ for use in detonators contg a nitrocompd as a base chge) i)SwedP 40749 (1916) & CA **10**, 2525 (1916) (Priming compn for percussion caps consists of MF or LA mixed with K chlorate or other oxidizer)

Claessen's Propellants. The following compns were patented by C.Claessen: a)BritP 16725(1906) & CA **1**, 942 (1907); GerP 194874 (1906) & CA **2**, 2017 (1908) and FrP 368344 (1906) & CA **3**, 119 (1909) (NC & NC-NG proplnts contg completely substituted ureas which serve as flash-reducers and stabilizers) b)USP 909546 (1909) & CA **3**, 1088 (1909) (NC-NG proplnts contg ca 5% of diethyldiphenylcarbamide) c)USP 914113 (1909) & CA **3**, 1416 (1909) [A proplnt contg NC 70, NG 23, vaselin 5 & K lactate 2% (which can be replaced by K tartrate, oxalate, maleate, pyromucate or salicylate)] d)USP 979431 (1910) & CA **5**, 993 (1911); BritP 11365 (1910) & CA **5**, 2333 (1911) (NC & NC-NG proplnts with fenchone, $C_{10}H_{16}O$, added for the purpose of aiding gelatinization, reducing the combustion temp, and increasing the stability) e)BritP 23145 (1910) & CA **5**, 3158 (1911); BritP 29882 (1910) & CA **5**, 2953 (1911) and GerP 256572 (1910) & CA **7**, 1976 (1913) [In the manuf of solventless, double-base proplnts, in tubes and the like, the mixt of NC with less than 30% NG and a small amt of a non-volatile gelatinizer-stabilizer is made plastic and then shaped under high pressures (150 to 700 atm) and at temps 85 to 95°] f)BritP 3116 (1911) & CA **6**, 1992 (1912) (More detailed description of manufs of proplnts listed in item e) g)BritP 24713 (1913) & CA **10**, 118 (1916) (Proplnts prepd by gelatinization of NC with not more than 30% of nitro derivs of benzene, toluene, naphthalene or phenol, without the use of volatile solvents; the gelatinization being effected by heat and rolling pressure) h)BritP 24839 (1913) & CA **9**, 1249 (1915) (Use of Hexanitroethane in manuf of proplnts. Eg: NC 68, HNEt 16, TNT 9 & diethyldiphenylcarbamide 7%) i)SwissP 70994 (1915) &CA **10**, 2525 (1916) [Manuf of proplnts, consisting of NC & less than 30% of non-volatile nitrocompd which gelatinizes NC, by passing the mixt (in which 4% of w is incorporated) several times betw hot rollers at temp of 50-60°. This treatment gelatinizes NC and drives off most of the moisture)

Clarites. Expls based on K chlorate which contained Xanthorroea balsam, a gum originated at Botany-Bay and formerly used for prepn of PA. Several formulations, such as a)K chlorate 50 & balsam 50% and b)K chlorate 57, balsam 29 & camphor 14% were submitted to the Govt of colony of Victoria in Australia but were not authorized for use
Ref: Daniel (1902), 143

Clarke, E.S. of England patented in 1897 & 1898 a "safe" tamping for use in gaseous or dusty coal mines. In this invention, a mixt of hydrated Na carbonate 97.5 with Na sulfate 2.0 & red ferric oxide 0.5% was compressed in the form of tubes 30mm OD and 15mm ID, which could be cut into pieces of desired lengths. One piece of such tube was placed at the bottom of the bore hole and this was followed by the chge of expl and another piece of the tube. A regular tamping could close the bore-hole. The idea was to cool the gases of expln to below ignition temp of firedamp, by allowing them to permeate the material of tubes so that some heat could be spent for evap of w of crystn and also for decompn of Na carbonate
Refs: 1)E.S. Clark, EnglP's 20223 (1897) & 11674 (1898); JSCI **17**, 872 (1898) 2)Daniel (1902), 144

Clarke's Propellants. There were two varieties: a)Smokeless cannon proplnt, patented in Apr 1868, which was prepd by treating woodpulp (previously impregnated with tannin or alum, and then dried) with mixed nitric-sulfuric acid. Then the resulting NC was separated from acid, washed with w (contg some Na carbonate or bicarbonate), impreg-

nated with concd soln of K or Na nitrate, dried and colloided b)Smokeless proplnt patented in Nov 1868 and designated as *glyceropyroxyline* was prepd by treating vegetable fibres (previously impregnated with glycerin) with mixed nitric -sulfuric acid, followed by washing and drying. It was claimed that a more homogeneous blend was obtd by this method than by just mixing NC with NG

Ref: Cundill (1889) in MP **5**, 309 (1892)

Clark's Explosive, patented in US in 1900, consisted of Na nitrate, sulfur & charcoal with added PA & powdered Sb. It was recommended for use in mining

Ref: Daniel (1902), 144

Clathrates and Other Inclusion Compounds. Clathrate is one of the types of inclusion compds, also called *insertion-occlusion- or enclosure compounds* (Einschlussverbindungen in Ger). An inclusion compd may be defined as a single-phased solid consisting of a combination of two compds one of which (solid, liquid or gas) is contained within the crystalline lattice of another, the latter formed by such adducts as urea, thiourea, choleic acid, hydroquinone, cyclodextrin, etc. The components of inclusion compds, each capable of existing separately, are not united chemically, but are held together by secondary valence forces and by hydrogen bonds. All of these compds may be considered as consisting of two distinct components, the "host" and the "guest". Sometimes more than two components form an inclusion compd

In the type called *clathrate,* known also as the *cage inclusion compound,* the "guest" is retained in closed cavities or cages provided by the cryst structure of the "host". Generally, the cage and its enclosed molecule or molecules are taken as a unit cell. The first representative of this type is the hydroquinone-sulfur dioxide (where hydroquinone is the host and SO_2 the guest), first prepd in 1849 by F. Wöhler, but it was only in 1947 that this compd was shown to be an inclusion compd. This discovery was made by Powell et al (Ref 2), who also coined the name "clathrate", which means "enclosure" in Latin. Among other clathrates prepd later by various investigators, may be cited: hydroquinone-inert gas, phenol--xenon, phenol-sulfur dioxide, monoamminenickel (II) cyanide, phenol-mesityl oxide, urea-oleic acid urea-hydrogen peroxide, and aniline-tetranitromethane clathrates. Although the number of clathrates available at present is rather limited, some interesting applications have been already realized as indicated by Mandelcorn (Ref 12, pp 837-38). For example, the hydroquinone-inert gas clathrates, which are solid granular materials, can give off (on heating or on dissolving) gases up to 10% of total wt of the clathrate. As these gases are inert and inflammable, a suggestion may be made to investigate the possibility of using some clathrates as coal mining expls, where heaving action rather than brisant is required. The aniline-tetranitromethane clathrate might prove to be a useful HE. The urea-hydrogen peroxide clathrate might prove to be useful as a proplnt. It is suggested by us to investigate the possibility of enclosing some brisant, but unstable expls (such as PNA; potassium Tetranitroethanate, etc) in a cryst lattice of a stable compd, thus obtaining a stable HE clathrate

The possibility of using clathrates as solid proplnts was investigated by Francis H. Snyder & Associates, Inc, but the results of their work are classified (See Ref 11, and four conf quarterly reports listed in that Ref)

Another type of inclusion compd is the *channel or canal compound.* Here the straight chain compds, such as hydrocarbons, acids, esters, alcohols, aldehydes, ketones, etc are enclosed in the channels formed by compds, such as urea, thiourea, choleic acids, cyclodextrins, etc. As examples of channel compds may be cited, the urea-decone compd, $[CO(NH_2)_2]_9.C_{12}H_{26}$, and various zeolites. (See also Ref 10, pp431-43)

Another type of inclusion compd is the *layer or sandwich compound.* This includes certain hydrated clays (such as halloysite and montmorillonite) which form layer- or sandwich-inclusion compds with polar organic molecules (such as alcohols, glycols, some hydrocarbons, etc) which replace the water, loosely bound in clays (Ref 10, pp445-7)

Other types of inclusion compds have been described in the literature but info on them is relatively scarce or there is still dispute regarding their structure (See Ref 10, pp430 & 447)

Refs: 1)Beil-not found 2)H.M.Powell et al, JCS **1947**, 208 & **1948**, 61, 71 & 571; Nature **163**, 566 (1949); Endeavour **9**, 154 (1950) 3)F.Cramer, AngewChem **64**, 437 (1952) & **68**, 115 (1956) (Review on clathrates) 4)F.Cramer, "Einschluss-

verbindungen", Springer, Berlin (1954) 5)Ullmann **6** (1955), 253-66 (Einschlussverbindungen) 6)G.Montel, BullFr **1955**, 1013-17 & CA **50**, 22 (1956) [A review on the props of insertion (occlusion) compds] 7)F.D.Cramer, RevsPure ApplChem **5**, 143-64 (1955) & CA **50**, 163 (1956) (A review on cage and channel structures) (80 refs) 8)W.Hückel, "Theoretical Principles of Organic Chemistry", Elsevier, Amsterdam, **1** (1955), 166-69 & **2**, (1958) 446-47 9)Fieser & Fieser (1956)-not found 10)Kirk & Othmer, First Suppl (1957), 429-48 ("Inclusion Compounds", by D.Swern) (49 refs) 11)S.C.Nagel of Francis H. Snyder & Associates, Inc, "Research on Clathrates as Possible Solid Propellants" (Unclassified Title), Final Report, July 1959; Materials Laboratory Contract No AF33(616)-6013, Project No 3059, WPAFB, Ohio (Conf; not used as a source of info) (See also 4 conf refs) 12)L.Mandelcorn, ChemRevs **59**, 827-39 (1959) & CA **54**, 1145 (1960) (Clathrates; a review with 65 refs)

Clausewitz, Karl von (1780-1831). Prussian Army officer of Polish origin. He is known for his books on science of war, especially the "Vom Kriege", in 3 volumes (1833), translated into English under the title "On War"
Refs: 1)Webster's Biographical Dictionary (1959) 315-16 2)L.Montross, "War Through the Ages", Harper & Bros, NY (1960), 582-85

Clay. (Argile in Fr, Thon in Ger, Argilla in Ital, Arcilla in Span and Glina in Rus) General term for certain earthy materials obtained on weathering or alteration of Al-bearing silicate rocks, especially those of the feldspar type. They are plastic when wet and harden when heated to redness or higher (Refs 1,2,3,5,6,7,10 & 11)

Clays are used as ceramic raw materials for pottery, crucibles, mortars, etc. One of the Japan hand grenades (Type 4) consisted of a hollow sphere made of pottery clay. It was filled with 3.8 oz of *Type 88 Explosive*, also called *Karrito* (Amm perchlorate 66, Si carbide 16, woodpulp 12 & oil 6%) (Ref 8). Accdg to Davis (Ref 4), clay was used for making perforated plugs in pyrotechnic rockets. Sato (Ref 9) patented its use in the following expl compn: AN 45-60, RDX 15-20, NG 0-10 & hydrated potter's clay 25%. The resulting product contained 2 to 6.25% of water (See also Bentonite in Vol 2, pB34-L and China Clay in Vol 2, pC179-R)
Refs: 1)Mellor **6** (1925), 331-407 2)Ullmann **10** clay) 3)Thorpe **3** (1939), 191-204 & **5** (1941), 207-08 (Fire clay) 4)Davis (1943),73ff 5) Kirk & Othmer **4** (1949), 24-86 6)Ullmann **3** (1953), 331-407 (Aluminium und Tonerde) 7)R. E.Grim, "Clay Mineralogy", McGraw-Hill, NY (1953) 8)Anon, "Japanese Explosive Ordnance" TM **9-1985-4** (1953), 228 9)A.Sato, JapP 988 ('54) & CA **48**, 14210 (1954) 10)A.B.Searle & R.W. Grimshaw, "The Chemistry and Physics of Clays and Other Ceramic Materials", Interscience, NY (1959) 11)CondChemDict (1961), 282

Cleacid. A Ger code name for Lead Azide.
Ref: Pérez Ara (1945), 564

Cleaning of Glassware Used in Calibration of TNT Thermometers. See Vol 2, pC7-R, under Calibration of TNT Thermometers

Cleaning of Laboratory Glassware and Cleaning Mixtures. See Lange (1961), 1776

Cleaning of Mercury. Mercury used in nitrometer detns can be sufficiently purified by touching its surface with several pieces of filter paper (in order to remove sulfuric acid), followed by filtration either thru several layers of filter paper, folded in a glass funnel and perforated at the bottom with several pin holes (Ref 2), or thru special filters, such as described in Ref 5

If mercury is contaminated with amalgams or with metallic oxides, a more elaborate method of purification should be used. For example, mercury previously used in polarographic tests can be purified in the following manner:
a)Remove the scum, if any, by means of filter- or tissue paper b)Transfer the mercury into a heavy-walled suction flask equipped with a one-hole stopper thru which passes a glass tube leading nearly to the bottom of the flask c)Cover the mercury with a layer of dil nitric acid (1:3), connect the side arm of suction flask to the water vacuum pump, and, by maintaining moderate suction, bubble air thru the mercury for several hours in order to oxidize the metallic impurities d)Remove the acid and wash the mercury with several portions of water e)Filter thru several layers of filter paper folded in a glass funnel and perforated at the bottom with pin holes f)Transfer the mercury into a shallow dish and leave it in an oven, provided with a good ventilation, at 100°

for several hrs g)Refilter it if the surface is not bright (Ref 2) (See also Refs 3 & 4)

If very pure mercury is required, (such as for use in calibration of lab glassware), it is necessary to distil it. A good method for its distn is described in Ref 1

Refs: 1)H.S.Booth & N.S.Jones, JIEC **19**, 104-7 (1927) (A still for purification of mercury) 2)Clift & Fedoroff, Vol 3 (1944), Chap 2, pp32-3 3)Lange (1961), 1778 4)ChemRubHdb (1962), 3421 5)Fisher Co, Pittsburg, Pa, Catalog 63, p434, Item 9-771-5 (Filter, Mercury)

Cleaning of Nitrometer Parts. It was described by Clift & Fedoroff, Vol 1 (1942), Chap 1, pp27-8

Cleaning Solutions for Laboratory Glassware. If glassware is not very greasy, it can be cleaned by soaking it first in water contg soap or detergent, then draining & drying and transferring it into a hot mixt of crude concd sulfuric acid, tech grade Na dichromate and some w, prepd as described in Ref 1. After allowing it to soak in the cleaning mixt for a while, the glassware is rinsed thoroughly under tap w and dried

For glassware covered with a coat of heavy greasy material, preliminary cleaning should be done either with acetone or with a concd soln of NaOH in dil alc

Refs: 1)Lange (1961) 1776 2)ChemRubHdb (1962) 3420

Clearing of Mine Fields. See under Mines, Mine Fields and Their Clearing

Clees Cinematograph. A device invented before WWI and formerly used for ballistic measurements

Ref: Cranz **3** (1927), 344-46

Clement or Fuchs Powder, patented at the end of 19th century, consisted of K chlorate, saltpeter, charcoal, sulfur & finely pulverized tortoise shell

Refs: 1)Cundill (1889) in MP **5**, 339 (1892) 2)Daniel (1902), 145 3)Giua, Trattato **6** (1959), 399

Clepsydra or Klepsydra. See under CHRONOGRAPHS

Clermonite. A Belg 40-grain, fibrous, shot-gun proplnt manufd by the Cooppal Co

Ref: Marshall, Dict (1920), 22

Cleveland Open Cup (COC). An apparatus used for testing flash and fire points of flammable liquids

Refs: 1)Scott & Furman **2** (1939), 1732-36 2)ASTMStds **4** (1962), 1121-25 & **7** (1962), 31-5

Cleveland Ordnance Plant. A field installation, formerly known as "Cleveland Tank Plant." It is located at Cleveland, Ohio, USA

Ref: OrdTech Term (1962), 73

Cliffite and Super-Cliffite. Cliffite was a coal-mine expl made by Curtis & Harvey, and formerly on the Permitted List: NG 47, CC (collodion cotton) 3 & starch 50%. Super-Cliffites were of different compns: *No 1* - NG 9.5, CC 0.5, AN 59, woodmeal 6, NaCl 15 & Amm oxalate 10%; and *No 2* - NG 9.5, CC 0.5, AN 59.5, woodmeal 6, NaCl 19.5 & Amm oxalate 5%. The limit chge of No 1 was 26 oz & No 2-30 oz, and the power by swing of ballistic pendulum 2.53" for both expls vs 3.27" for Gelignite contg 60% NG

Ref: Marshall, Dict (1920), 22

Clift, George D. (1891-1962). An Amer chemist and expert in the field of explosives. He worked for nearly 50 years in the expls field. He collaborated with B.T.Fedoroff in compilation of "A Manual for Explosives Laboratories," published in 4 volumes during WWII by Lefax Soc. Inc. of Philadelphia, Pa. He was also one of the Co-authors of Vols 1 & 2 of this Encyclopedia and the author of several Picatinny Arsenal Technical Reports (Nos 2125, 2159, 2293, 2300, 2358, 2381 & 2510) and Chemical Center (Edgewood Arsenal, Md) Reports

Cloramite. A mining expl manufd after WWI by the Dinamificio di Orbetello, Italy: Ballistite or Cordite (leftovers of WWI) 64, Amm perchlorate 20, Na nitrate 15 & K dichromate 1%

Ref: Molina (1930), 362

Cloratita. A mining expl introduced before WWI by the Cloratita SA de Flix, Tarragona, Spain and used in Europe during 1914-18. It consisted of a special cartridge contg weighed quantities of finely pulverized K or Na chlorate. The cartridge was transported to place of work where it was impregnated, just before inserting it into a

borehole with a known amt of petroleum, usually 7 to 10%
Ref: Vivas, Feigenspan & Ladreda, **2** (1946), 347

Closed Bomb (or Vessel) and Instruments for Measuring Pressures Developed by Explosives or Propellants.

Closed Bomb or Vessel. It is, essentially, a thick-walled steel (or special alloy) vessel provided with one or more openings and one or more pressure-measuring devices. Thru one of the openings, which is provided with a removable plug, a charge of explosive or propellant is introduced and, after pressure measuring devices are installed, the charge is either exploded or deflagrated. Using these devices it is possible to determine one or several of the following characteristics: a)Peak or maximum pressure of gases b)Rate of change of pressure with pressure (relationship dp/dv vs p) and c)Change of pressure with time (p vs t) or pressure-time relationship

More detailed info will be given further in this section

Pressure Developed on Explosion or Deflagration. It can be either calculated or determined experimentally and expressed in kg/cm^2 (kilograms per square centimerer), in t/in^2 (Tons per square inch), in atm (atmospheres) or in psi (pounds per square inch)

The formula for approximate calcn of pressure of expln, in a const vol chamber is as follows:

$$p = \frac{nRT}{V}\left(1+\frac{B}{V}+\frac{nC}{V^2}\right), \text{ where}$$

p = gas press, V = volume per unit weight of gas, n = number of gram molecules in unit mass of gas, R = gas constant, T = absolute temperature in °C and B & C the functions of temperature and gas composition (Ref 36, p11 & Ref 61, p2-13)

If only major products of gas: CO_2, CO, H_2, H_2O and N_2 are considered, the functions B & C can be calcd from the linear sums:

$$B = (CO_2)B_{CO_2} + (CO)B_{CO} + \ldots (N_2)B_{N_2} \text{ and}$$

$$C = (CO_2)C_{CO_2} + (CO)B_{CO} + \ldots (N_2)B_{N_2},$$

where (CO_2), (CO) & (N_2) denote the number of gram molecules of the CO_2, CO & N_2 per gram of the gas of expln and B_{CO2}, C_{CO2}, etc are values of B & C for pure gases CO_2, etc (Ref 36, p11)

Using these equations for *Cordite SC*, loaded at d 0.2 g/cm^3 and developing a temp of 3100°K, the pressure p was found to be equal to 17.6 $tons/in^2$ (Ref 36, P 17)

Experimental methods for measuring pressure may be subdivided into "absolute" and "relative" methods. In the 1st method the pressure is balanced against the gravitational force on a known mass [such as when a column of liquid (usually mercury or water) is supported by the pressure or when the pressure supports a solid weight upon a small piston]. In the 2nd method a manometer or gage depending upon the elastic deformation of a body (such as a spiral spring, a diaphragm or a "crusher" body) or upon change in electrical props of a body (such as in piezoelectric and strain gages) are used

Determination of pressure developed on combustion of propellants in weapons and in rocket motors, or on detonation of explosives in projectiles is of great importance in ordnance. It permits the calculation of the safe thickness of the walls of weapon-barrels (to prevent their damage or even bursting) and the thickness of the walls of projectiles to assure their bursting into fragments on deton of expls. Studies of proplnts can be conducted either in closed bombs or in cannons (or rockets), directly. These tests also give an idea of the ballistics of the ammunition and serve as an aid in the design of the propellant-cannon-projectile combination or other similar combination. Studies of expls intended as bursting charges in projectiles are usually conducted in *closed bombs* and the results verified by field tests using projectiles with wall thicknesses calcd from closed bomb data

Before proceeding further on this subject it might be interesting to review the historical development of pressure measurements in ordnance

Historical. The first recorded attempt to measure the pressure generated when gunpowder was fired in a confined space (such as a bomb or gun barrel) was made in Italy in 1738 by d'Antony. This was followed by Robins in England (1742), d'Arçy in France (1760) and by Count Rumford in Germany (1792) (Ref 6, p192; Ref 35, p83). The device of Rumford consisted of a thick-walled iron vessel hermetically closed except for a small hole at the top. A known charge of powder (with a fuse attached) was placed inside the vessel and the hole covered by means of a heavy iron or lead cover of known weight. Then the chge was ignited and if the cover was blown off by the

force of gases resulting from expln of pdr, a heavier cover was tried in the next test. This was continued with heavier and heavier covers until the pressure did not move the cover. Knowing the wt of the cover at which the hole remained closed and knowing the area of the hole, it was possible to calculate the pressure (now known as "maximum" or "peak") developed by a chge of pdr of known wt (Ref 26, p73). This device was improved in the middle of the 19th century by Bunsen. In his apparatus the gases pressed against a disc connected to the lever of a balance. By sliding a weight on the lever it was possible to determine the pressure created by expln inside the vessel (Ref 12a, p83)

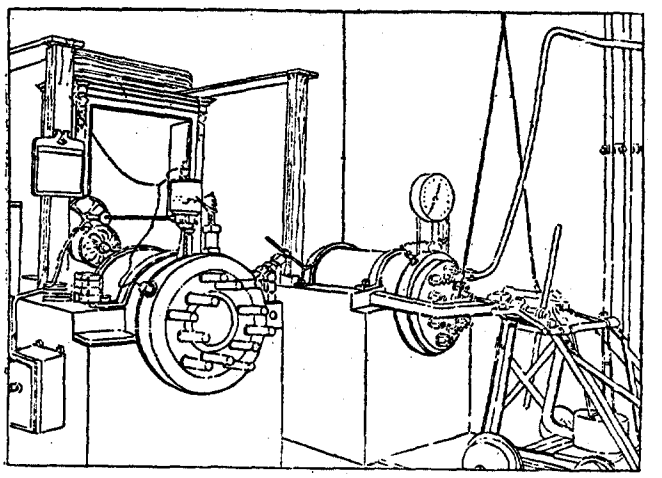
More accurate measurements could be obtained in the 2nd part of the 19th century using instruments invented independently by Maj T.J.Rodman in US (1857-1861), Gen N.Mayevskii in Russia (ca 1860), Sir A.Noble & Sir F.Abel in England (1860-1871) and M.Deprez, E.Sarrau & P.Vieille in France (1871-1883). Of these instruments, the device of Rodman, known as the "Rodman Gage" was the prototype of the present "crusher gages" It consisted of a small cylinder with a piston, to outer side of which was attached a sharp steel knife, the edge of which just touched a block of soft (annealed) red copper. The cylinder was screwed in a hole bored in the wall of a closed vessel or a gun barrel, so that the pressure of gases developed on expln inside the vessel or gun barrel could act upon the base of the piston which forced the knife edge into a disc of Cu. The depth of penetration was taken as a measure of the pressure to which the piston was subjected. The forces required to produce depressions of various depths in the Cu were predetermined by actual trials and hence the maximum (peak) pressure of the gases could be calcd. For expls developing only small pressures, lead discs (in lieu of Cu) were used and in some Rodman gages steel balls were used instead of steel knives to press against soft copper (Ref 1 p441)

Sir A. Noble of England modified in 1868 the above gage and this is now known as *crusher gage*. In Noble's device a small Cu cylindrical block was "crushed", by pressure of gases, between two flat surfaces and from the diminution of the height of the block the max pressure was calcd, or taken from calibration tables. A simpler crusher gage consisted of a small Cu cylinder placed directly inside the closed vessel or at the back of proplnt chge in a gun barrel (Ref 1, p445)

Among other investigators, who contributed to the development of methods for measuring pressures of gases in closed vessels or directly in gun barrels, may be mentioned: Navez (Belgium); Uchatius (Austria); Gadolin, Chel'tsov & Dolgov (Russia); Kelner, Deering, Crehore & Squier (US); Bichel, Petavel, Nossow, von Wuich, Schweickert, Nernst, Becker, Rumpff, Cranz, Zeiss & Schardin (Germany); Berthelot, Charbonnier, Burlot, Muraour & Aunis (France); and Sir J.J.Thompson (England)

Closed Bombs. A definition of a closed bomb is given at the beginning of this section. There are several types of such bombs, of which the following are examples:

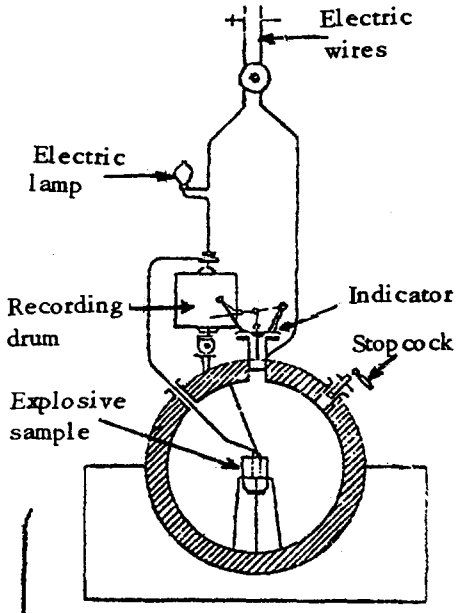
Bichel Bomb. It is briefly described in Vol 1, pVIII of this Encyclopedia. As supplement to this are illustrations from the books of Colver (1918), p606; and Vivas, Feiganspan & Ladreda, Vol 4 (1945), p101



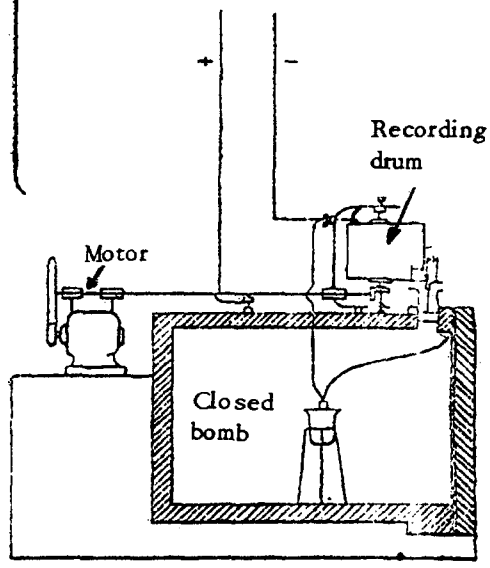
BICHEL PRESSURE GAGE

These books, as well as the book of Marshall (Ref 1, p444) and Bur Mines Bull 346 (Ref 7, pp 85-95) give a detailed description of bomb and instructions for its manipulation. This bomb was developed in 1898 in Germany. Hercules Powder Co modified the bomb devising a very effective arrangement for its closing. A bomb similar to Bichel, but vertical, was developed in Russia by Dolgov. Its brief description is given in Ref 56a, pp75-6

Vieille Type Bombs. The original bomb, developed in France in 1883 by E.Sarrau & P.Vieille, was later modified by E.Burlot & others, becoming known as "La Bombe Type Vieille" or "Le Vase



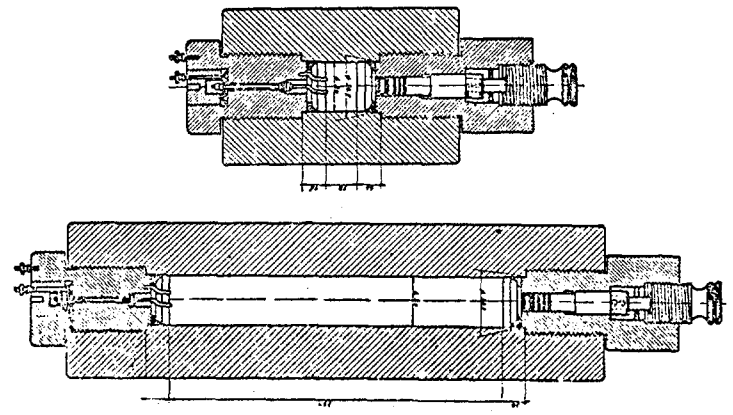
CROSS SECTION OF BICHEL BOMB



SIDE VIEW OF BICHEL BOMB

Clos". It has been successfully used not only in France but also in many other countries, among them Italy, Spain, Russia and Latin American countries (Ref 6, pp195-197; Ref 11, pp72-87; Ref 13, p91; Ref 14, pp 84-7; Ref 29a, pp 83-4 and Ref 35, p85)

The bombs employed before WWII (and probably now) in France by the CSE (Commission des Substances Explosives) were of either 150cc or 25cc capacity. They are represented here in Fig which is reproduced from Ref 11, p73

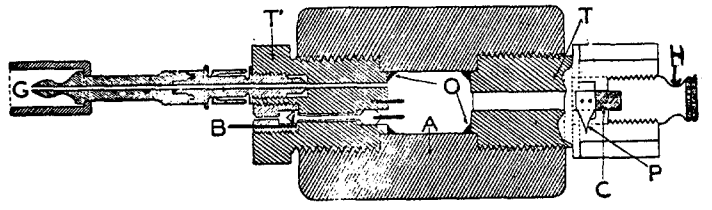


CLOSED BOMBS OF CSE

Each bomb is a steel cylindrical vessel with thick walls. One end of cylinder is closed by a threaded plug against which is placed, towards the inside of the bomb a pressure measuring device (usually a copper crusher gage) and then a charge of propInt or explosive. The other end of vessel is closed by a threaded plug which is provided with an electrical firing device. More detailed description is given in Ref 11

The procedure used with this bomb, called in French "Épreuve à la bombe" is described in detail in the book of instructions issued by the "Commission des Substances Explosives", 4e partie. This book may be obtained on request from the "État Majeur d l'Armée, 2ème Bureau, Paris, France (Ref 72)

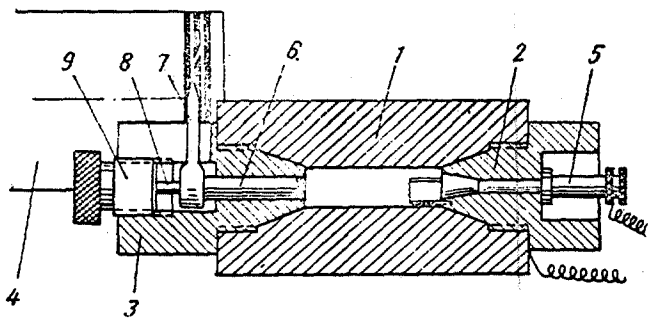
Pérez Ara (Ref 26, p65) describes another bomb, called "Burlot-Malsallez". The bomb represented here in Fig may also be used for calorimetric tests



BOMB OF BURLOT–MALSALLEZ

Here A is steel body, T-threaded perforated plug

contg steel piston, C-copper crusher, H-plug serving as an anvil, O-obturators (made of goldplated copper), P-stylus, T'-plug bearing firing mechanism B and an arrangement G for taking gas samples. A good illustrated description of Sarrau-Vieille type bomb, which was used in Russia, is given in Ref 68, pp442-44



CLOSED BOMB OF SARRAU-VIEILLE

In the Fig reproduced here from Ref 6, 1 is the body of the bomb, 2 & 3-perforated closing plugs, 5-insulated metallic rod serving for electrical firing the chge of proplnt, 6-steel piston serving for transmitting the pressure of gases to crusher 8 resting against the steel anvil 9, which is threaded. Diminution of height of crusher serves for measuring the maximum pressure-developed in the bomb. For detn of pressure-time relationship the stylus 7 (attached to cylinder 6) and the drum 4 covered with smoked paper are used. When the drum rotates at known speed and the stylus moves together with piston 6, a line is traced on the smoked paper. This line represents pressure-time relationship. The speed of drum may be detd by tracing on the paper of a sinusoid by means of a 2nd stylus which is attached to one of the legs of tuning fork of known vibrations per minute.

Several other types of closed bombs are known of which the bomb of "Krupp-Schmitz", of 3350cc capacity is the largest (Refs 21, 27a & 55)

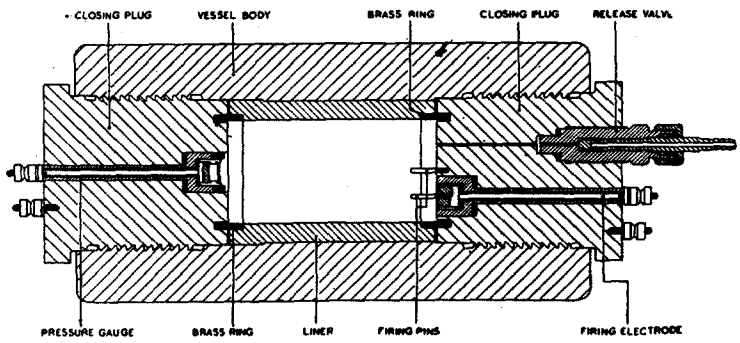
Closed bomb & pressure measuring devices which have been used in Spain are briefly described in Ref 24, pp85-6 (See also Ref 26, p80)

German Closed Bombs. Accdg to Ref 61, closed vessels were used during WWII in Germany for the following purposes: a)the development of new proplnts b)Studies of some props of proplnts c)Obtaining data for ballistic calcns

Tests designed for (a) and (b) were carried out chiefly at the Düneburg factory of Dynamit AG while those for (c) were made at the Essen factory of Krupp. Some closed vessel testing was conducted in the DWM (Deutsche Waffen- und Munitionsfabriken) research laboratory at Lübeck

More recent info on this subject may be found in Ref 55, 59 & 64

British Service Closed Vessel. Its illustrated description is given in Ref 36, pp160-62 and represented here in Fig



BRITISH CLOSED VESSEL

The body of the vessel is cylindrical in shape and is made of heat-treated nickel-steel. It is threaded internally at both ends to receive closing plugs. Buttress threads are used as such threads are less liable to seize than the V-threads. The inside of cylinder is fitted with a nickel-chromium molybdenum-steel liner, the purpose of which is to facilitate the repair in the event of damage occurring to the obturator seatings. Brass rings, which are replaced once in a while, provide gas-tight joints between plugs and the vessel bore. One of the plugs carries a pressure gage, usually of the piezoelectric type, while the other plug carries an insulated firing electrode connected to two firing pins to which are soldered two ends of "fuse" wire, which is a nichrome resistance wire 0.001" in diam. In order to prevent damage to this wire it is protected by luting. This closing plug also carries the valve for introducing an igniter gas mixture into the vessel and for releasing the combustion products after firing. Both closing plugs are hexagonal above the threaded portion. The vessel described in Ref 36, p161 and represented here in Fig is of 700cc capacity and is made to withstand pressures up to 18t/in^2 (Brit ton=2240lb or ca 1016kg)

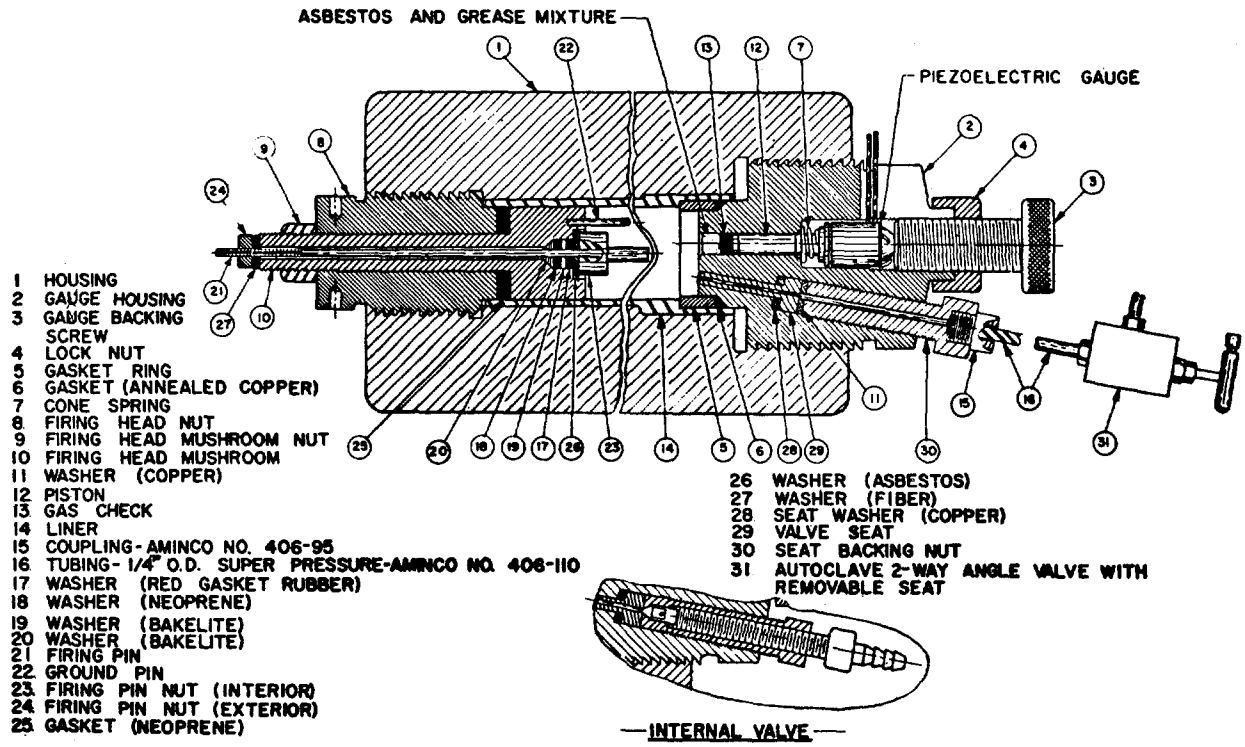
Note: Accdg to L.Shulman of PicArsn some cur-

rent American closed bombs can withstand pressures of 45t/in^2 or even higher

The above discussed bomb can be enclosed in a jacket for circulating a liquid, to enable firings to be made at different temps (Compare with American Closed Bombs conditioned to low temperatures)

Procedure for British Service Closed Vessel, as practiced in 1951 is described in Ref 36, p162. After cleaning thoroughly the interior of vessel, the closing plug contg the firing device & the release valve is screwed into the vessel and the ensemble made to acquire the desired temp by running hot or cold liquid thru the jacket. The proplnt chge, previously accurately weighed and stored for 24 hrs at desired temp, is then placed in the chamber taking care not to break the "fuse" wire of the firing device. After leaving the vessel (closed with one plug) to rest for 1-2 hours (in order to reach constant temp), the 2nd plug (previously fitted with a pressure gage) is screwed into the vessel. A vacuum pump is connected to the "release valve" and the vessel is evacuated to a pressure ca one-third of the atm. Ethylene is then introduced in an amt to reach the pressure ca 140mm Hg, followed by oxygen to create the pressure of ca 300mm Hg and finally by air to bring the pressure to ca 760mm (atmospheric). After closing the valve, the firing leads are connected, the gage is adjusted as required and the chge is fired. Various types of pressure gages can be used with this vessel (See further in this section, under Instruments for Measuring Pressure of Gases)

American Closed Vessels (or Bombs). Several Types are known. Most of them are used for investigation of burning characteristics of burning propellants. Such a bomb represented here in Fig was reproduced from Ref 70, p21



Closed Bomb Assembly

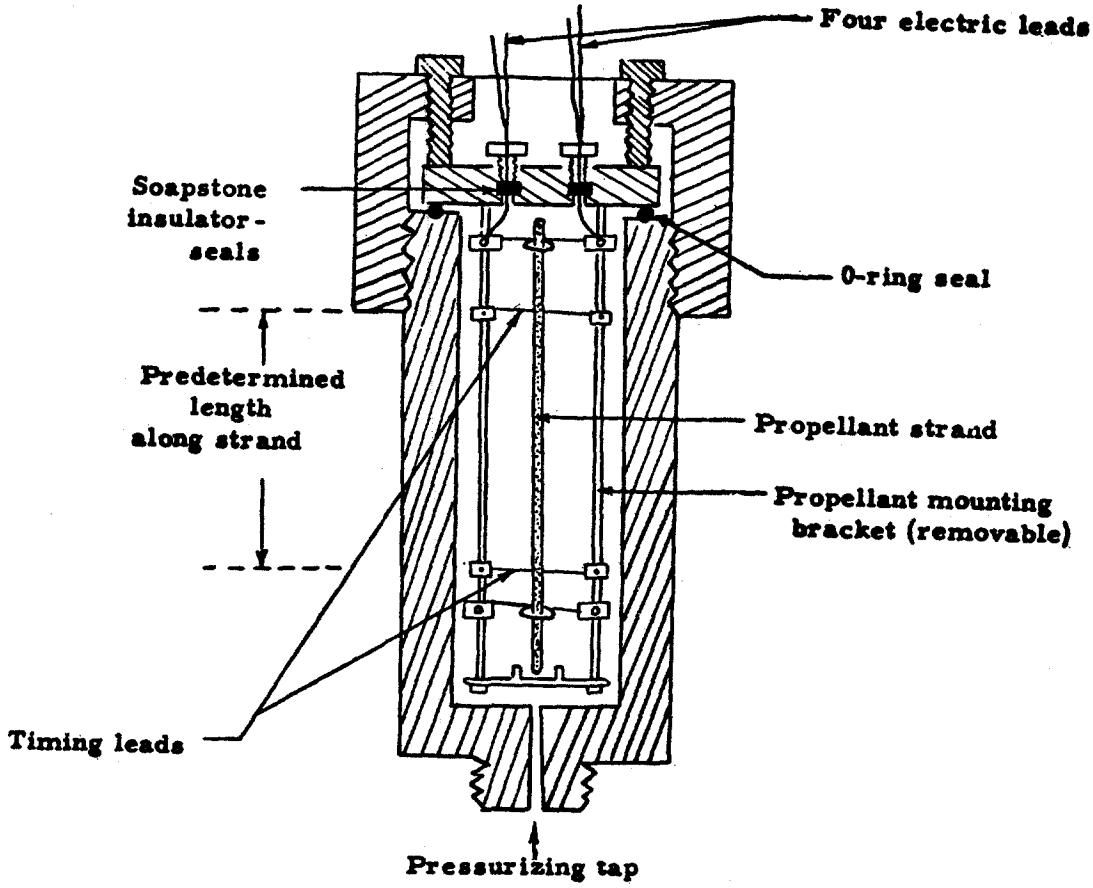
The procedure for using this type of bomb is briefly described in Ref 46, but a detailed description is given in Ref 21a. A similar bomb is used at Picatinny Arsenal. In this bomb, both perforated and nonperforated grains can be investigated. Burning characteristics may be detd at temps as low as -60C (See Refs 46, 62b & 71a)

To determine the burning characteristics of a proplnt, the grains are placed in the bomb and ignited. The pressure and rate of change of pressure build up are characterized by the amt of charge produced by the piezoelectric pressure gage. The charges built up by the

gage are fed into a recording apparatus, such as an Oscillograph (Ref 46, p2)

From the data obtained from this apparatus, the following characteristics of a proplnt can be calcd: a)Pressure and rate of change of pressure b)Relative quickness c)Relative force and d)Linear burning rate (Ref 46,pp3 -11)

Another type of Amer closed bomb, shown here in Fig, is described in Ref 69. The vessel is a heavy-walled cylinder made of steel. The proplnt strand is usually ca 1/8" in diam and 6-8" long. It is coated prior to burning with some inert plastic or paint to produce a cigaret type of burning. The strand is supported inside the vessel by a wire bracket, which is removable. Two electric leads of fine fuse wire are connected to upper and lower part of the strand at a known distance between them. Before the test, the vessel is filled, thru the "pressurizing tap", with an inert gas to a desired pressure



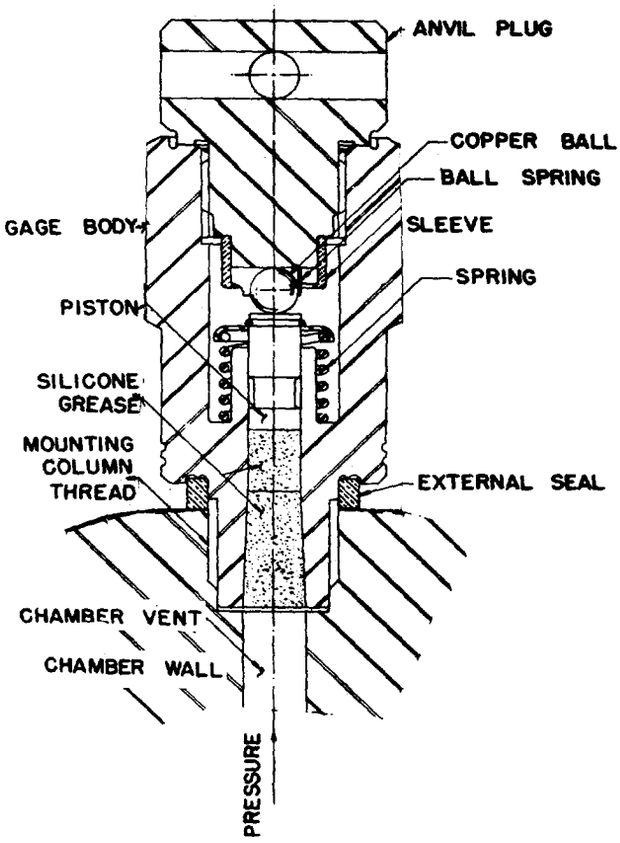
Pressure vessel for measuring burning rates of propellant as a function of pressure

Then the tap is closed and the strand ignited at the top. When flame reaches the upper "timing lead" it breaks the fuse wire. This activates electrical relay causing a timing device to start. When the flame reaches the lower wire the relay would stop the timing device. Thus a measurement can be taken for the burning to travel betw the accurately known distance along the strand. This method is well suited for lab investigations, because only small quantities of proplnts are required for each test

Note: Accdg to L.Shulman of PicArsn, this app can be used only for "nonperforated" grains (such as used in rockets). It cannot be used for "perforated" grains (such as used in cannon proplnts). This is because the flame would go down the perforations and

break both fuse wires before total strand is consumed

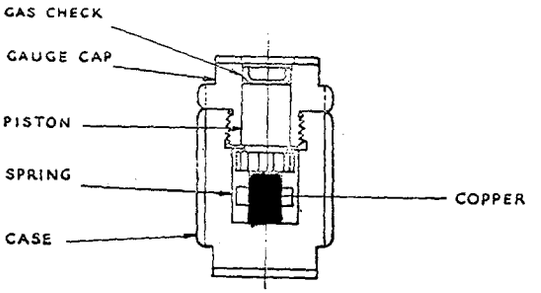
A *vented-vessel* technique for detg burning rates of rocket proplnts is briefly described in Ref 70, p A-10. Here the proplnt is burned in a simple rocket motor provided with a variety of nozzles, the purpose of them is to provide a means of varying the pressure in the vessel. The test motor and proplnt are kept under controlled (and predetermined) temperature conditions until needed. The pressure-time of the burning chge is measured with a strain gage which is connected to a Wheatstone bridge. The unbalanced emf of the bridge is amplified, applied to an oscilloscope, and recorded on film using a drum camera. In this apparatus the effect of time on burning rate may be measured after suddenly dunking the vessel containing the burning proplnt into cold water. This stops the burning at a predetermined time



EXTERNAL CRUSHER GAGE T14 (US)

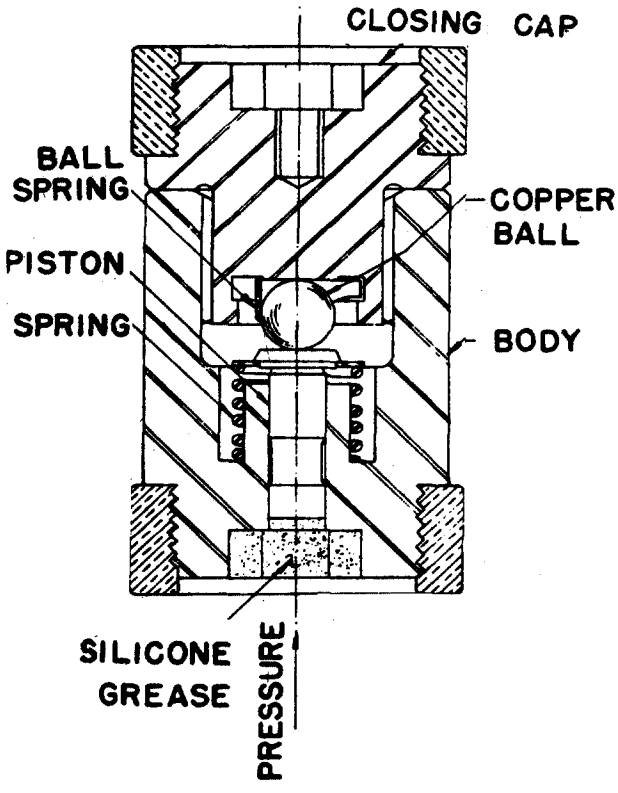
Instruments for Measuring Pressure of Gases Developed on Explosion or Deflagration. Crusher Gages. They consist of a thick-walled steel, cylindrical housing, open at one end and closed at the other. Resting on the closed end is a copper (or lead) cylinder (or ball) (called "crusher") of known dimensions and on top of it is placed a steel piston one face of which is exposed to the pressure of gases developed on deflagration of a proplnt. The gases push against the crusher and if the pressure is sufficiently high, an appreciable deformation of the crusher takes place. The value of the pressure is obtained from calibration tables

There are two types of crusher gages: exterior and interior. a)The *exterior* gage is attached on the outside of a closed vessel (or a barrel of a gun) by means of a mounting thread column. The attached illustration represents the US "External Crusher Gage", T14. Its usable range of measurement is 6 to 58 thousand psi with piston area 1/30 sq in. b)The *interior* gage is not attached, but simply placed inside a vessel or barrel. It cannot be used in small arms. The attached illustration represents the British Service Crusher Gauge (Ref 36, pp 163-64)



BRITISH SERVICE CRUSHER GAUGE

Another illustration represents the US "Internal Crusher Gage M11". A description of procedure used at Aberdeen Proving Ground, Md is given in Ref 73, pp 1-6. The usable range of measurement of this gage is 12 to 115 thousand psi with piston area 1/60 sq inches.



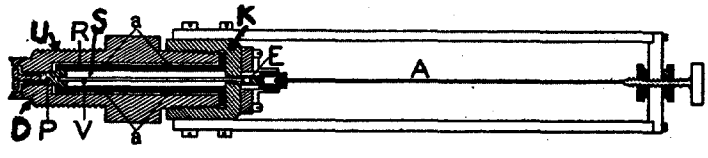
INTERNAL CRUSHER GAGE M11 (US)

The above described internal crusher gages are used only for measuring the *maximum pressure* developed on deflagration or detonation, but the external crusher gage may also be employed for approx detn of *pressure-time* relations. For this a stylus is attached to the steel piston and as the piston is pushed by pressure, the stylus traces out a line on smoked paper attached to a drum revolving rapidly at known revolutions per minute. The original apparatus was developed by Vieille & Burlot and has been successfully used by the Commission des Substances Explosives (Ref 11, pp75-6; Ref 14, pp84-6). A very good illustrated description of Fr apparatus is given by Pérez Ara (Ref 26, pp79-82). The British used a similar attachment to crusher gage but discontinued its use after 1918, because by that time more precise gages made their appearance. (Ref 36, p163)

Spring (or Mechanical) Gages. Essentially they consist of a cylindrical piston which, on being subjected to the pressure of gases, compresses a strong spring. The higher the pressure, the greater is the movement of the piston. For recording the pressure, the piston is attached to a registering device consisting of a large lever and a stylus which traces a line on a drum rapidly rotated by means of an electric motor (Ref 1, p447). One of the earliest designs of spring gages was used in the Bichel Bomb which is described in Ref 1, pp 446-48 (See also Vol 1, pVIII of this Encyclopedia). The spring in this gage as well as in similar early gages, was helical in shape and not suitable for high pressures. It was not efficient even at medium pressures, because of low magnification (Ref 36, pp168-69)

G.P.Thring of GtBritain was the first to use a different type of spring and to record its displacement thru an optical system employing a tilted mirror (Ref 36, p169). His gage was designed for use on small arms and has been employed for some time at Woolwich Arsenal (Ref 15, p15) and it was described in the British "Textbook of Small Arms" (1929 edition)

A spring gage suitable for measuring high pressures in thick-walled vessels, such as a closed bomb or a barrel of a cannon was invented in England before WWI by Petavel and is known as *Petavel Manometer* or *Optical Spring Gauge*. Its illustrated description is given in Ref 1, p448 & Ref 26, p85

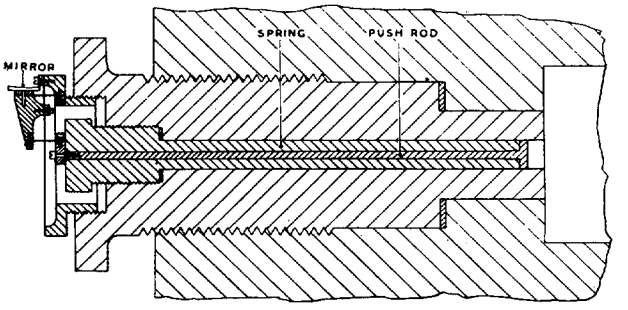


PETAVEL MANOMETER

The device consists of a rigid cylindrical container (body) which screws by means of the thread, U, into the closed bomb (or barrel of a gun), the end of the piston, P, being flush with the inside surface. An air-tight joint is formed by the ring, D, on the manometer pressing against a flat edge. The tubular spring, 5" in length, used in this gage should be very rigid in order to avoid vibrations. It is prevented from buckling by making it fit tightly to the tube, R, which is provided with two annular enlargements a and a. The left side of the spring is free and leans against the piston, P, whereas the other end is held by the nut, K. The movement of piston is communicated by means of the rod V, to an optical arrange-

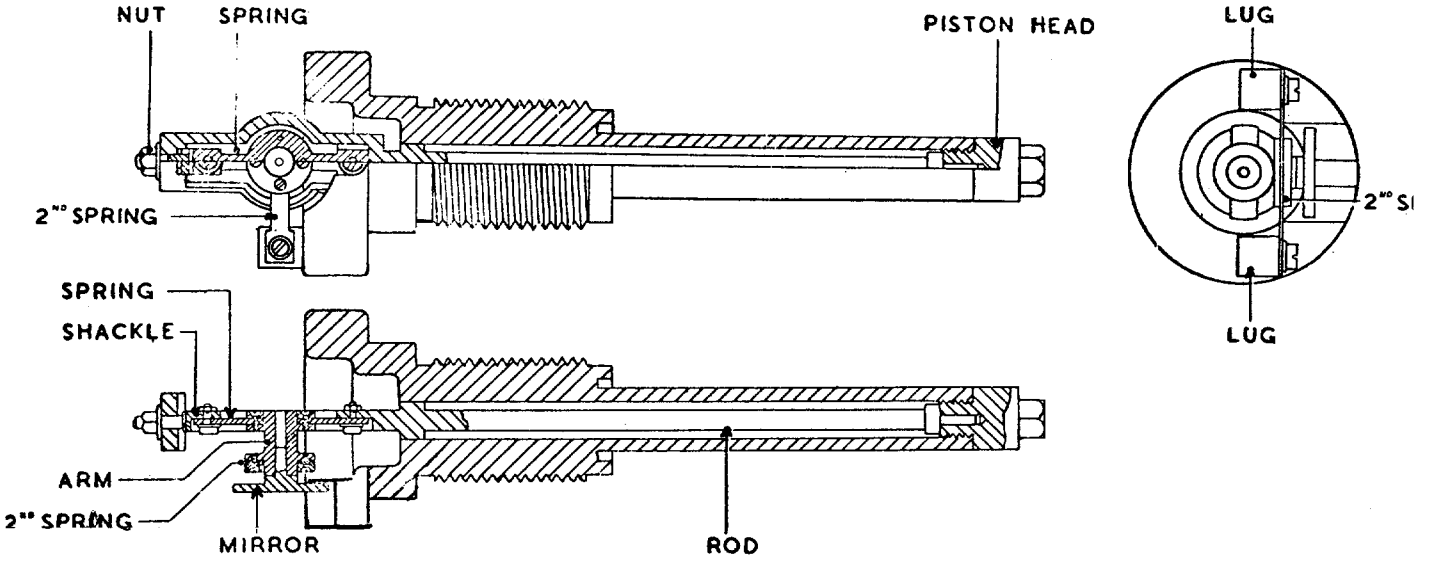
ment which amplifies small displacements of the spring (which are usually not greater than 0.25mm). The amplifier consists of a lever carrying a small mirror, E. The lever is kept pressed against a knife edge (not shown in figure) by the tension of the wire, A, which is stressed almost to its limit of elasticity. As the lever follows the motion of the piston, the mirror records it much magnified on the rotating drum of a photographic camera

Several types of spring gages based on the principles of Petavel were developed in GtBritain after WWI and adopted by ARE (Armament Research Establishment). Three of these gages are briefly described in Ref 36, pp169-70. The first design is a small gage developed ca 1931, known as "ARE Spring Gauge, First Design" originally described in Ref 8. The second, larger gage, known as "ARE Spring Gauge, Second Design" is shown here in Fig. It was originally described in Ref 12. No description of the above two gages is given in Ref 36



ARE SPRING GAUGE, SECOND DESIGN

A more recent type of Brit spring gage, called in Ref 36, p171 the "High-Pressure Spring Gauge" is illustrated here in Fig. Accdg to the description given in pp 170 & 172 of Ref 36, in this device, one end of the



HIGH–PRESSURE SPRING GAUGE

rod screws into the piston-head which, in turn screws into tubular part of the gage body. The other end of the rod is shaped to carry and to make tense a flat steel spring. One end of this spring is rigidly fixed to the rod, while the other end is attached to a shackle which passes thru the end of the rod and is threaded for a nut. When the nut is rotated clockwise, the tension is applied to the spring. A 2nd flat spring is secured to two lugs which are integral with the gage body. The two springs are in parallel planes but their axes are at right angles; they are connected by an arm holding a mirror. Pressure on the piston head compresses the tubular part of the gage body. This compression is transmitted thru the rod to the 1st spring; the motion of this spring, which is connected to the 2nd spring by the arm, tilts the mirror, which is illuminated by light from a slit. The

reflected beam makes a record on a film attached to a revolving drum camera. A special arrangement, briefly described in p172 of Ref 36, allows one to obtain a continuous time trace superimposed on the pressure record

Note: Accdg to Ref 36, p168, spring gages have some advantage over piezoelectric and strain gages because they require little specialized knowledge to handle them. They have some value in closed vessel work as a laboratory substandard

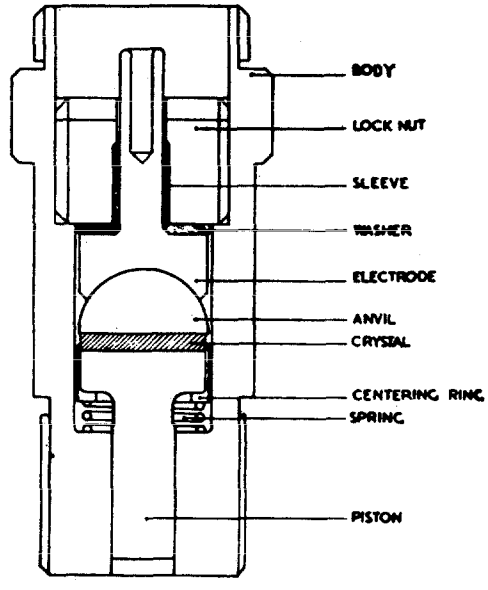
Piezoelectric Gages. These gages utilize the property of certain crystals (such as quartz or tourmaline), to develop, when stressed, electric charges of opposite signs at the end of crystal's electric axes. This property, known as *piezoelectricity* (from the Greek word "piezo" which means "to press") was discovered in 1880 by brothers Pierre and Paul Curie. No practical application of piezoelectricity was made until W.G.Cady developed, during WWI, the "piezoelectric resonator", an apparatus used for underwater signalling. More important was, however, the "echo method" developed in France by P. Langevin, also during WWI. This method became a valuable means for locating immersed objects and of exploring the ocean bottom. Langevin thus became the originator of supersonics (ultrasonics)

The first application of piezoelectricity to measure the pressure developed on expln was done by Sir J.J.Thomson in 1917 (Ref 36, p164) and this was followed by D.A.Keys in 1921 and by J.C.Karcher in 1922. The device constructed by Karcher at the US Bureau of Standards used quartz and proved to be successful to obtain the indicator diagram of a gun discharge (Ref 3)

Two types of piezoelectric gages are in common use, one utilizes a crystal of quartz, the other of tourmaline.

In a typical *quartz gage,* a thin plate (lamina) is cut from a crystal in such a manner that the flat, parallel faces of lamina are perpendicular to one of the three electric axes of quartz. When subjected to a compressional force transmitted by means of a piston (or a diaphragm), the lamina presses against an anvil and as result of this action, very weak electric charges of opposite sign are developed at both surfaces of lamina. These charges are proportional to pressures exerted on the crystal. The charges can be measured by means of a supersensitive mirror-galvanometer, with terminals connected to isolated wires leading to both surfaces of lamina. One terminal, as well as the body of the gage, is grounded. As the current is weak, it can be amplified as described under tourmaline gage

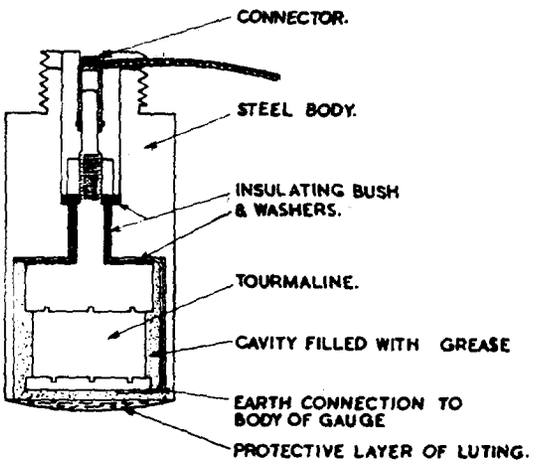
The Fig below represents one of the British quartz gages



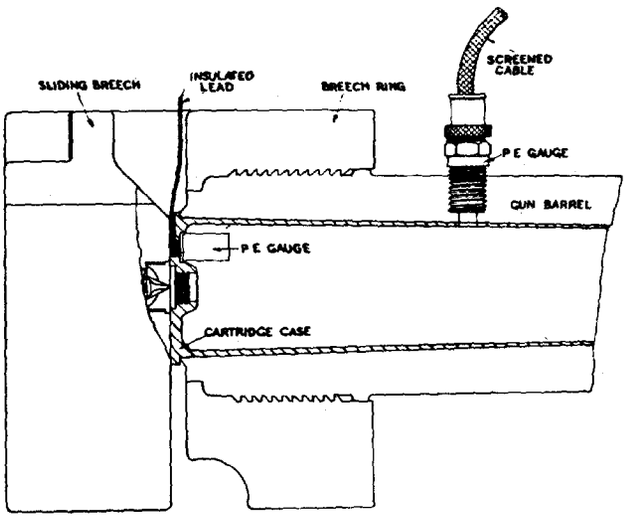
PIEZOELECTRIC GAUGE (QUARTZ)

Accdg to Pepin Lehalleur (Ref 14, pp92 -3), the quartz gage has certain advantages over the tourmaline gage, but the *tourmaline gage* has the advantage that the crystal can be subjected to hydrostatic pressure and it thus avoids the potential sources of inaccuracy associated with the use of pistons (Ref 36, p165). The elimination of piston leads to a very simple design, as can be seen from Fig which represents the British tourmaline gage

In this device, one face of tourmaline disc is connected to an insulated electrode, while the other face is connected to the body of the gage by means of a metallic plate cemented to that face. The interior of the body surrounding the crystal is filled with grease which protects the crystal from the hot ionized expln gases and transmits the pressure to it. The variations of pressure applied



PIEZOELECTRIC GAUGE (TOURMALINE)

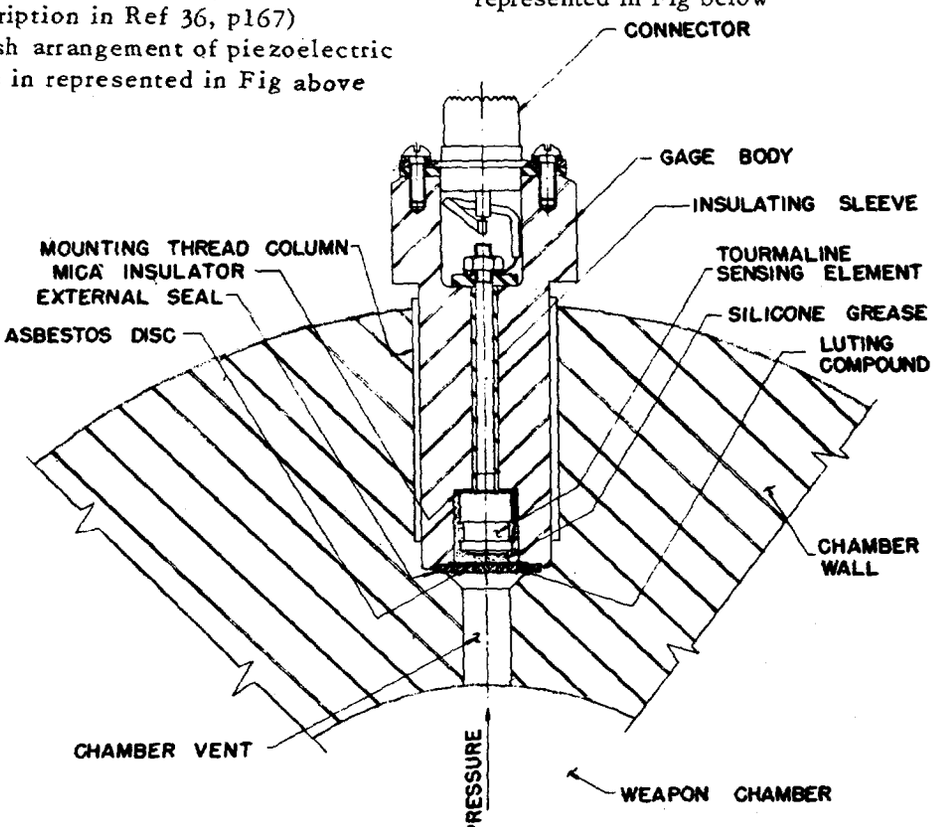


ARRANGEMENT OF PIEZOELECTRIC GAUGES IN GUN

to the crystal are recorded, as variations of voltages, by a cathode-ray oscillograph located at a convenient distance from the closed vessel or from the gun and linked to the gage by an electric cable (See more detailed description in Ref 36, p167)

The British arrangement of piezoelectric gages in guns in represented in Fig above

The current US piezoelectric tourmaline gage, known as "Piezoelectric (Tourmaline) Pressure Transducer (External Type)" is represented in Fig below



CROSS SECTION OF PIEZOELECTRIC (TOURMALINE) PRESSURE TRANSDUCER (EXTERNAL TYPE)

It is briefly described in Ref 73, pp8-9

Piezoelectric gages are very suitable for measuring rapid pressure changes because of the absence of inertia effect

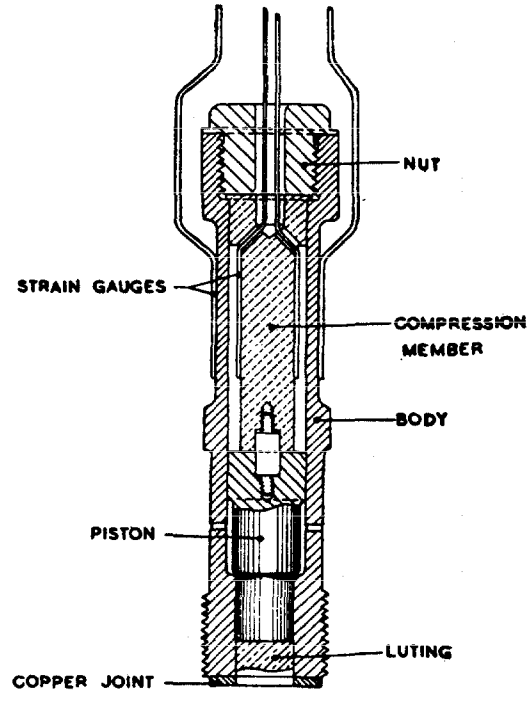
Piezoelectric gages may be used not only to measure the pressure developed in closed bomb or in weapons, but also to measure the blast pressure and pressure developed in recoil mechanisms (Ref 5)

Krause (Ref 16) describes a piezoelectric device, called "carbon piezometer", in which a carbon disc, similar to the one used in telephones, is utilized in lieu of quartz or tourmaline crysts

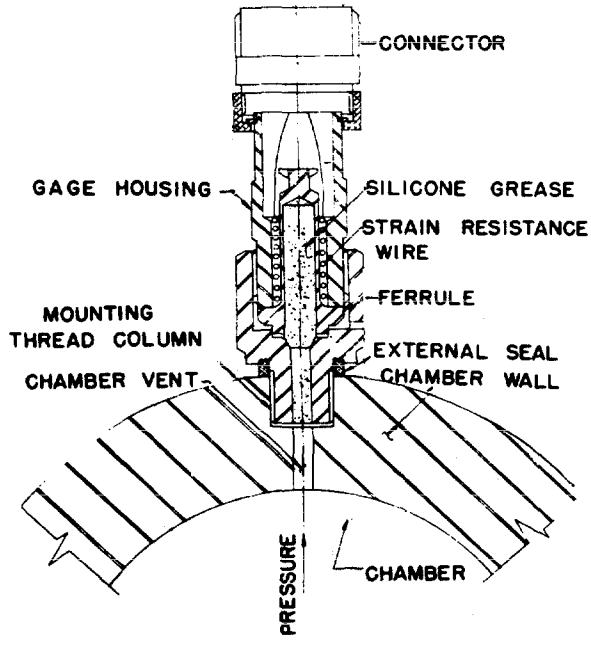
Strain Gages. Essentially, they are based on electrical measurements of strains produced by the action of stress. There are several ways for measuring strains, but the most convenient is the resistance method. The apparatus used for these measurements, known as "resistance manometer', depends upon the change in resistance of a metal (such as platinum, manganin or mercury) when subjected to pressure. The change in resistance is usually measured by means of a very sensitive Wheatstone bridge, but it may also be measured by a potentiometer or by an amplitude modulator. Electric strain gages are suitable not only for measuring the gas pressure developed on expln or deflagration but also the strains produced in materials (such as of gun barrels) on firings

The head of the Brit "strain-gauge", described in Ref 36, p168, is represented here in Fig. It consists of a tubular body threaded at one end to fit a hole in the wall of a chamber. Inside of tube is a closely fitted piston, a compression member (which bears on a nut) and two electric resistance strain gages cemented longitudinally to the compression member. Two more gages are attached outside the body. They serve to measure the difference in pressure between two heads placed in different positions in the chamber. The four gages are connected electrically to form a Wheatstone bridge, energized by a 40 volt battery. The cross-sectional areas of the body of each head and of the compression member are the same, so that the tensile and compressive strains are equal.

On applying pressure to the piston, the resistance of the strain-gage elements changes, giving an electrical output from the bridge



BRITISH STRAIN GAUGE



CROSS SECTION OF STRAIN RESISTANCE WIRE TRANSDUCER PRESSURE GAGE, MODEL C-AN

which can be measured by a galvanometer, or, in case of varying pressures, it may be recorded on a cathode-ray oscillograph

In USA many types of strain resistance wire gages are available; one of them is described in Ref 73, pp6-7, others in Ref 60a. The gage known as the "Strain Resistance Wire Transducer Pressure Gage, Model C-AN" is illustrated here in Fig, pC341-R. This type of transducer consists of tubular housing contg an aluminum or steel ferrule as its mechanical sensor. The ferrule is coated with a thin layer of cement which acts as an insulating and bonding agent for a 0.001" diameter resistance wire wound helically around the ferrule, until the length of wire represents a 500-ohm resistance. When pressure of gases produced on expln or deflagration of a charge inside the chamber is transmitted thru silicone grease to the ferrule, the diameter of the ferrule changes proportionally, and the bonded wire changes length in a similar manner. This length change is, in turn, proportional to a change in the wire's resistance. The ferrule is designed to produce a 4- to 5-ohm change in the wire's resistance at the ferrule's maximum pressure rating. In use, the wire wound on the ferrule comprises one arm of a Wheatstone bridge circuit.

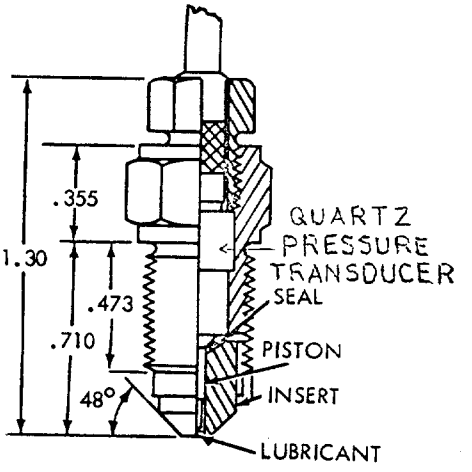
When compressed gases act upon the pressure gage transducer, the bridge circuit is unbalanced in proportion to the pressure. The signal, thus produced, is amplified and fed as a linear deflection into a cathode-ray tube, upon which appear millisecond time marks. The tube face is photographed by a shutterless strip film camera. Another method of recording consists of electronically sweeping the transducer signal across the face of a cathode-ray tube, together with millisecond timing marks. An oscilloscope camera with self-developing paper permits a round-by-round examination of the record. Pressure level indications, covering the expected range of pressures, are provided by calibration steps at suitable resistance values for both methods of recording. When processed, a calibrated pressure-time curve is obtained (Ref 73, pp6-8)

In addn to the above mentioned pressure -measuring devices, the following may be listed:

a) *Baldwin SR-4 Standard Fluid Pressure Cell,* manufd by the Baldwin-Lima-Hamilton Corp, Waltham, Mass. This device, briefly described in Ref 74, consists of a fluid pressure cell which translates changes in pressure into changes in voltage. The sensing element of the cell is a pressure-sensitive tube, to the outside of which are bonded special resistance wire strain gages

b) *Kistler Quartz Pressure Transducer.* It is a small cylindrical body (such as 0.6" high and 0.22" in outside diam), contg an "Inconel" diaphragm and crysts of quartz. Thru the open end of the transducer, the gases of expln act against the diaphragm and the pressure applied to it is converted to a force acting on the transducer quartz crystals. These, in turn, generate an electrical charge output proportional to the pressure output.

Pressure transducers are usually mounted in adapters, such as represented in Fig.



ADAPTER FOR SOME KISTLER QUARTZ PRESSURE TRANSDUCERS

They are inserted in threaded cavities of closed bombs. Adapters are used to facilitate installation, to provide thermal protection to the transducer, and to extend the pressure range of the transducer, or for any combinations of these reasons. When an adapter is not used, the transducer is mounted in an accurately machined cavity, with its diaphragm either flush with the wall of the closed vessel or recessed with a passage connecting to the

vessel (See Fig on p10, Ref 75)

Pressure transducers exist in several models ranging in maximum pressure ratings from 500 to 300000 psi. These devices, although usually restricted to dynamic measurements, are also capable of near static response, provided they are used with Kistler electrostatic amplifiers. Temperature range of transducers is from -400° to +500°F but they can be used for measuring pressure of gases developing temps as high as 3000°F for short periods of time.

The accurate measurement of low level explosive-, blast-, or shock-wave pressures below 10 psi (above atmospheric) requires that the transducer be protected from the severe environment by the use of thermal insulation or other methods. Recessing the transducer slightly and coating the diaphragm with a thin layer of silicone grease prevents flash temps from creating high thermal stresses in the diaphragm (Ref 75, pp1-8 & Fig on p III-601-6)

Several types of Kistler pressure transducers and adapters are used at Picatinny Arsenal (See also Bichel Bomb & Bichel Pressure Gage in Vol 1, pVIII of this Encycl and Chamber Pressure in Vol 2, pC147-R)

(This section was reviewed for technical content by Mr. Lester Shulman of Picatinny Arsenal)

Refs: 1)Marshall **2** (1917), 441-49 (Closed bombs and pressure measuring devices of Rodman, Noble & Abel, Bichel and Petavel) 2)D.A.Keys, PhilMag **1921**, 473-88 (A piezoelectric method for measuring expln pressures) 3)J.C.Karcher, USBurStandards Scient Paper No **445** (Part of Vol 18) (1922) (A piezoelectric method for instantaneous measurement of high pressures) 4)E.Burlot, MP **20**, 255-322 (1923) & **21**, 403-10 (1924) (Measurement of pressures developed in closed vessel on burning of proplnts by methods of Vieille and Burlot) 5)A.W.Ford, Army Ordn **7**, 217 (1926) & MAF **7**, 229 (1928) (Instruments for measuring the pressures at Aberdeen Proving Ground, Maryland) 6)Cranz **3** (1927), 192-257 (Various devices for measuring pressures of expln or deflagration) 7)C.E.Munroe & J.E.Tiffany, USBurMinesBull **346** (1931), pp85-99 (Measurement of pressures and compn of gases developed on expln by means of Bichel Pressure or the Crawshaw-Jones apparatus) 8)A.W.Crow & W.E.Grimshaw, TrRoySoc **A230**, 42-4 (1931) (App for measuring pressure in a closed vessel) 9)H. Joachim & H.Ilgen, SS **27**, 76-9 & 121-25 (1932) (Gas pressure measurements using piezoelectric indicator) 10)Marshall **3** (1932) 133 (Closed bomb and pressure measuring devices) 11)Vennin, Burlot & Lécorché (1932), 72-87 (Closed vessel and various pressure measuring devices used between WWI & WWII in the laboratories of the Commission des Substances Explosives) 12)C. M.Balfour, Engineering **134**, 231-32 (1932) (Pressure measurements in ballistic research) 12a)Sukharevskii & Pershakov (1932) 83-99 (Detn & calcn of gas pressures developed on expln in closed vessel) 13)Stettbacher (1933), 78 (Cylindrical closed bombs for measuring pressure of expln); 91-2, Fig 70 (Pressure bomb of Vieille) 14)Pepin Lehalleur (1935), 82-97 (Closed vessels and pressure measuring devices used in testing of proplnts) 15)H.N.Marsh, ArmyOrdn **17**, 15-17 (1936) (Measurement of cannon pressures) 16)A.W.Krause, ArmyOrdn **18**, 31-3 (1937) (Carbon piezometer applicable to Ordnance) 17)R.H.Kent, ArmyOrdn **18**, 281-84 (1938) (Piezoelectric gage) 18)P. Bernard MAF **17**, 323-45 (1938) (Pressure measurements by piezoelectric gages) 19)H.Muraour, MAF **17**, 579-94 (1938) (Measurements of pressure in closed bomb) 20)Hayes (1938), 92-6 (Pressure measurements) 21)J.Dupuis & M.Chalvet, MAF **18**, 37-69 (1939) (Testing of pressure developed on burning of proplnts in closed bomb of Krupp-Schmitz) 21a)W.F Jackson, "Closed Bomb Method of Powder Testing", DuPont Co, Explosives Dept, Burnside Lab, Memorandum Rept **24** (1941) 22)G.Mueller, PATR **1163** (1942) (Detn of ballistic characteristics of proplnts for large caliber cannons using closed chamber tests) 23)A.Strasser, PATR **1235** (1943) (Studies of characteristics of stick proplnts by closed bomb test) 24)Vivas, Feigenspan & Ladreda **4** (1944), 85-6 (Closed bomb and pressure measuring devices used in Spain) 25)R.E.Dufour, Underwriters' LabsBull Research, No **30** (1944) & CA **38**, 3516 (1944)

(Closed bomb for detn of pressure developed by dust explns) 26)Pérez Ara (1945) 65-68 73-90 (Measurements of gas pressures devel oped on expln) 26a)S.J.Jacobs & W.B. Buck, OSRD Rept **6329** (1946) (Closed bomb burning of HE's and proplnts) 27)J.S. McCorkle, NAVORD Rept **73-46** (1946) (New short-time underwater expln gage) 27a)Muraour (1947), 83-4 & Fig 16 (Bomb of Krupp-Schmitz); 84-90 (Detn of pressure by means of Vieille's bomb) 28)J.F.Moulton & J S.McCorkle, NAVORD Rept **400** (1947) [A short response time mechanical gage (SRT Gage) for measuring underwater expln pressures] 29)P.Slavik, Research **3**, 98-106 (1947) (The wire resistance strain gage) 29a)Muraour (1947), 73-4 (Detn of pressure); 83-92 (Detn of characteristics of proplnts) 30)K.H.Swainger, Nature **159**, 61-2 (1947) (Electric resistance wire strain gages to measure large strains) 31)Cole, Underwater Explosions (1948), 146 -98 [Various devices for measuring underwater explns pressures. Included are: Abbot pellet crusher gage,Schuyler crusher gage, Soft ball (copper) crusher gage, Hilliar piston gage, Diaphragm gages of Modugno and of UERL (Underwater Explosives Research Laboratory at Woods Hole, Mass), British diaphragm gage, known as "pot guage" and several types of piezoelectric gages] 32)H. Muraour & G.Aunis, MAF **22**, 517-93 (1948) and **23**, 859-66 (1949) [Detn of "quickness of combustion" (vivacité de combustion in Fr) of colloidal proplnts in a closed bomb] 33)W. E.Gordon, "Pressure Measurements in Gaseous Detonation by Means of Piezoelectric Gages", pp579-86 in the Third Symposium on Combustion, Williams & Wilkins Co., Baltimore (1949) 34)H. Muraour & G.Aunis MAF **23**, 419-78 (1949) (Investigation of various crusher gages) 35)Corner, Ballistics (1950), 85-7 (Closed vessel and pressure measuring devices used in ballistics) 36)SACMS, Ballistics (1951), 160-72 (Closed vessel work and the measurement of pressure in vessels and guns) 37)H.Muraour & G.Aunis, MAF **25**, 117-65 (1951) (Studies of laws of combustion of colloidal proplnts in a closed bomb using Cu crushers and piezoelectric gages for measuring pressures) 38)M.S. Travers, MAF **25**, 167-73 (1951) (Remarks on the paper of Muraour & Aunis) 39)A.Langevin, MAF **25**, 361-82 (1951) (Detn of pressure in a closed vessel by means of Cu crusher gage and quartz piezoelectric gage) 40)E.Auerbach, PATR **1881** (1952) (Use of closed bomb data for estimating charge weights for 105mm Howitzer) 41)P.Tavernier et al, MP **34**, 237 -53 (1952) (Special apparatus for measuring gas pressure in manometric bomb and in cannon) 42)J.Foster, Jr & R.T.Tassini, Pic ArsnTesting Manual No **3-1** (1952) (C) (Pressure gages for detg pressure in guns, rockets, etc) 43)Anon, Badger Ordnance Works, Project **P-2003** (1953) (Closed bomb instrumentation) 44)H.Muraour & G.Aunis, MP **35**, 287-301 (1953); Explosivst **1954**, 154-57 & **1955**, 6-9 (Studies of laws of combustion of colloidal proplnts in a closed bomb) 45)F.Rössler, ZAngewPhys **6**, 175-82 (1954) (Optical methods used in investigation of adiabatic combustion of solid proplnts) 46)A.O.Pallingston & M.Weinstein, PATR **2005** (1954) [Method of calculation of interior ballistic properties of proplnts (such as relative force, relative quickness and linear burning rate) from closed bomb data] 47)J. Viard & H.Crenn, MP **37**, 319 (1955) (Simple electronic integrator for detn of value $\int pdt$ in closed bomb test) 48)J.H.Whitmore et al, Sunflower Ordnance Works Rept SUN **270-4** (1955) (Ballistic tests using closed bomb) 49)G.Seitz, Explosivst **1955**, 173-78 & 201-06 (Investigation of burning of proplnts in a closed bomb) 50)H.I.Lewis, PicArsnSFAL Rept **424** (1956) (Instrumentation for closed bomb method of proplnt testing 51)A.I.Rubin, PATR **2237** (1956) (Computing linear burning rate of proplnts from closed bomb data by means of analog computer) 52)M.Weinstein & A.O.Pallington, PATR **2240** (1956) (Preliminary study of M2 & M9 proplnts betw 137°F & -65°F, using closed bomb technique) 53)L.Shulman, PATR **2350** (1956) (Use of closed bomb data for assessment of 60mm Mortar Increment Propellant) 54)A.I.Rubin & A.G.Edwards, PATR **2367** (1956) (Use of piezoelectric gages in closed bomb tests of solid proplnts) 55)H.Rumpff, Explosivst **1957**, 43-5 [Differential pressure measurements on burning of proplnts, using an oval-shaped bomb developed in 1953-55 by Rumpff; previously used bombs for this purpose were of Krupp-Schmitz (1913), Liebessart (1921), Thring (1927), Rumpff (1930) &

Zeiss-Ikon (1932)] 56a)Yaremenko & Svetlov (1957), 75-6 (Dolgov's closed bomb) 57)W.Gohlke, "Einführung in die Piezo-elektrische Messtechnik", Akad Verlagsgesellschaft, Leipzig (1958) 58)C.S.Ingram & M.A. Kozak, Sunflower Ordnance Works Report SUN 270-4 (1958) (Design and evaluation of the Sunflower closed bomb recorder) 59)H. Athen, "Ballistik", Quelle & Meyer, Heidelberg (1958), 203-09 (Measurements of pressure developed in guns and closed vessels) 60)W.F.Wallace, PATR **2488** (1958) (New formulas for rapid calcns of linear burning rates of proplnts using info obtd by closed bomb testing) 60a)Anon, "Strain Measurements -Instrumental", Ordnance Proof Manual OPM **80-55** (1958) 61)B.T.Fedoroff et al, PATR **2510** (1958), pGer 28 (Closed bomb testing conducted in Germany during WWII) 62)A.G. Edwards,PATR **2557** (1958) [A method for simulating gun firing using the analog computer and burning rate curves derived directly (by oscilloscope) from closed bomb test] 62a)J.Harris & L.Shulman, PATR **2558** (1958) (Burning characteristics of standard proplnts betw 21° & -51°C) 63)A.J.Cable, AGARD Rept **177** (1958) (Pressure measurements in the Armament Research Establishment) 64)R.E.Kutterer, "Ballistik", Vieweg, Braunschweig (1959), 205-18 (Various pressure measuring devices such as the "Zeiss -Ikon", "Kruppsches Messei" and others) 65)L.E.Smith, Jr, Sunflower Ordnance Works Rept SUN **270-41** (1959) (Ballistic test methods-closed bomb variables) 66)L. Shulman & L.Bottei, PATR **2693** (1960) (Use of closed bomb test methods for assessing 81mm Mortar Propellant) 67)A.Grinoch, Pic Arsn SFAL TechMemo **223-60** (1960) (Frequency response analysis of transducer used to measure pressure in the closed bomb system) 68)K.K.Andreev & A.F. Belyaev, "Teoria Vzryvchatykh Veshchestv" Oboronghiz, Moskva (1960), 432-44 (Pressure of expln and of deflagration) 69)Anon, Ordnance Corps Pamphlet, ORDP **20-106** (1960), Part 1, "Source of Energy" pp2-12 to 2-15 (Detn of pressure in a constant volume chamber); pA-9, Fig A-4 (Pressure vessel for measuring burning rates of propellants as a function of pressure) 70)A.M.Ball, Ordnance Corps Pamphlet ORDP **20-175** (1961), "Solid Propellants", Part 1, p21, Fig 16 (Closed bomb assembly) 71)A.Stanfield, PATR **ND4-TR:2** (1961), (Conf) "Feasibility of using Closed Bomb Evaluation of Rocket Propellants in Ballistic Quality Assurance" (Not used by us as a source of info) 71a)L. Shulman et al, PATR **FRL-TR-41** (1961), "Burning Characteristics of Standard Gun Propellants at Low Temperatures, 21°C to -52°C" (This is the 3rd in a series of PATR's on the closed bomb evaluation of standard proplnts. The first two reports are listed here as Refs 52 & 62b. With the exception of the M-17 proplnt, all US std proplnts tested undergo small changes in burning rate at reduced temps. Single-base proplnts are less temp dependent than double -base proplnts. The maximum pressure of the proplnts decreases ca 7% betw 21°C & -52°C) 72)CSE (Commission des Substances Explosives), Book of Instructions, Fourth Part, Articles 189-210. Can be obtained on request from the Service des Poudres, thru the État Major de l'Armée, 2e Bureau, Paris, France (Detailed description of the French closed bomb test for proplnts known as "Epreuve à la bombe" or "Essai de poudre en vase close") 73)Anon, "Ordnance Proof Manual, Aberdeen Proving Ground, Maryland, Vol 4, **OPM 80-10** (1963), pp 1-10 (Weapon pressure instrumentation) 74)Baldwin-Lima-Hamilton Corp, Waltham 54, Mass (Pamphlet giving operating instructions for "Baldwin SR-4 Standard Fluid Pressure Cell") 75)Kistler Instrument Corp, Clarence, NY (Pamphlet giving operating instructions for Kistler Quartz Pressure Transducers Models 601, 605 and others)

Closed Chamber Test. Same as Closed Pit Test

Closed Loop Test Apparatus for Determining the Heat Transfer Characteristics of High Energy Monopropellants has been described in PicArsnTechMemorandum **1119** (1962), by J.R.Grossman

Closed Pit (or Chamber) Test and Other Fragmentation Tests. A fragmentation test comprises the static detonation of a HE

charge in a projectile, bomb, rocket warhead. grenade, mine or torpedo for the purpose of determining the number, distribution pattern, weight groupings, velocity, and other characteristics of the resulting fragments. The object of the test is to determine the efficiency of an ammo item, or of any of its components

The following four fragmentation tests have been used at Aberdeen Proving Ground (Ref 1): A)Closed Pit (or Chamber) Test B)Open Pit Test C)Panel Test and D)Velocity of Fragments Test, whereas the closed pit test has been used at PicArsn. All of these tests are briefly described below:

A. *Closed Pit (or Chamber) Test.* In this test, an item, placed in a wooden or fiber box, is buried in sand (or fireproofed sawdust) and then detonated. The equipment and procedure used at *Aberdeen Proving Ground* is described in detail in Ref 2, pp 3-4

In Ref 3 are described the equipment and procedure used at *Picatinny Arsenal.* There exist two identical Büchner-funnel-like chambers, called "fragmentation tubs", located in two adjoining buildings. Each tub is made of 1" steel plate. The diam of the tub is 15.4 ft and the depth 14.75 ft. Its bottom opening, 13" in diam, is fitted with a hand-operated door which controls the flow of sand with fragments onto a gyroscopic recovery screen placed below. A magnetic separator, designed to recover the fragments that are too small to be recovered by gyrosifter, is placed below it. The equipment, except the magnetic separator,is shown in the Fig, pC347

Before each test the tub is cleaned and filled to about 1/3 with washed & dried sand or with sawdust (previously fireproofed by spraying it with a soln consisting of water 85, borax 7, boric acid 3 & diammonium phosphate 5%, followed by drying). On top of sand (or sawdust) is placed a square box made of soft wood 1/2" thick contg the unfuzed projectile (or other item) to test set on its base in the center of the box. The internal dimension of the box should be equal to 3-times the diam of projectile. The fuze is then fitted to the proj and its electrical wires connected to the blasting circuit. Then the top of the box is fastened by means of two small nails and the sand (or sawdust) is slowly poured from the storage hopper, located above the tub, until the wooden box is covered to the depth required for the size of item to be fragmented. After the personnel leave the bldg, the explosive in the item is detonated by manipulating an electric switch located outside the bldg. Then the personnel, equipped with respirators, re-enter the bldg and begin recovery of fragments by opening the door at the bottom of the tub and allowing the sand (or sawdust) to go thru gyrosifter and magnetic separator. The fragments are recovered and classified by weighing and grouping accdg to the following weight zones (in grains): Zone0–0 to 75; Zone 1–76 to 150; Zone 2– 151 to 750; Zone 3–751 to 2500; and Zone 4– 2501 and over

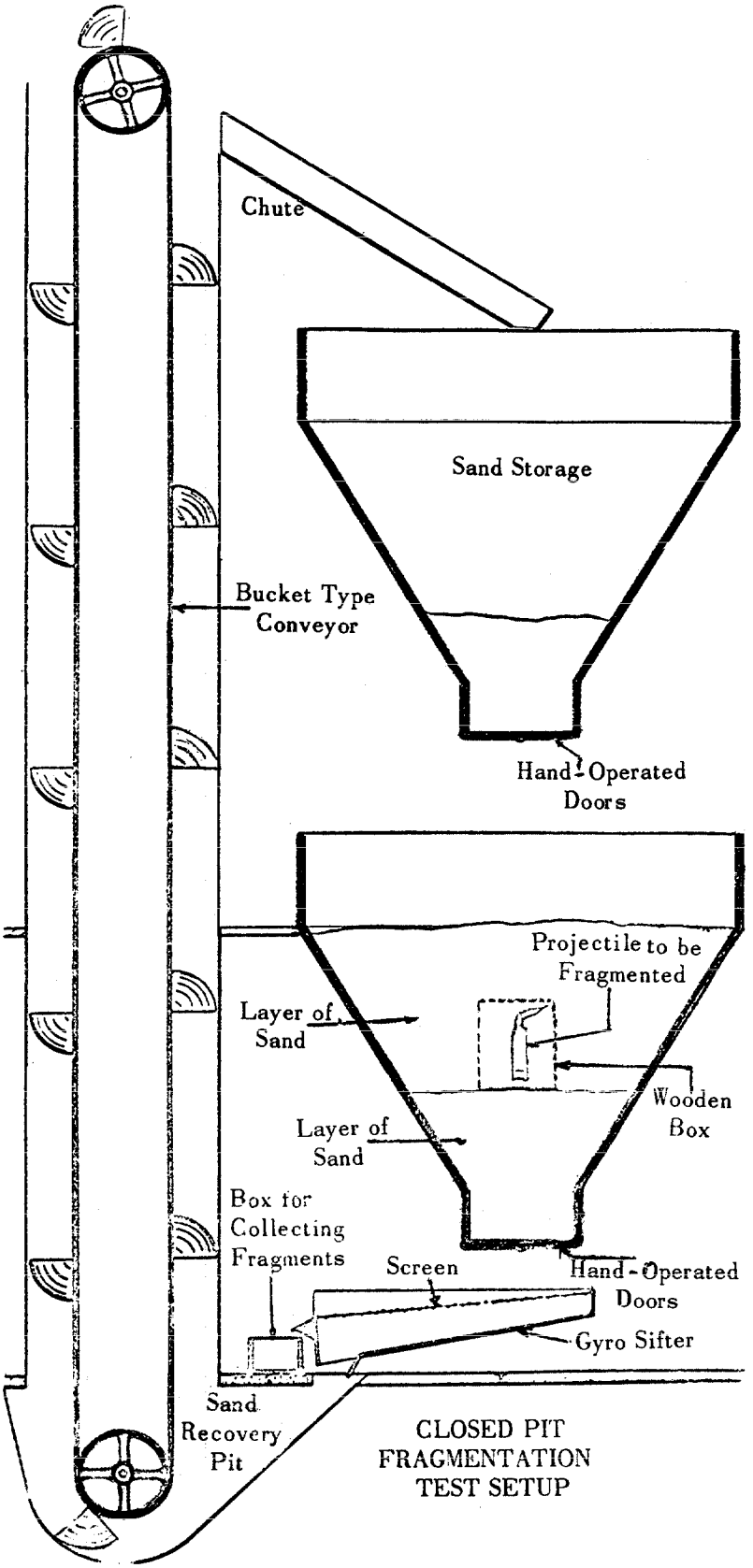
After weighing and classifying the fragments they are placed, arranged by zones, on a white flat surface and photographed

As an example is attached a photograph of fragments produced on bursting of 155mm HE Shell, M101 (Ref 3). See pC348

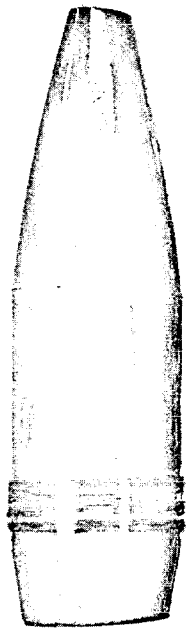
Accdg to Ref 5, the closed pit test is somewhat unreliable because of loss of very small fragments, and because of secondary break-up of fragments by the effect of surrounding sand

B. *Open Pit Test.* This test is designed for the purpose of recovering a portion of the fragments of detonated bomb in such a manner that they will be segregated into groups which correspond to various areas of the bomb -body, nose or tail

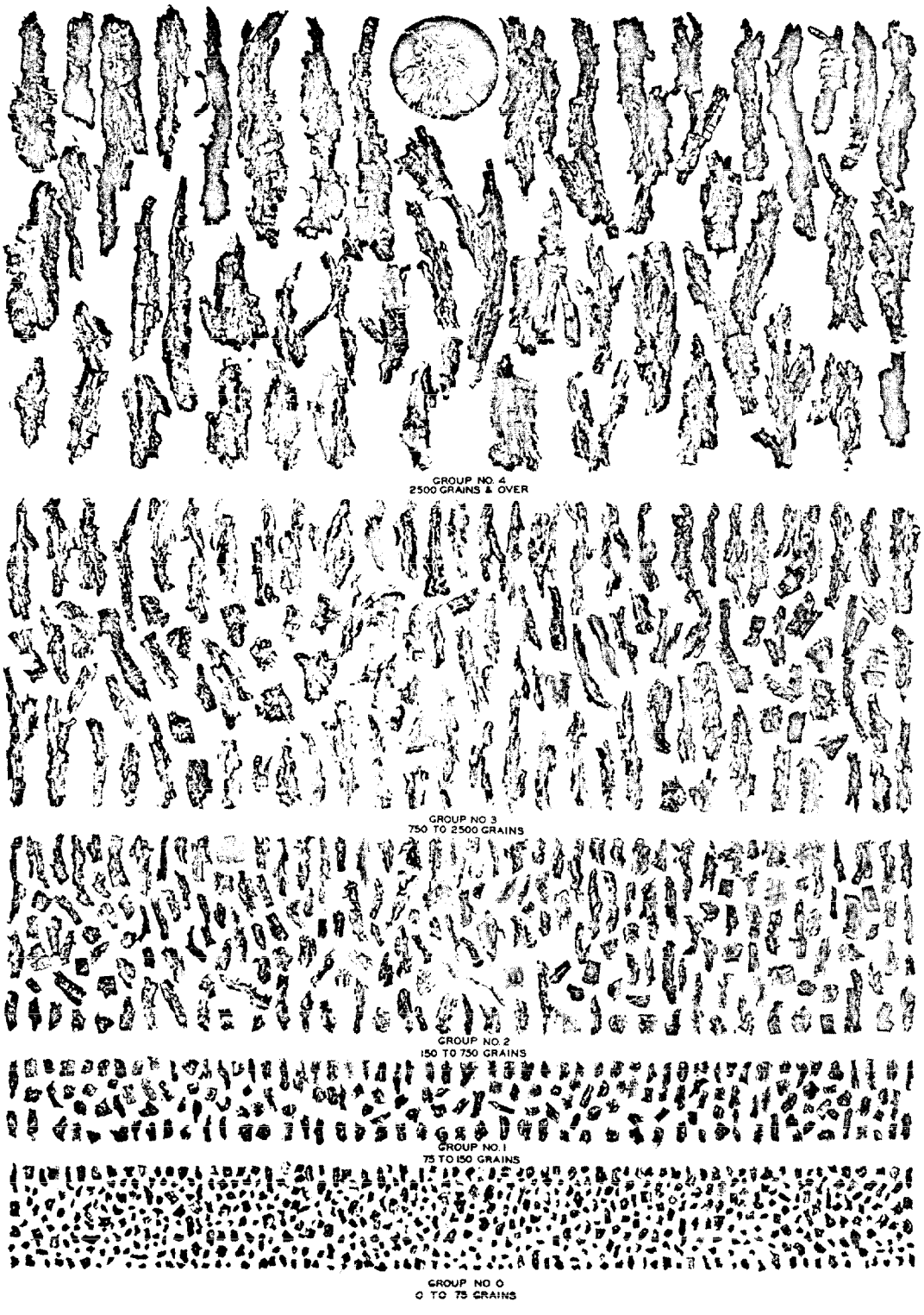
The open pit is a large rectangular box, 12' x 16'10" inside dimensions. Its bottom is 2" thick and walls are constructed of two thicknesses of 6" beams. The box is divided by 2" boards into four compartments with inside dimensions 12 ft long, 4 ft wide and 2 ft deep. The box is buried in the earth with its top flush with the ground and filled with clean, dry sand to a height 12 to 24" depending on the size of the bomb to test. The sand should be heaped along the perimeter and the partitions of the box. Then the box is covered with a fiber-board 6" thick and the unfuzed bomb is suspended by its lugs from a wooden gallows at a specified height directly over the center of the pit. The bomb is usually suspended horizontally, parallel to the long



CLOSED PIT FRAGMENTATION TEST SETUP



UNFRAGMENTED SHELL



FRAGMENTATION OF A 155MM HE SHELL, M101

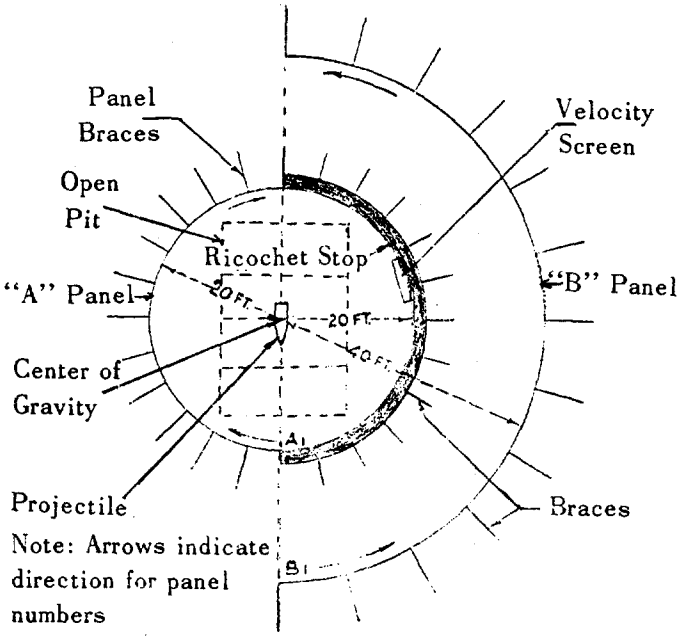
axis of the pit, but when analysis of nose or tail fragments is required, the bomb may be suspended vertically

After giving signal warning and seeing that all personnel are under cover, the bomb is fuzed, its leading wires connected to a blasting device, situated 2000-3000 ft from the pit, and the charge is detonated. Then the sand is sifted and the fragments recovered

In the open pit test only a small portion of the fragments hit the pit area, but since the angle subtended by the pit and the height of suspension are known, it is possible to compute all the info that can be obtained in a closed pit test

Open pit test may be conducted in conjunction with panel and fragment velocity tests (Ref 2, pp4-6) (See Fig under Panel Test)

C. *Panel Test.* This test may be used for either bombs or projectiles. The panel setup for testing bombs consists of one solid, horizontal, semicircular arc located at a prescribed distance from a bomb (5 to 160 ft depending on the wt of the expl charge). The arc is constructed of Douglas fir plywood panels, 4' wide & 8' high mounted vertically on a wooden framework, held by braces. The angle subtended by the arc should be 5 degrees greater than 180 degrs to assure complete coverage of the nose and tail fragments. The bottom edges of all panels should be at least 6" above the ground, but the height of 24" is preferable. The panels should be nailed lightly to frame, since with rigid fastening the panels might split by the force of shock wave. If fastened lightly they might become dislodged but not damaged so that they can be remounted and re-used



PANEL TEST SETUP

For testing projectiles, two semi-circular arcs are used, the arc B having a radius twice that of the arc A (See Fig). These arcs have a common center and they face each other. Their central angle is of 180 degrees. The panels for testing projs are constructed from No3 white pine boards (dressed), 1" thick, mounted vertically upon the framework

When a bomb or projectile is detonated, virtually all the fragments striking the ground at an angle of 10 degrees or less will ricochet and considerable number will ricochet up to an angle of 15 degrs. In order to avoid overestimating the number of fragments produced in a panel test, ricocheting fragments are prevented from striking the panels by use of a baffle, known as "ricochet stop" (See Fig of Panel Test Setup)

This consists of two concentric semicircular plywood walls, with the space betw them (ca 2' wide) filled with earth. Its top is made to slope away from the center of setup so that a line joining the lower of the panel passes just above and parallel to the top of the ricochet stop. In case of testing a proj, the radius of ricochet stop is about half of the panel B radius

After the panels are set in place, the bomb or proj is suspended (with fuze removed) from gallows, at a prescribed height. The center of gravity of the item should be in the center of panel setup. After the area is cleared of personnel and the warning signal given, the item is fuzed and the round is detonated, by closing the elec circuit of fuze. Then the panels are examined and the fragments intercepted by each panel are counted. The fragments may either indent (penetrate), or pass completely thru the panel (perforate). Penetrations measuring less than 0.25" in depth are usually not recorded (Ref 3, pp5-9) (See also items E, F & G)

As was mentioned above, this test, as well as open pit fragment velocity tests, can be run simultaneously (See Fig under Panel Test)

D. *Fragment Velocity Test.* The purpose of this test is to determine the velocity of fragments resulting from the detonation of a bomb or projectile. This is accomplished at Aberdeen PG by the use of Aberdeen Chronograph. This instrument is briefly described in this Encyclopedia, under CHRONOGRAPHS,pC304-R

In fragmentation test a chronograph with six or eight channels is employed; two channels leading to the item to test, the remainder to one or more velocity screens. These should be mounted parallel to the major axis of the bomb or projectile with their centers in line with the center of gravity of item to test. The position of the screens should be such that they will be protected from ricocheting fragments. Each screen consists of a rectangular wooden frame, usually 36" by 72", covered in the back with galvanized window screening, while the front is covered with four sections of the same screening, which are insulated from each other. These screens are, in turn, insulated from the back screen by means of waxed paper, or by another dielectric. The chronograph, located 2000 to 3000 ft from the item to test, is placed under a bombproof cover.

For conducting this test, the bomb or proj is suspended, unfuzed, on the gallows in the same manner as in conducting the open pit or panel tests, previously described. An electric insulated wire, of the length equal to slightly more than the distance between chronograph and item to test, is folded in the middle and its loose ends are connected to two posts of the chronograph. The other folded end of the wire is wrapped, in the shape of a loop around the item to test. After connecting the other chronograph posts to the screens, the testing area is cleared of personnel and the item is fuzed and detonated. This breaks the wire wrapped around the item,thus causing a spark to jump from one of the spark points of chronograph to the rotating drum, puncturing the record strip and leaving a mark. The 2nd mark on the strip is made when one of the fragments penetrates one of the screens, thus establishing the contact betwn the front and back screening wires. By measuring the distance betw two spark perforations and knowing the linear speed of the record strip, the time of flight of fragment betw the item and screen may be detd. From this, knowing the distance of travel of fragment, its velocity at the midpoint is detd (Ref 3, pp 9-10)

The procedure for combined open pit-, panel- and fragment velocity testing is given in Ref 3, pp10-11

E. *Low Panel Test.* This test is similar to a certain extent to that described as item C. Here, four cylindrical quadrants of wooden panels surround the bomb at distances from it of 10, 20, 30 & 40 meters. Each panel is 7/8" thick, 12" wide and 5.57 ft high to simulate the size of a man. A bomb is detonated statically and the number of fragments passing thru the panels at different distances is counted and the distribution of fragments is recorded (Ref 1, p213)

F. *Silhouette Test.* In this test a bomb is dropped into the center of a series of wooden boards or panels arranged in open rectangular formation, 15 by 45 ft, and the number of fragments passing thru the panels is detd. The panels are of the same size as in the previous test. The number and distribution of fragments are recorded (Ref 1, p213)

G. *High Panel Test.* A brief description of this test, as given by Ohart (Ref 1, p213), is so vague that it is impossible to understand how it is conducted

H. *German Fragment Density Test* (Splitter-dichteprobe in Ger). In this test, invented by Dr. G. Römer, the expl item to be tested is detonated in the middle of wooden boards, 2cm thick, surrounding it as shields at a known distance. The number of fragments per sq meter piercing the boards is counted and the average distance at which there would be one fragment per sq m is calcd from a specially constructed curve. In order to obtain reliable results it is necessary to detonate at least 10 items to test. Following are some values for the average distance to obtain one penetration per sq m using a 105mm shell: TNT 39-40m, 50/50 Amatol 35m, 50/50-TNT/NaCl 26m & 40/60-TNT/NaCl 23m (Ref 4, pGer 32)

Refs: 1)Ohart (1946), 213 2)Anon, "Fragmentation Tests for Bombs and Projectiles",

Ordnance Proof Manual, OPM **40-23** (1947) Aberdeen Proving Ground, Md 3)J.H.McIvor, Picatinny Arsenal Testing Manual, No **5-1** (1950) 4)PATR **2510** (1958), pGer 52 5)Glossary of Ord (1959), 73 6)PATR **2700** Vol 1 (1960), pXII

Closed Vessel Test. See Closed Bomb (or Vessel) test, etc

Cloth, Cartridge. See under Cartridge Bags in Vol 2, pC77-L and in OrdTechTerm (1962), 73

Cluster. A collection of small bombs (held together by an adapter), or a grouping of two or more rocketengines fastened together as a single propulsion unit

A cluster of fragmentation, gas or incendiary bombs is usually so arranged that more than one bomb can be suspended and dropped from a single station of the bomb rack of an airplane

Refs: 1)Ohart (1946), 241 2)OrdTechTerm (1962), 74 3)US Military Specifications. See Index, Vol 2 (1958), 54-5

Cluster Adapter. In order to be able to carry more aerial bombs in a given space of a bomber, the bombs are assembled in clusters and held together by a special mechanical device called "cluster adapter". In the so-called *aimable* cluster adapter, the bombs can be aimed before dropping and this permits confining the dispersion of bombs to a smaller target area than in the case of *nonaimable* adapters (Compare with Aimable Cluster described in Vol 1, pA114-L)

Refs: 1)Ohart (1946), 241 2)OrdTechTerm (1962), 4

Clydite. A coal-mine expl formerly made by Nobels at Ardeer. It was similar to *Nobel Carbonite* (NG 26, K nitrate 29, Ba nitrate 4, woodmeal 40.5, "sulfurated benzene" 0.25 and Na & Ca carbonates 0.25%), except that K nitrate was replaced by Ba salt, and it could contain up to 8% Amm oxalate

The same replacement could be made in the low-freezing *Nobel Arctic Carbonite*, which contained NG 15.5, nitro-hydrocarbon 10.5, K nitrate 42.0, woodmeal 31.7 & Ca carbonate 0.3%

Ref: Marshall, Dict (1920), 19 & 22

CMC. Designation for **Carboxymethylcellulose, Sodium Salt,** known also as *Sodium Cellulose Glycolate,* Rcell. $OCH_2.COONa$; colorless, odorless and tasteless, non-toxic, hygroscopic substance, easily dispersible in hot or cold w, but insol in most organic solvents. Can be prepd by treating alkali cellulose with Na chloroacetate:

Rcell ONa + $Cl.CH_2.COONa \longrightarrow$ Rcell.$O.CH_2.COONa$ + NaCl, where Rcell means radical of cellulose. A side reaction, the conversion of monochloroacetic acid to glycolic acid occurs simultaneously. The compd has been commercially available in the US since 1945 (Ref 1), but in Europe it had been used for several years before, chiefly in wallpaper paste as a thickener (Ref 2). It is now used as a substitute for some natural water-sol materials, such as Na alignate, Irish moss, gelatin & tragacanth gum; also as an adhesive, water-binder, suspending agent, protective colloid, etc (Refs 2 & 3). It has also been used as the granulating agent (crystal growth control agent) in the prepn of crystalline, non-dextrinated LA. US Military requirements and tests for CMC are given in Spec MIL-S-51132(MU) of 1964. The first US Spec [MIL-L-46225A, "Lead Azide RD-1333" (1963)] describing LA prepd by pptg it from CMC was issued as classified, although this LA was reported in an unclassified report by R. L. Wagner [PATR **2662** (1960)], and in this Encyclopedia Vol **1** (1960), pp A558-A559. The most recent Spec MIL-L-46225B (June 1964) describing LA RD-1333 has now been issued as unclassified.

Refs: 1)Hercules Powder Co, "CMC. A Water-Soluble Cellulose Gum", Wilmington, Del (1945) 2)Kirk & Othmer **3** (1949), 385-7 3)CondChemDict (1961), 1037

Note: CMC can probably be used as a combustible coating or binding agent (in lieu of starch, etc) in some powdery dynamites

CN, CNB, CNC and **CNS.** US Military designations for the Chemical Warfare Agents,

briefly discussed in Vol 2, p C167-L

Coad Explosives. Blasting expls, such as: a)NG 75, K nitrate 5 & woodmeal 20% b) NG 30, K nitrate 50 & woodmeal 20% c) NG 30, BkPdr 50 & woodmeal 20%
Ref: Daniel (1902), 146

Coagulation of Nitrocellulose Solutions. NC solns may be coagulated by the addn of non-solvents or suitable electrolytes. According to Apard (Ref 1), the addn of finely ground PbO, CaO or $Ca(OH)_2$ to a 1% soln of NC in acetone, eth-alc or amyl acetate causes first an increase in viscosity & then gelation. Morozov (Ref 2) reports that NC solns can be coagulated, in the presence of small amounts of alkali, by boric acid & some metal oxides. With oxides of Cu, Fe, Mn, Ni & Zn the resulting gels pass into soln on standing; with Pb or Al oxides syneresis of the gel occurs. Papkov & Chveleva (Ref 3) studied the coagulation of acetone solns of NC by aqueous $ZnSO_4$, $MgCl_2$ & $AlCl_3$

Gloor & Spurlin (Ref 4) obtd clear green solid gels on addn of small amts of bronzing powder & copper salts to NC solns. The higher the molecular wt of NC, the smaller was the amt of gelling agent reqd to produce a strong gel (Ref 9). Pamfilov et al (Ref 5) showed that when treating NC solns with metals and their salts, the time reqd for gelation to occur was longer for the more stabilized NC. Nitric acid & nitrogen oxides tend to accelerate gelation. The gelation of butyl acetate-alcohol solns of NC by antimony oxide, titanium oxide & others was studied by de Waele (Ref 6). According to Vodyakov (Ref 7), the amt of metal in NC gels obtd by absorption of metallic oxides or hydroxides, or by electrolysis of an acetone soln of NC, is approx proportional to the N content of the NC. [See also Cellulose Nitrate, Action of Solvents (Swelling, Gelatinization, Solubility and Plasticization), Vol 2 of Encyclopedia, pC115-R, under Cellulose and Derivatives]

Zenftman & Forlin (Ref 8) patented fuse compns, employing a plastic binder, that could be continuously extruded provided a special antihardening agent was included in the formulation. One such compn was composed of: fuse powder (Pb_3O_4 41, KNO_3 33 & Si 26%) colloided NC (NC 50, DBuPh 48 & DPhA 2%) 20, and antihardening agent (citric acid, $KHC_2O_4.H_2O$ or Butyl Cellosolve)0.4%
Refs: 1)A.Apard, CR **186**, 153 (1928); MP **23**, 63 (1928) & CA **22**, 1511 & 3525 (1928) 2) I.R.Morozov, ZhKhimProm **8**, 863 (1931) & CA **26**, 296 (1932) 3)S.Papkov & M. Chveleva, KollZts **73**, 202 (1935) & CA **30**, 929 (1936) 4)W.E,Gloor & H.M.Spurlin, JACS **58**, 854 (1936) & CA **30**, 4742.(1936) 5)A.V.Pamfilov et al, KollZh **4**, 569 & 587 (1938) & CA **33**, 6119 (1939) 6)A.deWaele, JOil & Col **22**, 57-72 (1939) & CA **34**, 3077-78 (1940) 7)L.T.Vodyakov, TrudyKazanskago-Khimiko-Tekhnologicheskago Instituta Imeni S.M. Kirova **8**, 61 (1940) & CA **35**, 2715 (1941) 8)H.Zenftman, & J.E. Forlin, USP 2590060 (1952) & CA **48**, 6700 (1954) 9)H.M.Spurlin in Ott, Vol 5, pg 3 (1954), p1087ff

Coal. (Called Houille in Fr, Steinkohle in Ger, Carbone fossile in Ital, Carbón de piedra in Span, and Kamennyi Ugol' in Rus) **And Coal Mining.** Coal is a combustible solid, usually stratified, which originated from partially decompd vegetation in previous geologic ages. Decompn takes place in absence of air, in presence of moisture and in some cases under pressure & at high temperatures. It is composed chiefly of carbon, hydrogen & oxygen, with minor amts of nitrogen & sulfur, and varying amts of moisture & inorganic impurities; however it is not a hydrocarbon. There is a wide range in the compn and the physical & chemical props of different kinds of coal
Historical (See Refs 1, 3, 20 & 28). It was claimed that the Chinese used coal as early as 1000BC, but there is no documentary evidence. More certain it is that coal was known in the Greco-Roman World ca 2000 years ago. During Roman occupation of England, coal was mined on a small scale as early as 400AD. Primitive mining of coal began in Germany ca the 10th century. One of the first documents on mining of coal in Europe waswritten ca 1200AD by a Belgian monk, Rainier. The mineral coal was called in Fr *"charbon de roche"* (rock charcoal) to distinguish it from "charbon de bois" (wood

charcoal) As early as the 13th century coal started to be mined on the southern shore of Firth of Fourth (Scotland) and then at Newcastle and the Northumbrian Coast. As Brit coal was always shipped by sea, it was known in Europe as "sea coal"

As the coal mined in early days on the British Island was mostly bituminous, it emitted on burning noxious gases and smokes which was not the case with charcoal used extensively at that time. As long as supply of timber (from which charcoal was made) was abundant, coal was not a desirable fuel, especially in large cities where contamination of air should be avoided

The importance of coal started to be recognized when it was tried as a fuel in brick ovens. An extensive use of coal began in the middle of the 17th century when Engl ironmaster Dudley established several iron smelting works for which coal was more suitable than charcoal

The first coal deposits on the North American continent were discovered ca 2nd half of 17th century in Nova Scotia and ca 1701on the banks of James river, near Richmond, Virginia. It was not until 1745, however, that coal started to be mined in Va on a commercial scale. About 1751 coal was discovered in West Virginia (Appalachian bituminous),in 1755 in Ohio and in 1770 in Pennsylvania

Up to the time of American Revolution, the production of coal in Amer colonies was very small because a large quantity of it was imported from England or Nova Scotia. As a great shortage of coal developed during the Revolution, the local production was increased and a search for new coal fields was spurred. As result of this search, coal was discovered near Frostburg, Maryland (1804), where it started to be mined on a large scale during the construction of turnpike from Cumberland to Wheeling (1814). Prior to this (1810), large scale mining of coal began near Pittsburgh, Pennsylvania. By the early 1830's many small mining companies were in existence along the Ohio, Illinois and Mississippi rivers, and in the Appalachian region. In 1840 1 million tons were mined and from then on, the growth of production was steady until it reached a maximum of over 600 million metric tons in 1947. After this, however, the production of coal started to decrease in USA, while in other countries it continued to increase. The production of coal in 1960 (in million metric tons) was (accdg to Ref 28, Vol 5, p974), as follows: USA 400, GtBritain 200, Russia 513 (Against 166 in 1954), China 420 (Against 130.7 in 1957), E. Germany 228, W. Germany 240, Poland 114 and the World 2500

Classification, Properties and Uses of Coal. Coal is marketed in lumps, black or brownish in color, and in a wide range of sizes. Because of the technical and commercial importance of coal, many systems of classification have been proposed. Years of work by a technical committee of US and Canadian representatives resulted in the adoption in 1937-39 of "American Standards" for coal classification which are now widely accepted and used

Classification by **type** differentiates the coals accdg to the proportion of various plant ingredients and accdg to appearance. *Standard types* used in US include: *common banded, splint, cannel* and *boghead* coals.

Classification by **rank** serves as a measure of their degrees of coalification and of their heating values, as expressed in BTU/lb. When arranged in increasing C content and BTU value, they are as follows: *lignitic, subbituminous, bituminous,* and *anthracitic* coals (Ref 26, D388-38; Ref 28, Vol 5, p962)

Classification by **grade** covers the quality of the coal as detd by size, proximate analysis, ultimate analysis, ash-softening temp, ability to carbonize, calorific value, etc (See Ref 26, D339-37, p 1232)

A different classification is that developed by the US Geological Survey and US Bureau of Mines (See Ref 1, p343)

Following are the definitions of the above terms: *Common banded* is the common variety of bituminous and subbituminous coals; it consists of a sequence of irregularly alternating layers of lenses of a homogeneous blk material having a brilliant vitreous luster; a grayish-blk less brilliant striated material usually of silky luster; and thin bands or lenses of soft powder and fibrous particles, known as *fusain* or *mineral charcoal.* The difference in luster of the bands is greater

in bituminous than in subbituminous coals (Ref 3, p114 & Ref 26, D493-39). *Splint* is a variety of bituminous & subbituminous coals, commonly having a dull luster and grayish-blk color; it is of compact structure and often contains a few thin, irregular bands with vitreous luster (Ref 3, p115 & Ref 26, D493-39). *Cannel* is a variety of bituminous or subbituminous coal of uniform and compact fine-grained structure in which bands are absent (Ref 3, p115 & Ref 26, D493-39) (See also Vol 2, pC25-R of this Encyclopedia). *Boghead* is a coal resembling cannel in appearance and in behavior during combustion; it is characterized by a high percentage of algal remains and volatile matter. Upon distn it gives very high yields of tars and oils (Ref 3, p115 & Ref 26, D493-39). A dynamite in which boghead was used as an adsorbent for NG is listed in Vol 2, pB220-L of this Encyclopedia. *Peat* is essentially the first stage in the development of coal by partial decomp in water of various plants, especially of mosses. The ASTM does not list peat as a coal, but US Geological Survey and US Bur Mines do. Pressed into briquettes and dried, peats are used as fuels. Russia is one of the biggest consumers of such fuels. Dried and pulverized peat has been used as a fertilizer and as an ingredient of some commercial expls (Ref 1, p337; Ref 10a; Ref 24, p854 & Ref 28, Vol 17, p426.) *Lignitic* is a variety of coal intermediate betw peat and subbituminous coal; it is usually brownish -blk in appearance and contains much volatile matter and 30 to 40% moisture. It is subdivided into *lignites* which are consolidated clay-like materials and into *brown coals* which are unconsolidated friable materials in which the texture of original wood is distinctly visible. Lignite coals have been used as fuels, some of them in the form of briquettes or powdered. The BTU/lb value of moist material is ca 8300. Some commercial expls contained lignitic coals as ingredients (Ref 1, p338; Ref 10, p337; Ref 24, p666; Ref 26, D388-38; and Ref 28, vol 14, p117). *Subbituminous* is a variety of coal intermediate between lignitic and bituminous,usually has a glossy blk color and pitchy luster. Its moisture content is 15 to 30% and BTU/lb value 8300 to 11000. Used for the same purposes as lignitic coal (Ref 24, p1084; Ref 26, D388-38; Ref 28, vol 5, p962); *Bituminous* (also known as "soft coal") is a variety of coal, intermediate betw subbituminous and anthracitic; it is usually blk in color with vitreous luster; fixed carbon content (see its definition under Coal, Analytical Procedures) varies betw 69 & 78% and volatile matter betw 22 & 31%; BTU/lb value runs betw 11000 & 14000; it is most abundant variety of coal; used as a fuel, coke production, manuf of producer gas, illuminating gas & fuel gas (Ref 1, p333; Ref 3, p108; Ref 24, p157; Ref 26, D388-38; Ref 28, vol 5, p962). Uses of bituminous coals as ingredients of expls are discussed under Coal Dust and Its Uses. *Anthracitic* (also known as *hard coal*) is a variety of coal contg the highest percent of fixed carbon (86 to 98%) and very little of volatile matter; it is blk in color with a brilliant luster; it burns with a short blue flame giving off very little smoke or odor; used as household & industrial fuel, for manuf of producer gas & water gas and in metallurgy (Ref 1, p344; Ref 3, p107; Ref 24, p91; Ref 26, D388-38; Ref 28, vol 5, p962)

Other props of coals, such as specific gravity, relative friability, decomposition temperature, specific heat, thermal conductivity, etc are given in Ref 3, pp88-91

Toxicity and fire and expln hazards of coal are discussed in Ref 18

Coal is consumed in greater tonnage than any other commodity produced by man. Nearly all coal is utilized by high-temp reactions. In the US about 80% is burned directly for the generation of electricity, steam power for transportation, industry & heating and most of the remainder is carbonized to produce coke, coal gas, ammonia, coal tar, chemicals, etc. Some coal is hydrogenized to obtain liquid fuels (see also COAL PROCESSING FOR OBTAINING MORE VALUABLE PRODUCTS)

Coal dust or powdered coal has been used for prepn of "colloidal fuels" (qv), as a fuel in special burners and as a component of some commercial expls (See Coal Dust and Its Uses)

Explns caused by coal dust present in coal mines are discussed under "COAL MINE EXPLOSIONS AND FIRES"

Coal Mining. Early methods of coal mining were very primitive and breaking of rock and coal was done by striking with a pick and hammer without the use of BkPdr, which was the only known explosive at that time. Blasting of coal with BkPdr did not begin until early 19th century, although it was used earlier for blasting rock (Ref 20, pp18-19)

The early coal workings were shallow, comprising either a short vertical shaft which was belled at the bottom, or a vertical shaft and short horizontal galleries. The shallow workings were usually more troubled by the presence of carbon dioxide (called "chokedamp") than of methane (called "firedamp") and they depended on natural ventilation. With the expansion of coal industry in England, which took place during the reign of Elizabeth I (1558-1603), the digging started to go deeper and deeper underground. At the beginning of the 18th century, some coal mines attained the depth of 300-360 ft, although the common depth of shafts was only 120-180 ft. As the depth of mines became greater, the natural (free) ventilation became poorer and accumulation of "firedamp" more frequent. This required an "artificial ventilation". This problem is briefly discussed under "COAL MINE EXPLOSIONS AND FIRES"

Accdg to Ref 28, Vol 5, p967, the av depth of Brit mines is now ca 1100 ft, although some mines in Lancashire and Staffordshire are more than 4000 ft deep. The av thickness of workable coal seams is ca 4ft in Gt Britain, ranging from 1 to 40 ft. Some coal seams in China are as thick as 400 ft. Accdg to Ref 3, p105 a large majority of the US mines are producing coal from beds 3 to 6 ft thick

Due to the fact that the coal mining industry is one of the largest consumers of explosives, it is advisable to give a brief description of methods of mining

Majority of the larger coal mines are now mechanized, which means that they are equipped with power drills, power coal cutters, power slicers, power loaders, narrow gage RR tracks and trains, etc (Ref 20, pp9-15; Ref 28, Vol 5, pp966-968). Automation in coal mines is briefly discussed in Ref 2, vol 5, p970

The preliminary operation in *underground mining,* known also as *deep mining,* is designed to reach the coal layer (seam). This can be done, as practiced in Gt Britain, by means of either a *shaft* (a vertical opening driven by mechanical means thru the rock to the seam); *slope* (an opening driven at an angle 15-25° horizontal to the seam); or *drift* (an opening driven horizontally from the outside to the seam). The last method can only be used if the seam is exposed at the surface elevation ("outcrop") or has been exposed by stripping away the rock above the coal. Many mines employ the combination of these and all mines must have minimum of two access openings to facilitate the circulation of air thru the mine and to provide alternate ways of escape in case of emergency. The next step, if using the method known as the *advancing longwall mining method,* the whole of the seam within the specified area (panel) is removed in one continuous operation by taking successive slices of the entire length of a long face, which is usually 800 to 1500 yds long (Ref 28, vol 5, pp965-67)

In the method usually practiced in US mines, a series of large tunnels (called rooms) are driven thru the coal seam in two directions at right angles, so dividing it into a number of blocks. Sufficient pillars of coal are left to support the overlaying coal. Then the roof above the seam is made safe by timbering or rock bolting and one or more slots - a few inches wide and extending several feet into the solid coal - are cut along the length of coal face by a large mobile circular power saw. The cut (or slot) provides a free face and facilitates the breaking up of the coal subsequently blasted by explosives from the seam. Next step is to drill the holes by power drills in which the expl charges are placed. Special expls, known as "permissible", are used to break down coal (See under COMMERCIAL OR INDUSTRIAL EXPLOSIVES), but there is a method of mining coal without using expls.

For this, steel cartridges, several feet long, filled with air compressed to 20000psi or with liquid carbon dioxide, are placed in long, narrow holes and then the gas is suddenly released with practically expl violence, thus breaking the coal into large lumps. After breakage the coal is loaded directly into track-mounted mine cars or into rubber -tired electric trucks known as shuttle cars. Sometimes it is loaded directly into chain- or belt-conveyers. After the coal is loaded into shuttle cars or conveyers in the production area, it is transferred to the main haulage system, which consists either of rails or conveyers

After the coal is mined, the miners retreat while removing coal comprising the pillars. This usually causes the collapse of rock strata above the seam, thus leaving an underground area, known as *gob*, which is not safe to enter.

The above described US method of mining is known as *conventional* or *cyclic* (Ref 28, vol 5, pp966-67)

There is also a *continuous* method which was introduced in 1940. Here a single machine known as "continuous miner" breaks the coal from the seam, without using expls, and transfers it to the haulage system. Several types of these machines were developed, some of them capable of mining more than 5 tons per minute (Ref 28, Vol 5, p967)

Underground coal mining in countries other than USA or Gt Britain is briefly described in Ref 28, Vol 5, pp967-70

In addition to the above discussed *underground* method, there is also the *surface method*, known as *strip mining* in the USA and *opencast mining* in Gt Britain. In this method, the overlaying strata are removed by bulldozers and the exposed coal is broken into lumps by blasting and loaded into trucks. As the work is conducted in open, no ventilation is required and the expls used for blasting coal do not need to be "permissible". Taylor & Gay (Ref 20, p145-51) describe this method as practiced in Gt Britain, and EncyclBritannica (Ref 28, Vol5, p967) describes it as practiced in USA

Unfortunately some states (such as Pennsylvania) did not require that the trenches, left after removal of coal, be filled with earth and leveled, thus restoring the land to its original condition. As a result of this, large sections of land in US are shamefully disfigured by mounds of earth which run alongside the open trenches

In addn to *underground* and *strip mining* a new method, known as *auger mining* was developed after WWII. The method consists of boring, by means of an instrument resembling an auger, a series of parallel horizontal holes into the coal seam which has been exposed by "outcropping" (coming out of the stratum to the surface) or by "strip" mining. The method is often used when, during strip-mining operations, the thickness of overburden becomes too great for further strip-mining. In this case the "augering" machines, powered by internal combustion engines, drill from the open pit horizontal holes 2 to 5 ft in diam at depths up to 300 ft in the coal seam. The coal so removed is transferred to an elevating conveyer and loaded into trucks. No removal of overburden, no roof supports, no drilling of boreholes and no blasting are required with this method. Augers have now been adopted for use in some underground mines (Ref 28, Vol 5, p967) (See also Coal Dust and Its Uses; Coal Gasification, Underground; COAL MINE EXPLOSIONS; COMMERCIAL OR INDUSTRIAL EXPLOSIVES; COAL PROCESSING FOR OBTAINING MORE VALUABLE PRODUCTS; Coal Tar; and Coke)

Refs: 1)Thorpe **5** (1941), 333-59 (Fuels) 2)H.H. Lowry, "The Chemistry Of Coal Utilization", Wiley, N.Y. (1947) (2nd printing) 3)Kirk & Othmer **4** (1949), 86-134 (Coal) 4)E.S. Moore, "Coal, Its Properties, Analysis, Classification, Geology, Extraction, Uses and Distribution," Wiley, N.Y. (1950) 5)P.J. Wilson, Jr. & J.H. Wells, "Coal, Coke and Coal Chemicals," McGraw-Hill, N.Y. (1950) 6)D.R. Mitchell, Edit., "Coal Preparation," AmInstMining & Metallurgical Engrs, N.Y. (1950) 7)P.C. Pope, "Coal Production, Distribution, Utilization", Chapman & Hall, London (1950) 8)Kirk & Othmer **6** (1951), 893-913 (Fuels) 9)I.C.F. Statham, Edit, "Coal Mining", Engl Univs, London (1951) 10)Kirk & Othmer **8** (1952), 339-46 (Lignite and brown coal 10a)Kirk & Othmer **9** (1952) (Peat)

11)Ullmann, **4** (1953), 612-47 (Braunkohle) 12)A. Lissner & A. Thau, "Chemie der Braunkohle", W. Knapp, Halle (1953) 13)W. Gumz & R. Regul, "Die Kohl", Verlag Glückauf, Essen (1954) 14)E. Graf, "Technologie der Brennstoffe", F. Deuticke, Wien (1955) 15)N. Nedelmann, "Kohlechemie", West-Verlag, Essen (1957) 16)D.W. van Krevelen & J. Schuyer, "Coal Science", Elsevier, Amsterdam (1957) 17)Ullmann **9** (1957), 626-37 (Kohle) 18)Sax (1957), 490 19)Ullmann **10** (1958), 579-81 (Kohlenextraktion) 20)Taylor & Gay (1958), 1-17 (History of coal mining in Gt Britain); 145-51 (Opencast coal mining); 152-65 (The mechanism of blasting coal and rock by means of expls) 21)McAdam & Westwater (1958), 95-135 (Blasting in collieries); 162-65 (Blasting in opencast workings) 22)Anon, "The Metallurgical, Chemical, and Other Uses of Coal", Natl Res Assocn for Bituminous Coal, Pittsburg (1958) 22a)Blasters' Hdb (1958), 333-46 (Bituminous coal mines); 397-412 (Strip-mining of coal) 23)W. Francis, "Coal, Its Formation and Composition", Arnold, London (1961) 24)Cond-ChemDict (1961), 157 (Bituminous or soft coal); 161 (Boghead); 212 (Cannel); 283(Coal); 666 (Lignite); 854 (Peat); 1067 (Splint); 1084 (Subbituminous) 25)D.W. van Krevelen, "Coal; Topology, Chemistry, Physics and Constitution"; Elsevier, Amsterdam (1961) 26)ASTM Stds 1961, Part 8, Method **D388-38**, pp1227-37 (Classification of coals) 27)H.H. Lowry, Edit, "Chemistry of Coal Utilization", Pittsburgh Coal Research Center, USBurMines (1963) 28)Encycl Britannica **5** (1963), 961-75 (Coal and coal mining); Ibid, **9** (1963), 891-95 (Coal and related fuels); Ibid **14** (1963, 117 (Lignite); Ibid **17** (1963), 426-27 (Peat)

Coal, Analytical Procedures. The most frequently made analysis of coal is the *proximate analysis.* This includes detns of moisture, volatile matter, fixed carbon and ash. Sometimes it is combined with detn of sulfur and phosphorus, which really belongs to the *ultimate analysis,* and includes also detn of carbon, hydrogen, nitrogen and sulfur. In most cases, the heating value of coal expressed in BTU/lb is also detd.

Briefly, the *moisture* is detd by heating a 1g sample, spread on the bottom of a capsule, in an oven at 104-110° to constant wt; *volatile matter* represents the loss of wt when 1g of coal is heated in platinum crucible for 6 mins at 950°; this leaves *coke* as a residue; *ash* is detd by igniting and burning a 1g dry sample in a crucible to constant wt; *fixed carbon* is detd from the formula: 100 - (% moisture + % vol matter + % ash); *sulfur* is detd by the Eschka or other methods; *phosphorus* is detd in ash, as described in Ref 3, pp1159-60; carbon & hydrogen are detd by burning a weighed sample in the combustion tube and fixing the products of combustion in an absorption train after complete oxidation and purification from interfering substances; *nitrogen* is usually detd by the Kjeldahl -Gunnig method and *oxygen* by subtracting from 100 the sum of the percentages of hydrogen, carbon, nitrogen, sulfur, moisture and ash. A detailed description of proximate and ultimate analysis is given in Refs 1, 2 & 3

The method of test for gross calorific value of coal by adiabatic bomb calorimeter is described in Ref 3, pp1177-87 and method of fusibility of coal ash in Ref 2, D1857-61T and Ref 3, pp1170-77

Refs: 1)Kirk & Othmer **4** (1949), 91-4 & 119-23 2)ASTM Stds **1961**, Part 8, Methods **D271-58, D980-53, D1757-60T** and **D1857-61T** 3)Std Methods Chem Analysis **2A** (1963), 1137-1257 (In most cases the methods are the same as in ASTM Stds)

Coal, Anthracitic. See under COAL

Coal, Bituminous, See under COAL

Coal, Boghead. See under COAL

Coal, Brown. See under COAL

Coal By-products. See under COAL PROCESSING

Coal, Cannel. See under COAL and in Vol 2 of Encyclopedia, pC25-R

Coal Carbonites. Blasting expls suitable for coal mining: a)NG25.0, Na nitrate 30.5, K dichromate 5.0 & rye flour 39.5% and b) NG 30.0, Na nitrate 24.5, K dichromate 5.0 & rye flour 40.5%

Ref: CondChemDict (1942), 288 (Not found in later editions) (See also in Vol 2 of Encyclopedia: Blasting Explosives, p B202-L; Carbon Carbonite, pC60-L; and Carbonites, p61-R)
[Compare with Ger Kohlensprengstoffe (Coal Explosives), such as Kohlencarbonit, Kohlenkoronit III, Kohlensalit, Kohlenwestfalit I, Kohlenwestfalit IV and Kohlenwestfalit V, listed in PATR **2510** (1958), pp Ger 101-02]

Coal Carbonization. See under COKE

Coal, Common Banded. See under COAL

Coal Dust and Its Uses. Coal dust (or powdered coal) can serve as a fuel when using special burners installed in steam boilers, stationary or ships or locomotives. It can also be used in metallurgy, and as a component of so-called *colloidal fuel*(qv). It can be compressed (using a small amt of binders) into brickettes or egg -shaped pieces ("boulets" in Fr) for use as a solid fuel (Addnl Refs A to I incl)

When coal dust is mixed with oxygen (or air) in certain propns, the mixt can be ignited and exploded by means of an outside flame or spark. This property was attempted to be utilized in Germany during WWII by Dr Zippermeyer in the so-called "explosive powered vortices". He proposed to fill a projectile with powdered coal and to place in the center of the filler a charge of low explosive, such as double-base "noodle" proplnt (Nudelpulver in Ger). With this system the coal dust would have on initiation of low expl a forward component of velocity due to the motion of translation of the projectile, and a lateral component of velocity due to expln of low expl. A high expl cannot be used in such projs because it would impart too much lateral velocity and scatter the coal dust too widely. As the diffused coal dust mixes with the air in the presence of the explosive charge, which acts as an igniter, a coal-dust expln results. In his experiments Dr Z shot the proj from a mortar tube which was buried in the ground and with velocity of proj of at least several hundred meters per second, he succeeded to produce a considerable vortex effect. This proj was intended to be used for breakingoff the wings of low-flying planes, because it is known that a wing can be damaged even by a comparatively low pressure if it persists for one tenth of a second (Ref 4, pp183-84). This device is also described in Ref 6, pGer45-L and in Ref 7, p214, under the title "Luftwirbelkanone"

The possibility of producing expl energy by the rapid combustion of coal dust is briefly discussed by Reich in a letter to the editor of C&EN (Ref 5). Reich states: "Of far greater importance, however, than the use of coal as a source of controlled power is its potential use as a military expl. When coal is finely powdered and dispersed as an aerosol in oxygen, its combustion occurs with explosive speed and is capable of causing heavy destruction over a radius of from several dozen to several hundred meters from ground zero. This introduces possibilities of controlled bombardment of small precisely defined areas. Such weapons can be used by individual soldiers at close quarters. Although military security does not permit disclosure of the sizes of weapons, methods of triggering, etc , it is clear that this development will revolutionize warfare" (Compare with COAL DUST BOMB)

Coal dust (sometimes designated simply as "carbon") has also been used in many commercial expl compns as for example: *Ammonal* (Austrian) (See this Encycl, Vol 1, pA290, Table); *Ammonal* (Italian) (Vol 1, pA291-R); *Ammonal* (Spanish) known as *Amonal 1* (Vol 1, pA292-L); *Ammoncarbonit 1* (German) (AN 80.3, K nitrate 5.0, NG 4.0, collodion cotton 0.2, coal dust 6.0 & starch or flour 4.5%) (Ref 6, p Ger 5); *Ammonium Nitrate Explosives* (Coal as well as charcoal are used in some compns) (Ref 3, p336); *Ammonium Nitrate Explosive V* (AN 66.0, Na or K chloride 22.0, powdered coal 4.0, cereal- or woodmeal 2.0 & NG 4.0%) (Ref 3, p352); *Bogbead Dynamite* (Vol 2, p B220-L); *Cannel Explosive* (Vol 2, pC25-R); *Detonit VI or 14A* (AN 82.0, NaCl 10.5, woodmeal 2.0, coal dust 0.5, NG 4.0 & MNN 1.0%) (Ref 1 p435); *Dinamony* (Rus mining expls consisting of AN and fuels such as bituminous coal, lignite or peat; Eg: *Dinamon "T"* (AN 88 & peat 12%)(Ref 5a); *Dynammon* (AN 47.2, NA 45.0 & lignite 7.8%) (Ref 2, p289); *Dynammon I* (AN 88 & lignite 12%) (Ref 2 p289); *Judson Powder* [Low-grade dynamite prepd by coating 85-95 parts of "dope" (prepd by mixing powdered coal, Na nitrate and sulfur) with 15-5

parts of NG] (Ref 3, p334); *Neu-Nobelit XV* (Gelatinized NG 12.0, woodmeal & coal 4.0, nitrocompds 2.0, AN 54.0, & alkali chloride 28.0%) (Ref 1, p441); *Wetterammoncahüsit A* (AN 64.7, TNT 8.0, coal 2.0, woodmeal 1.3, gelatinized NG 4.0 & NaCl 20.0%) (Ref 1, p438); *Wetterammoncahüsit B* (AN 67.0, DNT 6.0, coal 1.5, woodmeal 1.5, gelatinized NG 4.0, & NaCl 20.0%) (Ref 1, p438); *Wetterastralit A* (AN 57.0, gelatinized NG 12.0 coal powder 2.0, woodmeal 2.0 & NaCl 27.0% (Ref 1, p 442); DNT 2.0, woodmeal 2.0, coal powder 0.5 & NaCl 33.5% (Ref 1, p442); *Wetterbaldurit A* (AN 82.0, gelatinized NG 4.0, MNN 1.0, woodmeal 2.0, coal 0.5 & KCl 10.5%) (Ref 1, p436); *Wetterdonarit A* (Same compn as of Wetterdetonit A) (Ref 1, p436); *Wetterfördit A* (AN 75.0, gelatinized NG 4.0, woodmeal 2.0, coal 0.75 & KCl 18.25%) (Ref 1, p437); *Wettersalit A* (AN 55.0, gelatinized NG 12.0, coal 3.0, woodmeal 2.0 & KCl 28.0%) (Ref 1, p442); *Wettersigrit A* (AN 57.0, gelatinized NG 12.0, coal 2.0, woodmeal 2.0 &KCl 27.0%) (Ref 1, p442); *Wetterwestfalit B* (AN 82.0, gelatinized NG 4.0, coal 1.5, woodmeal 1.5 & KCl 11.0%) (Ref 1, p437)

See also *Coal Dust* under **Coal Mine Explosions and Fires**

Refs: 1) Naoúm, NG (1928), 436-38 & 442 2) CondChemDict (1942), 288-89 (Not found in later editions) 3)Davis (1943), 334, 336 & 352 4) Simon (1947), 183-84 5)I. Reich, C & EN **32**, 4046 (1954) 5a)Yaremenko & Svetlov (1957), 149 6) PATR **2510** (1958), p Ger 5 & Ger 45 7)Lusar (1958), 214 8)H.H. Lowry, Edit, "Chemistry of Coal Utilization", Pittsburgh Coal Research Center, USBurMines (1963)

Addnl Refs: A)R.M. Bridgwater, Petroleum (London)**7**, 211 (1944) & CA **40**, 4870 (1946) (Brit research on petroleum substitutes: Pulverized coal as Diesel fuel) B)J. Commissaire, Chaleur et Industrie **28**, 137-52 & 187-94 (1947) CA **41**,7073 (1947) (Utilization of powdered coal as a fuel) C)Kirk & Othmer **4** (1949), 126 & 407 (Uses of finely pulverized coal in dynamites, as fillers for various purposes and as a fuel either in pulverized form or compressed into briquettes) D)A. Fitton, InstPetroleum Review **3**, 18-26 (1946) & CA **45**, 7771 (1951) (Discussion on the history of the use of pulverized coal and the possibilities and problems involved in its use in present-day industry) E)B. Ghosh & A.A. Orning, IEC **47**, 117-21 (1955) (Igniting pulverized coal when used as a fuel) F)Ullmann **9** (1957), 626-37 (Coal dust and its utilization) G)P.O. Rosin, JInstFuel **21**, 346-55 (1958) & CA **52**, 16721 (1958) (Mechanism of combustion of pulverized coal) H)H. Toulmin, Jr, USP 2938782 (1960) & CA **54**, 18304 (1960) (Mixts of coal dust with powdered metals, such as Al, as fuels for blast furnaces) I)G.G. Thurlow & others, JInstFuel **33**, 366-76, 377-85, 386-99 & 399-402 (1960); CA **54**, 21703-05 (1960) (Summary of research carried out on pulverized-fuel flames by the International Flame Research Foundation)

Coal Dust Bomb. A bomb, which could be suspended from a parachute and activated by a barometric device, was patented in Germany before WWII. The bomb contained a load of coal dust & of propellant and was provided with a priming device

Ref: K. von Haken, GerP 680483 (1939) & CA **36**, 2147 (1942)

Coal Dust and/or Firedamp Explosions. See under COAL MINE EXPLOSIONS

Coal Dust; Explosion Hazards from its Uses are briefly discussed in the following Refs:

Refs: 1)D.J. Price & H.H. Brown, "Dust Explosions", NatlFire-Protection Assoc, Boston (1922), 264pp 2)L.D. Tracy, US BurMines Bull **242**, 103pp (1926) & CA **20**, 1524 (1926) (Expl hazards from the use of pulverized coal) 3) S.K. Shaw & D.W. Woodhead, Safety in Mines Research Report No **131** (1956) & CA **50**, 16110 (1956) (Expln hazards from localized deposits of coal dust) 4)N. Brunzel, MittVerGrosskesselbesitzer, No **42**, 179-201 (1956) & CA **50**, 17377 (1956) (A review with 20 refs covering fundamentals of coal dust explns) 5)H. Hanel, Technik **11**, 785-92 (1956) & CA **51**, 6157 (1957) (Flammability and expln danger of dusts, including coal dust) 6)Sax (1957), 490 (Expln hazard slight when exposed to flame and fire hazard, moderate when exposed to heat; can react with oxidizing materials)

Coal Gas. See under COAL PROCESSING

Coal Gas Explosions. See Firedamp Explosions, under COAL MINE EXPLOSIONS

Coal Gasification Underground In this process, coal is not blasted and removed from underground, but is transformed "in situ" into gas, which is brought to the surface to serve either for heating or illumination. Although this process has been known for nearly a century, it is not much used, except in Russia

Four processes used after WWII in Russia are described by Clendenin (Ref 1). The following method is discussed in Thorpe (Ref 2):

Two inclined shafts are driven down to the coal seam and are connected by horizontal gallery. A small fire is started at the base of one of the shafts and an air blast (sometimes enriched with oxygen, sometimes contg steam) is forced underground from the surface along one of the inclined shafts. The gas generated by the action of this mixture on hot coal is forced to the surface thru the 2nd shaft

Several methods of gasification were patented outside of Russia. For example, in the process patented in USA (Ref 5) elec current, passed betw electrodes introduced into underground coal seam, generates enough heat to carbonize part of coal and creates low resistance path. Continued heating causes direct gasification of part of the fuel, and subsequent introduction of air permits recovery of addnl gasified products

Other methods of underground gasification of coal are given in Refs 3, 4, 6, 7, 8 &9. Two small books on this subject published in Russia are listed as Refs 6 & 9

American experiments in Alabama coal fields are discussed in Ref 7

Underground gasification of coal has the advantage of being labor-saving and of permitting the utilization of coals not easily won by ordinary mining methods. Some mines with thin seams of coal, considered to be uneconomical to work by blasting coal, might find underground gasification as an economical method

Refs: 1)J.D. Clendenin, ChemEngrg Progress **43**, 581-84 (1947) 2)Thorpe **9** (1949), 182 3) Svenska Skifferolje AB, GerP 937842 (1956) & CA **53**, 11806 (1959) 4)Y. Sugino, Netsu-Kanri (Heat Engineering), Tokyo **8**, No 9, 33-8 (1956) & CA **53**, 18441 (1959) 5)E. Sarapuu, USP 2795279 (1957) & CA **51**, 12472 (1957) 6)I.P. Zakutskii & O.V. Kruglov, "Podzemnaya Gazifikatsiya Kamennykh Uglei v Donbasse" (Underground Gasification of Coals in Donbass), Gos-Nauch-Tech Izdat, Lit po Ugol'Prom, Moskva (1957) 7)J.L. Elder et al, "Field-scale Experiments in Underground Gasification of Coal at Gorgas, Alabama Using Electrolinking Carbonization as a Means of Site Preparation", US Bur Mines Rept Invest **5367** (1957) 8)C.A. Masterman & P.E. Montagnon, BritP 807195 (1959) & CA **53**, 10711 (1959) 9)G.D. Lidin, "Sovremennyiye Metody Degazatsii Ugol'nykh Plastov" (Modern Processes for Gasification of Coal Beds), InstGornago Dela, Akad Nauk, Moskva (1960)

Coal Hydrogenation. See under COAL PROCESSING

Coalite (Explosive). Accdg to Ref 1, "Coalite" was an Engl name for the Ger expl *Thunderit*, which was admitted to England and used in coal mines. Its compn was: AN 91-93, TNT 3-5 & flour 3-5%, with moisture not higher than 0.5%. Accdg to Ref 2, "Coalites" were Amer permissible expls contg AN and NG.

Refs: 1)Daniel (1902), 146 & 767-68 2)Marshall, Dict (1920), 23

Coalite (Fuel). Accdg to Ref, "Coalite" is a smokeless fuel made by heating bituminous coal in a retort until much of the volatile matter has been given off

Ref: Merriam-Webster's (1961), 432

Addnl Refs: A)I. Miagawa & M. Hayakawa, ResReptNagoya-IndSciResInst No **1**, 21-3 (1949) & CA **48**, 10322 (1954) (Coalite, together with tar & volatile products, was prepd by low temp carbonization, up to 560°, of lignites) B)A. Nagaya, JapP 1127 ('50) & CA **46**, 7744 (1952) (Prepn of picric & oxalic acids and of other products by treating "Coalite" with nitric acid) C)T. Fushimi, JapP 7678 ('51) & CA **47**, 5098 (1953) (Dry distn of coal at temps up to 600° using alkaline substances as catalysts produced 80% Coalite & 8% tar)

Coal, Lignitic. See under COAL

Coal Liquefaction. See under COAL PROCESSING

Coal Mine Explosions and Fires. Explns in coal mines can be produced by ignition of either combustible gases, or coal dust, or

both, if they are present in atmosphere of mines in quantities sufficient to form expl mixts with air. Combustible gases present in coal mines are known as "firedamp", but this name is also used to designate mixts of combustible gases with air (Ref 30)

Coal Mine (or Pit) Gases. A combustible mixt of gases formed as result of the evolution (and possibly of decomposition) of volatile components of coals, especially of bituminous types. The main component of these gases is methane, CH_4, known as "swamp gas" and its mixt with air is called *firedamp* (Grisou in Fr or in Ital; Schlagwetter, Dampf, Grubengas or Wetter in Ger; Grisú in Span; and Gremuchii- or Rudnichnyi gaz in Rus). Other constituents of pit gas are: *whitedamp* - carbon monoxide, CO, which is combustible and extremely toxic; *stinkdamp* - hydrogen sulfide, H_2S, which is combustible & toxic; and *blackdamp or chokedamp,* carbon dioxide, CO_2, which is non-combustible and toxic when inhaled in large quantity

Mine atmospheres contg 5 to 15% of methane and no appreciable amt of carbon dioxide are explosive, and of these the most dangerous is the mixt contg 9.5% methane. Temp of expln of this mixt is ca 650° if duration of flame produced by a charge of expl used for blasting coal is ca 10 secs and if it is only 0.1 sec, the temp is ca 1000° (Ref 9, p222; Ref 31, p971)

Ignition and expln of combustible mine gases may be produced not only by the flame resulting from blasting with expls which are not safe to use in fiery coal mines, but also by other factors. The flame produced on expln is known as "primary or detonation flame." The so-called "secondary or after flame" which arises from the combination of reaction products of primary flame (such as of CO) with surrounding oxygen in the air, is also dangerous. Other causes of ignition include: any naked flame present in mine; hot or inflamed particles ejected from the expl; adiabatic compression of the gas by a shock wave at supersonic velocity; general adiabatic compression of a body or pocket of gas; electric sparks or sparks produced by drills hitting stones (which are sometimes embedded in coal) (Refs 7b, 7c, 16a, 22 & 31)

The presence of flammable gases in mine atms is very undesirable (not only because they might cause an expln, but also because they are toxic) and the bulk of them should be constantly removed from mine by means of efficient ventillation. If the content of methane is reduced to 1-2%, the atm may be considered non-flammable. The gas can also be removed (as it is often done in Europe) by drilling long boreholes in the strata ahead of the working face and piping the gas to the surface, where it can be utilized as a fuel. The calorific value of pure methane is ca 1012 BTU/cu ft (Ref 31)

Coal Dust (Poussière de houille in Fr; Steinkohlenstaube in Ger; Polvere di carbone fossile in Ital; Polvo de carbon de piedra in Span; and Kamennougol'naya pyl' in Rus). It is extremely finely pulverized coal (particle size below 5 microns) formed during extraction of coal from mines by cutting, drilling and blasting operations. This dust is harmful to the lungs and men operating drills, etc must wear masks

Accdg to Ref 9, p222, coal dust alone when present in air in quantities 300-600g per cu meter is dangerous, but not as much as methane -air mixts. If, however, dust is mixed with methane, then atms contg as little as 3% methane become expl. Kirk & Othmer (Ref 17 p309) state that flammability of a coal dust depends on several factors, including compn, particle size and concn. Dusts contg volatile matter (such as from lignitic and bituminous coals) are more expl than those from coals poorer in volatiles (such as semibituminous, anthracite or coke). Small particles are more liable to ignite, because of their greater surface area per unit of mass. As a limit of dust concn below which its mixt with air would not explode, K & O give for bituminous coals 0.003 to 0.080 oz/cu ft and for lignitic coals 0.04 to 0.065

In mines particularly dangerous on account of presence of methane and/or coal dust, only compressed-air powered machinery is permitted (Ref 31)

(See also Coal Dust and Its Uses)

Historical. Although some rather minor mine explns took place from time to time when coal started to be produced from underground mines (See COAL, Historical), no serious losses in life or property occurred as long as the mines

were not deep and coal was broken by pick or hammer w/o the use of expls. All early mine explns were caused either by open flames of illuminatingdevices (such as tallow candles or oil lamps) or by sparks produced by metallic tools striking rocks. One of the early explns (1648 at Harraton, England) was very disastrous, because 50 persons were killed

More frequent explns started to occur towards the end of the 17th century and miners, themselves, devised the following safety method. One of the miners covered himself with wet sackcloth and creeped on his stomach into gallery where firedamp existed. He held in front of him a long pole with several lighted candles at its end. The flame of candles ignited the firedamp and produced a light expln. As the flame ran along the roof of the gallery and the miner remained lieing on the ground, there was no danger for him. This operation was done after every prolonged stoppage of work in the mine

Despite these precautions, another big expln took place in 1705, near Gateshead, where more than 30 lives were lost. The depth of that mine was 180 ft

Use of BkPdr for blasting coal did not start until the beginning of the 19th century although it was used for breaking rock as early as 1630 (Ref 20, p18). As the flame produced on deflagration of BkPdr can ignite firedamp, the explns in coal mines became more frequent than when coal was worked with pick and hammer. As explns caused by BkPdr are very difficult to eliminate (except by very efficient ventilation), efforts were made to eliminate the hazard of open-flame illuminating devices. For this it was required to invent a "safety lamp" - one which would not ignite combustible gases or coal dust. One of the first of these lamps was that of W.R. Cluny, invented in 1813. This and the lamp of G. Stevenson, invented early in 1815, were not successful but they served as prototypes for the lamp of Sir Humphry Davy, developed at the end of 1815. The latter lamp proved so successful that its improved versions are still in use in smaller mines, mostly in countries other than USA. They are also used for the detection of firedamp or of lack of oxygen in mine atmosphere (See Coal Mines; Determination of Firedamp, etc)

Davy lamp, also known as "safety (or miner's) lamp", is an oil lamp built on the idea that if the flame is surrounded by a cylinder made of metallic gauze with 28 openings per linear inch, the products of combustion would be cooled on passing thru the gauze to a temp below ignition points of flammable gases on the other side of the gauze. In order to avoid danger resulting from failure of a single gauze cylinder surrounding the flame, Davy installed two concentric cylinders, one slightly smaller than the other. The upper parts of the cylinders are covered by disks made of the same gauze while the lower edges fit snugly to the fuel container. This lamp is safe to use except in a strong air current because of the danger of passage of the flame thru the gauze

Beginning early in the 1900's, portable electric hand- and cap lamps were introduced, but it was not until about 1920's when they became widely used. The most popular of these is the cap lamp connected by flexible, rubber insulated, cables to a battery attached to the belt of the wearer. They furnish a powerful beam of light and are provided with a safety device that shuts the electric current if the bulb is broken. These lamps, however, give no warning of presence of noxious gases or lack of oxygen as do Davy lamps; and for this reason each mine must be provided with several Davy-type lamps to serve as detectors (See under Coal Mines; Determination of Firedamp etc). US usage based on federal & state laws, recommends that a minimum of two Davy-type lamps be available in each mine for testing the presence of firedamp or oxygen deficiency

In addn to Davy-type and portable electric lamps, electric lighting from power mains may be used underground, provided adequate safety features and care are exercized in their use. The so-called "electropneumatic lamps" are extensively used in Europe (Ref 32, p809)

Another problem which caused lots of difficulties in underground coal mines was *ventilation*. At first, when the mines were not deep, "natural (or free) ventilation" thru shafts (See also under COAL; Mining) was sufficient, in most cases. As the mines

went deeper underground, the natural ventilation was improved by placing coal-burning furnaces in the shafts in order to rarify the air and thus produce upward movement of mine atmosphere. Although this method was of considerable danger in mines rich in firedamp, it was not until the beginning of the 19th century that high-pressure steam jets were introduced as an alternative to the furnace. This method was not used for a long time and was eventually replaced, beginning in 1849, by mechanical ventilation. This method has been gradually improved and is now used in all coal mines (Refs 23 & 31) (See also Ref 28)

Although many disastrous explns took place in coal mines ever since they started to go deeper and deeper underground, no studies of their causes and methods of their prevention (except ventilation) were made until the beginning of the 19th century. At that time all explns were attributed to the presence of firedamp, and coal dust was not considered as dangerous. The first indication that coal dust is flammable (and therefore dangerous) was made by M. Faraday ca 1845. He stated that expln of firedamp would raise coal dust from the floor, walls & ceiling and then ignite it if sufficient oxygen were present to support the combustion (Refs 12 & 31). Very few investigators took notice of Faraday's statement and the dust was still not considered to be dangerous. The experiments conducted in France beginning in 1867 by Verpilleaux showed that coal dust can be ignited and exploded not only when in mixts with methane & air but also in mixts not contg methane or other combustible gases. His findings were not definitely established until 1875, but even then some scientists, among them Mallard & Le Chatellier, still insisted that coal dust-air mixts are not expl. In the decade 1875-1885, Brit, French & Ger investigators: Galloway, Abel, Atkinson, Vital, Hilt & others, conducted tests with coal dust-air mixts either in specially built galleries or in abandoned mines. Investigations conducted in England by the Royal Commission on Accidents in Mines were summarized in a Report published in 1886 by HMSO (London). It stated that different coal dusts are flammable and consequently dangerous in varying degrees, but no dust is entirely free of risk. Experiments conducted in 1889-1891 by the Austrian Commission on Firedamp, at Mährisch-Ostrau Testing Gallery (now in Czechoslovakia, known as Moravska -Ostrava) showed that nearly all kinds of coal dust can be ignited by a 100g cartridge of dynamite freely suspended in dusty atmosphere

As some mining engineers and colliery officials were still unconvinced of the danger of coal dust, maintaining that previous experiments did not represent practical conditions, another (British) Royal Commission was appointed in 1891. After considerable investigation & inquiry, this Commission published in 1894 a report which contained conclusions similar to those of the 1886 report (Ref 12, p109)

The years 1906-08 marked a new era in the investigation of coal dust explns. Systematic, large scale experiments were begun in Gt Britain & in France and on a lesser scale in Austria & in Germany. Eventually interest was aroused in the US and, after a preliminary study of European methods, comprehensive investigations were begun in 1911. The work in Europe was interrupted by WWI, but was resumed afterwards (Ref 12, pp109-11)

As result of these investigations, the following explanation of coal mine explns seems to be plausible: In mines contg both firedamp and coal dust, the gas is ignited & exploded first and the flame ignites and explodes the dust if there is enough oxygen left in the atmosphere. The combined expln wave is propagated further and further thruout the mine. This happens because the wave is preceded by a rush of air which stirs up the dust and so prepares the way for the expln. The finer the dust and the richer it is in volatile components (such as in bituminous coal), the easier it is to ignite. Explns of combined firedamp & dust and of dust alone are more dangerous than of gas alone. This is because the expl wave in dusty mines gradually increases in velocity and may reach 1000 m/sec or even more. Explns of firedamp can usually be localized in their effect and their principal danger lies in the fact that a relatively minor firedamp expln may initiate a far more serious coal dust expln.

It has been found at testing galleries that generally, AN type expls ignite firedamp contg dust more readily than dust-free firedamp, while other expls ignite dust-free gas-air mixts just as easily as if dust were present (Refs 1, 2, 3, 5, 6, 8, 9, 10b, 12, 13, 14, 15, 16, 17, 18, 20, 21, 22, 23, 25, 26, 27, 29 & 31; Addnl Refs C, D, G, I, L, M, N, P, R, S, T, V, W, X, Y & Z)

Coal Mine Explosions; Measures for Preventing and Arresting Them. As a result of investigations conducted by various European organizations and individuals before and after WWI, the following methods were recommended in Europe to combat expln hazards: a)Infusion of water under pressure into the coal before it is broken down b)Spraying with water all accessible roads, roof and sides of mines with water in sufficient amt to ensure that the dust contains at least 30% of w c)Instead of water an incombustible dust can be used for spraying. It should be done is such a manner that the amt of coal dust in mixts does not exceed 50% (Refs 12, 22, 23 & 31)

The experiments of the "safety in Mines Research Board" (Gt Britain) conducted since 1924 have shown that the above recommendations are not sufficient to ensure safety in all coal mines. New regulations were drafted and they included, among other precautions, the provision of stopping zones heavily treated with stone dust, ash or water and overhead barriers loaded with stone dust or water (Ref 12, p119)

In USA, the practice prescribed by BurMines (1925) for combatting explns in mines other than of anthracite, includes the erection of stone dust barriers at specified places in addn to general stone-dusting. A standard minimum of 55% of incombustible material in mixts with coal dust is stipulated irrespective to the degree of the flammability of coal dust. Similar measures were enforced in Germany since 1926. The most important regulation is that concerning the use, in gaseous and/or dusty coal mines, of expls which would not ignite either firedamp or coal dust. A brief description of these expls known as "permissible" is given under COAL MINING EXPLOSIVES. Very few explns took place in USA since regulations of BurMines(ca 1910) were adopted, and the use of "permissible" expls became universal (Refs 4, 5, 7, 10a, 12, 20, 21, 22, 23, 24, 27 & 31; Addnl Refs A, B, E, H, J, Q, & Y)

Among other methods recommended for preventing coal mine explns may be cited: a)Spraying a foamed resin into mine passages and allowing it to harden in order to block the passage of firedamp (Ref 19); b)Introducing into bore-hole dry ice, together with cartridge, in order to prevent expln of firedamp (Addnl Ref F); c)Adding to coal dust an incombustible dust consisting of NaCl 80, French chalk 18 & Mg carbonate 2% (Addnl Ref K); d)Insertion of plate protection devices and wire gauzes to prevent spread of firedamp explns (Addnl Ref O); and e)Incorporation of some salts into coal dust (Addnl Ref U)

Notwithstanding the precautions adopted in USA and elsewhere in the world, quite a number of disastrous explns took place in this century. The following coal mine explns which caused more than 300 deaths are listed in Ref 31, p971: Courrières, France (1906 with 1099 killed); Monogah, West Virginia (1907 with 362 killed); Radbod, Germany (1908 with 360 killed); Pretoria,South Africa (1910 with 344 killed); Senghenydd, Wales (1913 with 439 killed); Honkeiko, Manchuria (1942 with 1572 killed); and Grimberg Monopol, Western Germany (1946 with 439 killed)

Coal Mine Fires. Fires in coal mines are particularly dangerous not only because a naked flame may ignite firedamp (or coal dust) and initiate an expln, but also because coal itself provides a virtually unexhaustible supply of fuel. Every precaution should be taken to prevent such fires and the use of combustible materials in mines should be avoided. As an example of fire caused by a combustible article, may be cited the disastrous fire in 1950 at Creswell Mine, England, responsible for the death of 80 people. It was caused by flammable rubber conveyer belting. To prevent a repetition of such disaster, the British National Coal Board ordered replacement of all underground rubber belting by non-flammable polyvinyl chloride belting

If the fire is small it can be fought by means of fire-extinguishers or water, but for big fires these methods might prove inefficient. A recent development in the field of under-

ground fire fighting is the *foam-plug* technique which consists in generating air-foam in quantity of several thousand cu ft per minute on the upwind side of the fire. This foam completely fills the roadway and is pushed by the normal air stream into the fire zone, thus blanketing the fire. Large scale trials of this method have been made in Gt Britain, USA, Poland and Russia (Ref 31, pp971-72)

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26, 29, 32, 33, 39, 40, 43, 46, & **48** dealing with various problems in connection with coal mine explns, their prevention and safety in mines) 21)I.Hartmann, USBurMines InfoCirc **7785** (1957) & CA **51**, 10908 (1957) (Development and control of coal dust explns in mines) 22)McAdam & Westwater (1958); 39 (Ignition of firedamp by expls); 39-40 (Ignition of firedamp by hot gases); 40-41 (Ignition of firedamp by hot particles); 41-44 (Ignition of firedamp by adiabatic compression) 23)Taylor & Gay (1958), 8-9 (Ventilation in mines); 10-11 (Firedamp and coal dust hazards); 47 (Precautions against coal dust explns) 24)R. Bouchet & P.Laffitte, CR **246**, 1958-61 (1958) & CA **52**, 14171 (1958) (When a detonating wave passes thru clouds of finely divided powder, such as coal dust, the wave can be extinguished by introducing relatively small amts of powdered inert materials, such as silica & potassium acid tartrate) 25)H.B. Humphrey, USBurMines-InfoCirc **7900** (1959) and Bulletin **586** (1960) (Historical summary of coal-mine explosions in the US from 1810 to 1958) 26)R.L.Grant, USBurMines Rept Invest **5486** (1959) (Influence of borehole freespace on ignition of firedamp by expls) 27)H.Starland, TekTidskr (Sweden) **88**, 1273-82 (1958) & CA **53**, 8629 (1959) (Review on dust explns, their causes, expln limits and safety measures) 28)D.C.Kingery, Introduction to Mine Ventilation, Principles and Practice, USBurMines Bull **589** (1960) 29)J. Nagy & W.M.Portman, USBurMines Rept Invest **5812** (1961) (Coal dust explns in air contg small amts of methane); JApplChem **12**, No 2, pp 1-135 (1962) & Explosivst **1962**, 224 30)Merriam-Websters (1961), 855 (Firedamp) 31)EncyclBritannica **5** (1963), 971-72 (Coal mine explns) 32)EncyclBritannica **19** (1963), 809

Addnl Refs: A)(?)Heyer, SS **21**, 22-3 (1926) (Prevention of firedamp & coal dust explns in coal mines) B)W.Landgraeber, SS **21**, 71-3 (1926) (Modern methods for combating firedamp explns) C)G.S.Rice, USBurMines TechPaper **448** (1929) (Coal-dust explns in mines - causes, effects and recommendations for prevention) D)G.S.Rice & H.P.Greenwald, USBurMines TechPaper **464** (1929) & CA **24**, 944 (1930) (Coal dust explosibility) E)A. Schrimpff, SS **25**, 197-98 (1930) & CA **24**, 6018 (1930) (Coal dust expln in a French coal mine resulted from a blown-out shot of an expl contg AN 90, K nitrate 5 & TNN 5%. This probably would not happen if this expl contained at least 4% of NG, as required by Ger regulations for this type of expls. NG is included in order to decrease the duration of flame produced on expln) F)V.Bohler, RevUniverselleMines **12**, 144-50 (1936) & CA **30**, 5415 (1936) (Prevention of firedamp and/or coal dust explns by introducing into the borehole dry ice together with the expl cartridge, provided with a detonator, and then closing the hole with clay. On firing, an inert atmosphere is formed around the place of expln and this prevents ignition of firedamp or coal dust) G)N.Simkin, Colliery Guardian **154**, 103-06 & 149-51 (1937); CA **31**, 2393 (1937) (Flammability of coal dust) H)J.Smellie & A.M.Bryan, TransInstMiningEngrs **94**, 171-91 (1937) & CA **32**, 1933 (1938) (The efficiency of noncombustible dust in prevention of coal-dust explns) I)W.Payman & R.V.Wheeler, TransInstMining Engrs **95**, 13-47 (1938) & CA **32**, 6463 (1938) (The ignition of firedamp by coal mining expls) J)E.Audibert & R.Cheradame, RevInd-Minérale No **412**, 105-22 (1938) & No **431**, 43-9 (1939); CA **32**, 4338 (1938) & **33**, 2717 (1939) (Review of methods using incombustible powders to prevent propagation of dust explns in coal mines) K)G.Allsop et al, TransInst Mining Engrs **97**, 20-39 (1939) & CA **33**, 7114 (1939) [Firedamp explns can be stopped by incombustible dust (such as mixt of NaCl 80, French chalk 18 & Mg carbonate 2%) projected in the path of an expln. This can be done by means of motors operated by thermal relays which, when subjected to the heat of an expln, close an elec circuit] L)H.P.Greenwald, USBurMines Rept Invest **3489** (1940) & CA **34**, 3091 (1940) (Explosibility of 22 coal dusts contg 8 to 31% volatile matter) M)W. Gassmann, Glückauf **79**, 369-77 (1943) (A review of firedamp explns caused in recent years by blasting in Dortmund mines) N)G.S.Scott, USBurMines Bull **455** (1944) & CA **38**, 6072 (1944) (Anthracite mine fires: their behavior & control) O)A.van Tiggelen, Bull Belg **55**, 202-44 (1946) & CA **41**, 4363 (1947) (Plate

protection devices and wire gauzes are effective against firedamp explns) P)M.V. Blanc et al, JChemPhys **15**, 798-802 (1947) & CA **42**, 762 (1948) (Ignition of explosive gas mixts by elec sparks) Q)W.Cybulski, BiulInstWegloe (Katowice) Komun No **61**, 128pp (1950) & CA **46**, 7770 (1952) (Stopping coal-dust explns by rock barriers) R)R.Goto et al, BullInstChemResKyotoUniv **20**, 46-7 (1950) & CA **47**, 8342 (1953) (The lower the content of the volatile matter, the lower was the flammability of coal dust and below 27.27% content the coal was no longer flammable. Addn of 40% pptd Ca carbonate, 45% marble, or 50% silica or alumina diminished flammability The smaller was the particle size of non-flammable addns, the greater was the reduction of flammability) S)K.Matsumoto, JMiningInstJapan **68**, 260-82 (1952) & **70**, 463-66 (1954); CA **48**, 3691 (1954) & **49**, 6608 (1955) (Coal dust explns) T)W.Cybulski, Prace Glównego Inst-Gornictwa, Ser A, Komun No **147**, 28pp (1954) (Engl summary) & CA **50**, 10373 (1956) (Expl concn limits of coal dusts); Ibid, Komun No **159**, 34pp (1954) (Engl summary) & CA **50**, 10410 (1956) (Safety of mining expls in the presence of coal dust) U)H. Ahrens, Ministry of Fuel and Power (Brit), Paper No **30**, 21pp (1954) & CA **49**, 13651 (1955) (Prevention of coal-dust expln by incorporating in mine expls some salts) V)H.Selle & J.Zehr, Staub **1954**, 583-600; Chim & Ind (Paris) **74**, 961 (1955) & CA **51**, 18609 (1957) (An opinion on experimental values of the lower limit of flammability of dust-air mixts on the basis of thermochemical calcns) W)F.Otaśek, Uhli (Czech) **5**, 202-08 & 382-90 (1955); CA **50**, 1315 & 11017 (1956) (Explosiveness of coal dust-methane-air mixts and effect of inert gases on the expln delimitation of methane-air mixts) X)W.L. Grant et al, US BurMines ReptInvest **5224** (1956) & CA **50**, 13402 (1956) (Influence of amt moisture on ignition of firedamp by atm expls) Y)Coll, RevIndMinérale **39**, 57-102 (1957) & CA **51**, 7013-14 (1957) (Several papers on coal dust explns and their prevention) Z_1)D. L.Carpenter et al, Combustion and Flame **1**, 63-93 (1957) & **2**, 35-54 (1958); CA **51**, 7719 (1957) & **52**, 8559 (1958) (Explosibility characteristics of coal dust clouds using elec spark ignition; influence of temperature) Z_2)A.A.Borissov et al, DoklAkadN **149**, 869-71 (1963) & CA **59**, 376 (1963) (Study of ignition in reflected shock wave of mixts of methane with air and inert diluents) Z_3)R.H.Essenhigh & J.Csaba, 9thInternCombustSymp, Cornell Univ, Ithaca, NY (1963), 111-25 & CA **59**, 12587-88 (1963) (The thermal radiation theory for plane flame propagation in coal dust clouds)

Coal Mines, Determination of Firedamp and of Coal Dust In Atmospheres of. Although many instruments have been designed to detect the presence of firedamp (or rather methane) in mine atmospheres, the principles of the flame safety lamp (Davy-type lamp) still form the basis of many detectors. The Davy lamp invented in 1815 is briefly described under COAL MINE EXPLOSIONS AND FIRES and it is stated that each US mine should have at least two Davy-type lamps to serve as detectors of firedamp or of lack of oxygen. If firedamp is present in small quantity, the flame of Davy lamp elongates and if the gas is present in considerable quantity, the lamp becomes filled with blue flame. For more definite detection of gas, the flame of the lamp is lowered until the yel part is at a minimum. Then the gas will be discernible as a small blue cap over the flame. This method is described in Refs 1, 9, 12 & 25. Some investigators consider that the safety lamp method of detection of firedamp is not very reliable (Ref 7)

Other methods of detection and analysis of firedamp are discussed in Refs 1, 4, 5, 6, 7, 8, 11, 13, 15, 16, 17, 19, 20, 21, 23 & 24. Descriptions of methods for detn of coal dusts in mine atmospheres and the methods for testing the inflammability or of the explosibility of coal dusts are discussed in Refs 2, 3, 10, 14, 18 & 22

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289, 29pp (1935) & CA **30**, 3238 (1936) (Lab studies of flammability of coal dusts) 3)G. S.Rice & H.P.Greenwald, USBurMinesInfo-Circ **6878**, 8pp (1936) & CA **30**, 4007-08 (1936) (Notes on testing the explosibility of coal dusts and a proposal to have an international test method) 4)L.Altbürger,Glückauf **72**, 987-88 (1936) & CA **31**, 7251 (1937) (A simple apparatus for firedamp testing) 5)Oldham & Son Ltd and H. Holt, Jr, BritP 462733 (1937) & CA **31**, 5631 (1937) (A device for detecting firedamp in mines) 6)R.Kattwinkel, Glückauf **74**, 482-84 (1938 & CA **33**, 3157 (1938) (New app for the study of firedamp) 7)F.Lebeter, Colliery Engrg **16**, 245-48 (1939) & CA **33**, 9647 (1939) (Discussion of the Ringrose and McLuckie appliances and statement that detection of firedamp by means of a safety lamp is liable to many errors) 8)T.D.Jones & W.L.Gyles, Colliery Guardian **159**, 588-90 (1939) & CA **34**, 3090(1940) (Detn of firedamp content in mine atmosphere) 9)K.Nehring, Glückauf **75**, 259-62 (1939) & CA **34**, 626 (1940) (Flame safety lamp as a detector for firedamp) 10)W.M.Schweickart, USP 2195842 (1940) & CA **34**, 5275 (1940) (App for empirical detnof dust in bituminous coal mines) 10a)J.Formánek, Horniký Vestnik **22**, 241-43, 256-60 & 281-87 (1940) & CA **37**, 6462 (1943) (Detn of composition and explosibility of firedamp) 11)J.H.Griffiths et al, ProcSouthWalesInstEngrs **57**, 164-77 & 248-58 (1941); CA **36**, 3669 (1942) [Detn of firedamp content in mine atm by means of the MSA (Mine Safety Appliance) methane safety tester] 12)P.Jungbecker, GerP 730978 (1942) & CA **38**, 646 (1944) (Safety lamp for detection of firedamp in mines) 13)G. Blecher, Glückauf **79**, 489-95 (1943) & CA **38**, 5408 (1944) (Detn of the flammability limits of mine gases) 14)H. Pieters & J. Hovers, Fuel, **27**, No. 4, 109-17 (1948) & CA **43**, 405 (1949) (A review of the most important methods for the detn of the explosibility of coal dust in air) 15)G. Champagnac & J. Crétin, AnnMines **139**, 7-16 (1950) & CA **45**, 6844 (1951) (New app for analysis of firedamp) 16) Machinenfabrik Wagener & Co, GerP 824567 (1951) & CA **48**, 10390 (1954) (A firedamp recorder consisting of a methane-sensitive catalyst with a reading device incorporated into a wrist watch) 17)K.Drekopf, Brennstoff-Chemie **33**, 146-51 (1952) & CA **46**, 6811 (1952) (Investigation of coal mine gases) 17a)R. L. Grant et al, USBurMines ReptInvest **5050** (1954) (A new method for measuring incendivity of firedamp) 18)I. Hartmann et al, USBurMines ReptInvest **5052**, 8pp (1954) & CA **48**, 7901 (1954) (Lab studies of explosibility of dusts from various Amer coals, under different conditions) 19)K. Winter, RevInd Minérale **36**, 43-9 (1955) & CA **49**, 8637 (1955) (Portable app for the detection of firedamp in mines) 20)A. Monomakhoff, RevInd Minérale **38**, 228-42 (1956) & CA **50**, 15345 (1956) (Description of various chem & phys methods used for detection and measurement of firedamp) 21) F.Otasek, Uhli (Czech) **6**, 161-69 (1956) & CA **51**, 12494 (1956) (A new method for detg explosive boundaries of firedamp/air mixts) 21a)M.G. Zabetakis et al, USBurMines InfoCirc **7901** (1959) (Detg the explosibility of mine atmospheres) 22)R.S.Schechter & T.L.Kang, ASTM Bull **241**, 34-7 (1959) & CA **54**, 5185 (1960) (Continuous method for testing explosibility of powdered materials, such as coal dust, etc) 23)L.Chaineaux & A. Monomakhoff, RevIndMinérale **41**, 459-65 (1959); Chim & Ind (Paris) **84**, 113-14 (1960) & CA **56**, 14547 (1962) [Étude de differents problèmes de grisoumétrie] (Schlagwettermessung in Ger) (Review of methods of methane detection and detns in mines; warning problems; and description of several apparatuses, including: "Explosimètre 58" (Developed by CERCHAR); "Verneuil téléindicateur 59" (derived from "Verneuil 54"); and some others developed by CERCHAR. Another app called "Analyseur-déclencheur-rapide 59" was in state of development at the time of publication of this article] 24)A.Monomakhoff, RevIndMinérale **44**, 225 (1962) & Explosivst **1964**, 48 (Description of two new devices developed by CERCHAR for detn of methane content in coal mine atmosphere) 25)EncyclBritannica **19** (1963) (Detection of firedamp by means of Davy safety lamp)

Coal Mining Explosives. See under COMMERCIAL or INDUSTRIAL EXPLOSIVES

Coal Mining Explosives, Testing for Permissibility. In order to manuf an expl which will not ignite expl mixts of air with firedamp and/or coal dust, it is advisable to consider

the following general requirements for such expl: a)Its oxygen balance to CO_2 & H_2O should be positive (2-4%), in order to provide sufficient oxygen for complete combustion of paper container (cartridge), and to prevent the formation (on expln of charge) of carbon monoxide, which is extremely toxic. No other toxic gases such as N oxides should form in large quantities b)Its strength (power) as detd by Trauzl Test or Ballistic Mortar Test values should be lower than for ordinary dynamites c)Its detonation rate should be lower than for ordinary dynamites, when using cartridges of prescribed diameters (such as 1¼") d)Its brisance should be rather low so that coal would not be broken into very small pieces e)Its heat of expln should be rather low, so that the maximum temperature developed on expln (which is calcd by dividing heat of expln by the sum of specific heats of components formed on expln), would not be higher than allowed by regulations of various countries. For example, in France the max temp is 1500° for expls used in coal layers (explosifs grisou-couche) and 1900° for expls used for blasting rock in gaseous and/or dusty coal mines (explosifs grisou-roche); in Russia, the limit is 2200°, provided other requirements are met (The method for calcn of "permissibility" is given under COMMERCIAL or INDUSTRIAL EXPLOSIVES). The low temp requirement means that the temp of flame produced on expln should be lower than for non-permissible expls f) It is desirable that the duration of flame and its size would be rather small. This consideration, however, is not of such importance as was claimed by early investigators (Ref 43, p507, footnote) g)The volume of gases produced on expln should be rather high so that the charge could exert a rather slow heaving action, but no shattering effect. i)The ratio: (Volume of gases in liters per kg) to (Heat of expln in kcal per kg) should be greater than for non-permissible expls, such as ordinary dynamites j)The expl should be relatively insensitive to impact of friction, but fairly sensitive to initiation by influence or by a blasting cap (such as No 6) k)The quantity of expl used for a shot without causing ignition of firedamp and/or coal dust should be detd and if the limiting weight (See Charge Limit in Vol 2, pC115-R of this Encycl) is much smaller than is desired, the expl could be considered as non-permissible. The limiting chge for "sheathed" expls is about twice as high as for non-sheathed permissible expls l)The expl should be chemically and physically stable under wide variations in storage and operating conditions m)The expl should pass all of the prescribed *gallery* tests (See further) *US Government Regulations and Tests for Permissible Explosives.* Each expl intended for use in Amer gaseous or dusty coal mines should be submitted to the Bureau of Mines Testing Station at Bruceton, Pennsylvania. There are 10 standard tests which are listed in Bureau of Mines Schedule **1-H** (Ref 45). The fee for all of these tests is $1200. As it is charged whether the expl passes all the tests for permissibility or not, it is advisable not to submit them until some of their thermochemical properties are calcd. For this purpose, the method of calcn given in this vol of Encycl, under COAL MINING EXPLOSIVES, Permissible may be used. If the calcn shows that the expl does not fulfil the theoretical requirements for "permissibility", its compn should be changed before submitting the expl to Bureau of Mines

The Bur of Mines tests listed in Ref 45, consist of the following:

A. *Chemical Tests,* which include:

a)Chemical analysis for ingredients using methods described in Ref 5 or in the books of Analytical Chemistry, such as Ref 45a, should correspond [within tolerances specified in paragraph 21(b), p6 of Ref 45] to the compn furnished by the applicant

b)Gaseous products of explosion are detd using the Bichel Pressure Gage (Ref 14, pp91-5; vol 1 of this Encycl pVIII and Vol 3, under CLOSED BOMB) for detn of carbon monoxide, and the Crawshaw-Jones Apparatus for nitrogen oxides (Ref 14, pp95-9). The amt of poisonous gases produced should not exceed 2.5ft^3 per lb of expl (71 liters per 454g)

B. *Physical Tests,* which include:

a)Apparent specific gravity and wrapper-to-explosive ratio. These values are detd for several cartridges taken at random from each shipment of expls. Methods of testing are

given in Ref 14, pp22-3

b)Ballistic mortar (or pendulum) test is used for detn of strength of expls. Detailed description of apparatus and test, including detn of **UDC** (Unit Deflective Charge) is given in Ref 14, pp40-9. The UDC is the wt of expl that has been found to deflect the Bureau of Mines pendulum to the same degree as the **PTSS** (Pittsburgh Testing Station Standard) Dynamite, which consists of NG 40, Na nitrate 44, woodpulp 15 & Ca carbonate 1%

Accdg to Dr Van Dolah (Ref 47), the UDC is no longer employed by the BurMines. The standard method for detg strength of expls is the ballistic mortar in which a 10g chge is fired in the usual manner and the results are expressed as a percent of the strength of TNT, using the ratio of 1 minus the cosine of the angle of deflection of the pendulum

c)Detonation rate is detd on a 50" column of 1¼" diam cartridges, and for the smallest diam submitted for testing, provided that this diam is less than 1¼". Nongelatinous expls are initiated with the BurMines test detonator alone, while the gelatinous expls are initiated with a combination of this detonator and a 60g Tetryl pellet. The Bureau's detonator contains as the base charge

0.25 ± 0.02g PETN. The expl should not fail to propagate completely in this test (See Ref 14, pp60-65)

d)Friction sensitivity is detd by *pendulum friction apparatus,* as described in Ref 14, pp78-84. Ten trials are made with the steel shoe released from a height of 1.5 meters (59") and if the evidence of sensitivity appears the test is repeated with the hard -fiber-faced shoe. The expl must not show, in any trial with hard-fiber shoe, a result more unfavorable than an almost undistinguishable local crackling (Ref 75, pp4 &5-6)

e)Explosion-by-influence sensitivity or air -gap sensitivity is detd by the *halved-cartridge method* using 1¼" diameter cartridges as described in Ref 14, pp 59-60. The sensitivity must be at least 3" (Ref 75, p4)

f)Gallery tests. See under Galleries for Testing Coal Mining Explosives, item M

Galleries for Testing Coal Mining Explosives. In order to be reasonably sure that an expl intended for use in a coal mine, contg an easily ignitable atmosphere of methane and/or coal dust, it is required in many countries to test it under conditions approaching the actual conditions encountered in a mine. The nearest approach to a mine is either an abandoned mine or a gallery filled with atm encountered in mines

A testing gallery is essentially a long, cylindrical vessel of large cross-section, circular or elliptical. One end of the gallery is closed either by a steel plate or by a brick (or cement) wall, while the other end is open. An opening in the closed end (base) is provided for insertion of the muzzle of a special cannon (mortar). The first few meters of the closed end are separated from the open end by means of a paper partition, and this section is called "explosion chamber" or "gas chamber"

For conducting the test, the expln chamber is filled with 8-9.5% methane/air mixt and/or coal dust/air mixt and a known weight of an explosive to test is fired (stemmed or unstemmed) from the special cannon into the chamber thru the opening in the base plate. The expl is considered to pass the test for permissibility if 5(or10) consecutive firings of prescribed weight cause no ignition (which is usually followed by expln) of chamber atmosphere. In Austrian gallery the charge is not fired from the cannon, but as indicated below

Following is the list of principal past and present galleries and brief descriptions of tests conducted in various countries:

A. *Austria.* Gallery at Mährisch-Ostrau, erected in 1891, was 293m long and was part of an abandoned mine. The shots were fired suspended in the mine, and not from a cannon, thus representing more or less the conditions of a chge exploding accidentally before being placed in a borehole (Ref 8, p 121 & Ref 25, p 108). Another gallery, 300m long, was erected in 1909 at Segen Gottes (in the Rossitzer district), but previous to this some tests were conducted in a short, surface gallery at Poln -Ostrau (Ref 25, p 114). No info at our disposal about current galleries. The place where the 1st gallery was located is now known as Moravska Ostrava and belongs to Czechoslovakia

B. *Belgium.* The gallery at Frameries used probably as late as WWII was 32m long with

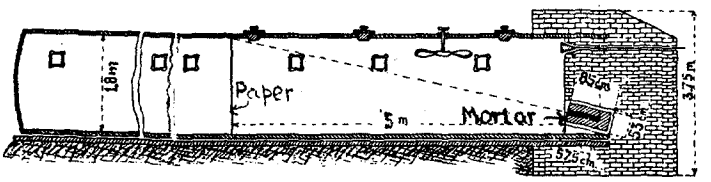
elliptical cross-section 1.4m wide & 1.8m high. Its expln chamber was 5.5m long. The test was conducted by firing unstemmed shots into 8.0-8.5% firedamp/air mixts (Ref 8, p121; Ref 17, p188 & Ref 21, pp409-10). The gallery used at the present time is at Pâturages. Its demensions are, accdg to Ref 41, p37: diam of circular cross-section 5 ft & length 67 ft of which 17 ft is separated by a paper partition to form the explosion chamber. The chge limit of an expl under test is detd by firing various wts from a special cannon, 2¼" bore & 20" long into the expln chamber filled with both 9% firedamp/air and coal-dust/air mixts. By this test, an expl will qualify as SGP (sécurité-grisou-poussière) if no ignition of chamber atm takes place with chges as high as 31 oz (although the max charge permitted underground is only 28 oz). More detailed description of the test is given by J.Fripiat in AnnMinesBelg **50**, 599 (1951)

C. *Czechoslovakia.* The official testing gallery, located at Moravska Ostrava (formerly Austrian Mährisch-Ostrau), is elliptical in cross-section, 4½ ft wide & 6 ft high. Its length is 80 ft of which 16 ft is the expln chamber. The charge limit of an expln under test is detd by firing it unstemmed from a special cannon (2.2" bore & 2 ft long) obliquely from below into the expl chamber filled with 8-9% firedamp/air mixt. The max wt of five consecutive chges causing no ignition of gas should not be less than 16 oz. Four shots equal to max charge limit and one shot equal to 21 oz are fired into the chamber filled with coal-dust/air mixt and if no ignition occurs, the expl is considered to pass the permissibility tests (Ref 41, p37). There was also a gallery at Semtin (Ref 21, p410)

D. *France.* The gallery erected in 1907 at Liévin was destroyed during WWI. The gallery erected after WWI at Montluçon was of 2m diam & 9.9m length of which 3.5m was partitioned to form the explosion chamber (Ref 17, p188; Ref 21, p410 & Ref 25, pp111 h 114). At present, accdg to Taylor & Gay (Ref 41, pp37-9), the CERCHAR (Centre d'Étude et Recherches des Charbonages de France) located at Verneuil, uses two testing galleries, each of 2m outside diameter, but lined with concrete so as to give a hexagonal cross-section. In one of the galleries, the expln chamber is 5m(ca 197") long and the extension 7m(ca 276"), whereas, in the other, the chamber is 7m and the extension 5m. The former is used for gas tests using a 3.8cm bore by 22cm long mortar closed with a steel disc and firing the chge of dimension 3.0x18cm; the latter uses the 3.8x34.0cm mortar and the chge 3.0x30cm unstemmed. Coal dust tests with both mortars are conducted in the extension to the 2nd gallery

Expls utilized in gassy or dusty coal mines are divided into: a)Explosifs couche and b)Explosifs couche améliorés. The former may only be fired with instantaneous detonators and their charge limit is 500g for shots in coal layer and 1000g for shots in rock. The latter expls may be fired with delay detonators and their charge limit is 1500g for shots in coal and 2000g for shots in rock. The delay betw the 1st and last shots of the round should not exceed 5sec, except in particularly dangerous mines where a max delay of 0.5sec is established

E. *Germany.* The first gallery built in Germany seems to be that at Neunkirchen bei Saarbrücken. Another older gallery was at Gelsenkirchen. We do not know when it was constructed, but it existed as early at 1908 (Ref 1, p584; Ref 8, p121 & Ref 11, pp 14 & 396). Accdg to Barnett (Ref 8, p121) & Pérez Ara (Ref, 27, p258), the gallery at Gelsenkirchen, which is represented here in Fig, was 34m long (of which 5m was the



GELSENKIRCHEN GALLERY

explosion chamber). It was elliptical in cross-section, 1.8m wide & 1.35m high. The mortar for firing expl chge, with a bore 4cm diam and 70cm depth, was placed in an inclined position at the closed end of the gallery. In each test five unstemmed shots were fired.

The gallery at Derne, near Dortmund was erected in 1911 (Ref 25, p114)

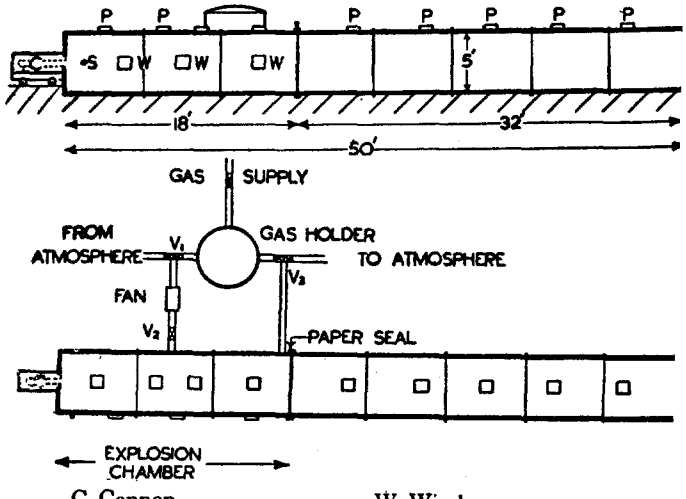
Accdg to Beyling & Drekopf (Ref 21a, pp

3 & 107), three official galleries (Versuchstrecken) existed in Germany before WWII: the Dern-Dortmund, Beuthen (Oberschlesien) and Freiberg (Sachsen). Taylor & Gay (Ref 41, p39), list the same galleries as being in current use. The 1st & 3rd galleries are elliptical in cross-section and are somewhat similar to the Czechoslovakian gallery at Moravska Ostrava. The gallery at Beuthen (erroneously spelled in Ref 41, p39 as Benthen) is circular in cross-section and larger than the other

The Dortmund-Derne gallery is 25m long and its elliptical cross-section is 1.30m wide and 1.80m high. One end of the gallery is closed by a brick wall, while the other is open. The expln chamber is 5m long and is separated from the open section of gallery by a paper partition. The chamber can be filled with 8-9.5% methane/air and/or coal dust/air mixts. A steel cannon 2.2" bore & 23.6" long is used for shooting the unstemmed chges starting with 400g wt and increasing it by 50g until an ignition of chamber atm takes place or until 600g wt is reached w/o ignition of the atm. If this chge is too big to be fired from the above cannon, then a smaller charge limit is established. If ignition of gas takes place with chges lower than 450g, the expl is not considered as "Wettersprengstoff". For a more detailed description of the test, as it was conducted before WWII, see Ref 21a, pp107-09. No info at our disposal about current Ger procedures

F. *Great Britain.* The first gallery was erected ca 1890 at Hebburn-upon-Tyne by the North of England Institute of Mining and Mechanical Engineers. Following the passing of the Coal Mines Regulation Act of 1896, a Home Office Testing Gallery Committee was appointed and, on recommendation of this Committee, the Government erected in 1897 the Woolwich Testing Station with a gallery 27.5ft long and 2.5ft in diameter. This station was transferred in 1911-1912 to Rotherham where a new gallery was erected. This gallery was 50ft long, 5ft in diam with an expln chamber 18ft long. Besides the Govt galleries at Woolwich & Rotherham there was also a testing station with a 300ft long gallery, erected by the Mining Association of Great Britain in 1908 at Altofts, Yorkshire. The installation was transferred in 1911 to Eskmeals where it was taken by the Govt. The gallery was distinct from the smaller galleries at Woolwich and Rotherham. In 1921, difficulties with supply of coal gas for the gallery at Rotherham led to a temporary transfer of testing to the Ardeer Test Station of the Nobel's Explosives Co, where a gallery identical with that at Rotherham was erected. In 1922 the Explosives in Mines Committee was created and the Rotherham station was transferred to a more accessible site, near Buxton, Derbishire. At about the same time the tests at Eskmeals were discontinued (Ref 1, p585; Ref 8, p123; Ref 17, p186; Ref 25, pp109-11 & Ref 41, pp 33-5)

The Buxton gallery which is presently used is the same size as was the Rotherham gallery, namely 5ft (ca 1.52m) in diameter and 50ft (ca 15.2m) long of which 18ft (ca 5.5m) is the expln chamber. A schematic view of the gallery is represented in Fig. It is taken from the book of McAdam & Westwater (Ref 40, p35)



C Cannon W Window
S Sampling point P Pressure-release vent
V_1 Two-way valve, from gas-holder or from atmosphere
V_2 Valve
V_3 Two-way valve, to gas-holder or to atmosphere

BUXTON TESTING GALLERY

The gallery consists of 8 sections of ½" thick mild-steel plates. The expln chamber, the first 3 sections, is closed at the left by a ¾" steel plate with a 12½" diam hole in the center. Surrounding this hole is a rubber ring which forms an air-tight joint with the muzzle of the cannon, when it is run into position to fire a shot. There is a space of ca 1" betw

the chamber and the remainder of the gallery to allow a sheet of oiled brown paper (serving as the seal) to be attached by means of two rings and a rubber & asbestos washer. The chamber is filled before each test with gas contg 9.0± 0.25% methane in air. The cannon is of steel and has a bore 120cm long and 55mm in diam

The presently used Buxton Test, known also as the Home Office Test, was introduced in 1932 as the British Official Gallery Test and it is still used for ordinary permitted expls. After introduction of new types of expls, such as "sheathed" (in 1934) and EqS (equivalent to sheathed) (in 1949) some modifications and additions were considered to be desirable and a new testing procedure was established in 1953

Before proceeding to test an expl in the gallery, it is necessary to ascertain that it detonates completely when 1-meter length of the chge made from cartridges of the smallest diam(usually 1 7/16") and wt to be manufd, is initiated by a detonator of the strength recommended by the manufacturer. For this test, the cartridges are held in loose contact in a column made from a three-thickness roll of the paper used for wrapping the expls and the resulting cartridge is laid on a flat steel surface, such as a railway rail, before initiation

British gallery tests for "unsheathed" expls are conducted as follows:

a)Five shots, each of 8oz and unstemmed, are fired by direct initiation (which means with the detonator inserted in the end of the cartridge nearest to the mouth of the cannon) into a 9% firedamp/air mixture. All cartridges are 1 7/16" diam

b)Five shots, each of 28oz, and stemmed with a 1" thick plug, are fired, by direct initiation into a similar atmosphere

c)Five shots as in b are fired into a coal-dust/air cloud, contg from 20oz to 5 pounds of coal dust per test

In order to be authorized as a "Permitted" explosive, no ignition should occur in any of the above tests. The explosive has also to satisfy certain requirements as to sensitiveness to propagation. The permitted maximum chge is 28oz, except for certain low density expls where the max chge is 18oz (Ref 40, pp 34-7 & Ref 41, pp34-5)

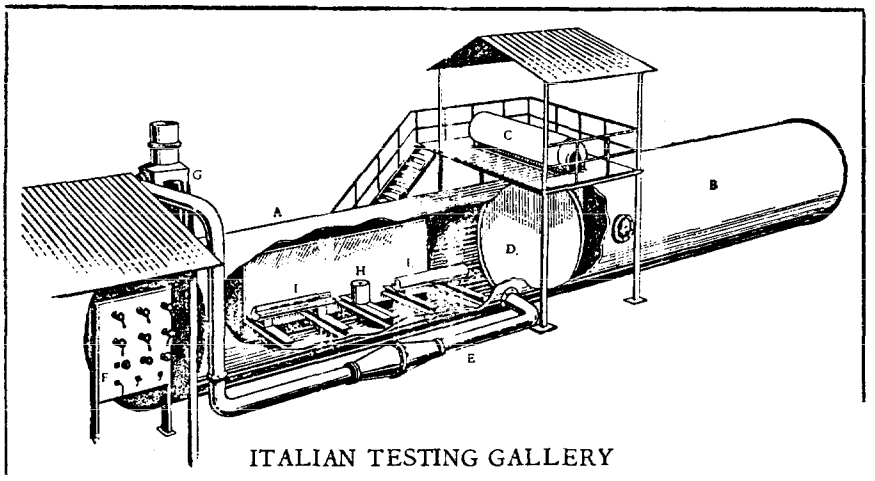
Tests for " sheathed" and for EqS (equivalent sheathed) expls, as required by regulations of 1953 are listed in Ref 40, pp38-9 & Ref 41, p36

G. *Italy.* A testing gallery (galleria di prova in Italian), such as used by the "Nobel-SGEM" (Società General di Esplosivi e Munizione) at Orbetello is briefly described by Belgrano (Ref 31a, pp168-71) and by Giua (Ref 44, pp 450-51). The gallery is cylindrical in shape, diameter 1.60m and length 20m, and made from iron plate 18mm thick. One end of the gallery is closed by a wall, while the other end is open. The first 5m section nearest to the closed end is partitioned by a paper screen to form the explosion chamber. This is filled before each test with an 8.5-9.5% methane/air mixt. The mortar consists of a steel jacket 530mm outside diam and 850mm long and a Ni-Cr steel core, 200mm outside diam, drilled inside to form the weapon 55mm inside diam and 550mm long. The charge of explosive to test is fired thru the opening in the wall into the expln chamber. In the book of Giorgio (Ref 48) is described a gallery which is similar to the one discussed in Refs 31a & 42 (See Fig on pC374; it is taken from Ref 48, p97)

Accdg to Dr. Vettori (Ref 46) there is only one gallery, the one at Orbetello and it is owned by Montecatini SGIMC (Società Generale per l'Industria Mineraria e Chimica). There is no testing laboratory in Italy comparable to that at USBureau of Mines

H. *Japan.* Accdg to Taylor & Gay (Ref 41, p 39), the testing gallery was located, before WWII, at Nagata and was constructed on the same lines as the British and American models. A 14oz chge of the expl to test was required not to ignite either firedamp or coal dust. No info is at our disposal concerning current galleries

I. *Poland.* Accdg to Taylor & Gay (Ref 41, p 39), the testing gallery used before WWII was located at Mikalow. It was circular in cross-section, 6'6" in diam and 143' long with a 10' long explosion chamber. The cannon was situated inside the expln chamber, had a bore of 2" and was either 27.5" or 47" long, depending on whether the expl was of normal or of low density. Five shots were fired into an 8-9.5% firedamp/air mixt and into coal-dust without stemming and with the detonator in front of the



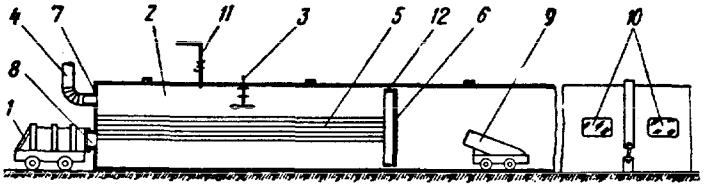
ITALIAN TESTING GALLERY

A – Explosion chamber
B – Expansion chamber
C – Paper roll
D – Paper diaphragm
E – Pipe conducting combustible mixture
F – Switchboard
G – Mixer of methane with air
H – Vertical mortar
I – Angular mortar
L – Angular mortar

chge. The maximum permissible chge, for use underground, was fixed as 18oz for normal expls and 25oz for low density expls. No info is at our disposal concerning current galleries

J. *Russia.* Accdg to Andreev & Belyaev (Ref 43, pp492-97), the Russian expls designed to be authorized for use in gaseous and/or dusty coal mines are required to pas the tests conducted at the gallery of the Makeev Scientific Research Institute for Safety in Mines. The gallery is called in Rus "Opytnyi Shtrek MakNII" where MakNII stands for " Makeyevskii Nauchno -Izsledovatel'skii Institut po Bezopasnosti Truda v Gornoy Promyshlennosti." The expls which pass gallery tests and comply with some other requirements, are now known as "predokhranitel'nyiye" formerly called "antigrisutnyiye")

The gallery which is represented here in Fig (reproduced from p493 of Ref 43), consists of a steel cylinder 1.65m diam and 15m long, open at one end and closed at the other with a steel plate (7), provided with an opening (8) for the muzzle of the mortar (1) and with the opening (4) to serve as a vent. The section of the gallery nearest to the closed end is partitioned by means of a paper diaphragm (6), held in place by a ring (12), to form the explosion chamber (2), of $10m^3$ capacity. The mortar (1) consists of a steel cylindrical block with a hole 55mm diam drilled to the depth of 900mm



RUSSIAN TESTING GALLERY, MAKNII, AND ACCESSORIES

In the above fig, number 3 indicates agitator, 11 - the inlet pipe for gas, 5 - the shelves for coal dust, 9 - the mortar for shooting coal dust into expln chamber and 10 - observation windows

In the tests designed prior to 1954, charges of expls in cartridges 30-32mm in diameter were fired, unstemmed, from mortar (1) into expln chamber (2) filled with an 8 to 10% methane/air mixt. The first 10 chges were of 350g and if no ignition of firedamp took place, ten chges of 450g were fired. The expl was considered to pass the test if no ignition takes place with 450g chges. If it is desired to establish charge limit, ten charges of 550g and finally of 650g were fired. The regulations of 1954 raised the

max wt required for permissibility from 450g to 600g

In testing the permissibility against coal-dust/air mixts, various methods have been used for introducing the dust into the expln chamber. One of the methods is to spread the dust on shelves (5) and floor of the gallery, using total of 3500g dust per $10m^3$ capacity. Another method is to place some coal-dust in mortar (1) on top of expl test and to shoot the mixt into chamber (2). In the method adopted in 1954, the coal dust is spread more evenly in suspended condition, than by other methods. For spreading the dust, a special mortar (9) is used. With the paper screen (6) removed and the muzzle of mortar (9) directed against the steel plate (7), the dust is projected from mortar (9) by firing a small chge of expl, just prior to firing a chge of expl to test from mortar (1), thru hole (8). The expl is considered to pass the test if five shots of 700g fail to ignite the dust

K. *Spain.* The book of M. Vivas, R. Feigenspan & R. Ladreda, " Pólvoras y Explosivos Modernos", J. Morata, Madrid, 4th edition in 5 vols (1944-1948) does not describe any gallery, Spanish or foreign. The book of Pérez Ara (Ref 27, pp255-58) describes some galleries located in USA, England, France, Belgium & Germany, but no Spanish galleries are listed. This means that we have no info about Spanish galleries

L. *Switzerland.* Accdg to the late Dr. A. Stettbacher (letter dated 9 June 1954), there are no testing galleries in Switzerland

M. *United States of America.* All tests are conducted by the Bureau of Mines at Bruceton, where several galleries are located. The main, or No 1, gallery consists of a cylinder 100ft in length with a minimum diam 6 1/3 ft. One end of the gallery is open, while the other, known as the head, is closed by a steel disc of the same diam as the gallery, provided in the center with a hole 12" in diam. The gallery is built of boiler-plate steel in five divisions, each consisting of three similar sections, 6 2/3ft long. The first three sections are made of ½" steel plates, while the remainder is of 3/8" steel plates. The cylinders forming each section are held together by lap joints. Each section has on its top a porthole, provided with a hinged cover. This can serve as a safety valve (to prevent the destruction of the gallery in case of an expln), and also as an approximate means of estimating the pressure developed within the gallery. The first three sections counting from the head of the gallery (making total length of 20ft) are usually separated from the open portion by means of a paper diaphragm. The closed section, thus formed, is known as the explosion chamber. This chamber is covered with an insulating jacket to assist in maintaining a constant temp (usually 20-30°C) before each test. Inside the interior of the gallery are shelves of 3/8" steel, 4" wide & 20ft long on which the coal dust is placed. Four of these shelves are placed, one above the other and 8" apart, on each side of the gallery, to which they are attached by means of brackets or studs. For observing the progress of any flame produced in the gallery during testing, each section is provided, on its side, with a thick plate glass window. The cannon is a cylinder 24" in diam by 36" long, with a bore-hole 21.5" deep mounted on a 4-wheel truck with runs on a 30" gage track (Ref 14, pp40-3). After loading the cannon with a explosive to test, the mount is rolled against the steel head of the gallery and a close fit is made betw the muzzle of the cannon and the steel ring which is found inside the hole of the head. The cannon is arranged so that the axis of its borehole coincides with the longitudinal axis of the gallery. Then the explosion chamber is filled with mixt of natural gas/air to form the atmosphere contg 8.0 ± 0.3% of methane + ethane and the cannon is fired straight into the chamber (Ref 14, pp49-53)

The following permissibility gallery tests are required for newly submitted expls, by Bur Mines regulations listed in Schedule I-H (Ref 45):

15.10(3) Gallery test 4. Ten trials, each with a 1.5-lb(680g) tamped but unstemmed charge (See Note below) of expl are made, firing each chge from the above described steel cannon into the explosion chamber contg 8 lbs of bituminous coal dust placed on the shelves and a natural gas/air mixt contg 4.0 ± 0.2% methane with some ethane. The temp of chamber should be 25° ± 5° before each firing. No ignition of mine atmosphere should take place in 10 trials (Ref 14, p54; Ref 45, p3 & Ref 47)

Note: Accdg to Dr. R.W. Van Dolah (Ref 47),

in the usual language of miners, **tamping** and **stemming** tend to get confused. In this case, however, tamping means slitting the cartridges, and deforming them to completely fill the borehole by pressing on them with a wooden rod. Stemming is inert material added after the explosive charge (in the borehole) to provide some confinement

15.10(4) Gallery test 7. This is briefly described in Ref 45, but not in Ref 14 nor in other BurMines publications. Here the value W_{50}, which is the weight for 50 percent probability of ignition, is detd. Accd to Dr. Van Dolah (Ref 47), the gallery used in test 7 is essentially the same as gallery 1 described in Ref 45; it has, however, been enclosed to reduce noise effects, The value W_{50} is detd by using the Bruceton up-and-down method(See Note below) and firing a minimum of 20 tamped chges of varying wts, stemmed with 1 lb of dry-milled plastic fireclay, from a steel cannon into the gallery's expln chamber atmosphere contg 8.0±0.3% of Bureau's natural gas, at a temp of $25^{\circ} \pm 5^{\circ}$. The expl should yield a W_{50} value equal to or greater than 450g to 95% confidence (Ref 45, pp3 & 4). Any new expl compn submitted for approval as permissible that fails gallery test 7 will be rejected and not submitted to gallery test 1, as it is done in case of field samples (Ref 47)

Note: Accdg to Dr. Van Dolah (Ref 47), the **up-and-down method**, first developed in 1944, has been most commonly used for the impact sensitivity test of expls, but can be applied to many other test methods, including the gallery tests

Accdg to W.J. Dixon & F.J. Massey, Jr, "Introduction to Statistical Analysis", McGraw-Hill, NY (1957), 319-21, the technique is as follows, when applied to sensitivity to impact:

Procedure: Choose some initial height h_0 and a succession of heights h_1, h_2, h_3...above it together with a succession of heights h_{-1}, h_{-2}, h_{-3}....below h_0. If the 1st sample explodes at h_0, test the 2nd sample at h_{-1} and if it does not explode, use h_1 for the 2nd sample, etc. In general, a sample should be tested at the level immediately below or immediately above the level of the previous test depending on whether there was or was not an expln in the previous test. The primary advantage of this method is that it automatically accumulates testing values near the mean and thus increases the accuracy with which the mean can be estimated

In the case of gallery tests, the weights W_0, W_1, W_2, W_3... and W_{-1}, W_{-2}, W_{-3}... are used instead of heights

Other permissibility tests, which are not gallery tests, are briefly described at the beginning of this section, under item A -Chemical Tests and item B-Physical Tests (See also Ref 45, pp3 & 4)

The gallery tests listed below are required for **field samples.** These are of a regularly manufd and previously approved brand of permissible expls removed by the Bureau's representative from a mining company magazine or directly from the field. These samples are collected at any period after acceptance as "permissible explosives" in order to determine whether they continue to conform to the basic specifications of BurMines (Ref 47)

15.21(3) Gallery test 4. The sample must pass 5 shots (instead of 10 required for newly submitted samples) with a tamped but unstemmed chge of 1.5lb (680g) (Rer 45, p5 & Ref 47)

15.21(1) Gallery test 7. This test is conducted in the same manner as described for newly submitted expls, paragraph 15.10(4). The field sample must yield a W_{50} value equal to or greater than 450g to 95% confidence (Ref 45, p5)

Any field sample which is approved as a newly manufd permissible expl after Feb 21, 1961, the date of issuance of Schedule I-H, is declared nonpermissible if it fails to pass gallery test 7. This requirement will not apply, however, during a period of 5 years, following the issuance of Schedule I-H, to any sample approved under prior Schedules, such as I-G. If these samples fail gallery test 7, but pass all other tests, the gallery test 1 will be the next step (Refs 45 & 47)

Paragraph 15.21(2) Gallery test 1. This test is conducted by firing (from a steel cannon) ten 220g tamped and stemmed charges into a gallery atmosphere contg $8.0 \pm 0.3\%$ of the Bureau's natural gas, at a temp of $25 \pm 5^{\circ}$. The stemming consists of one lb dry-milled plastic fireclay. If no expln results, the expl remains on permissible list. (Ref 45, p5) (Compare with Ref 14, p53)

Any field sample failure will be immediately reported to the applicant and he must immediately remove from the market any unused portions of the explosive bearing the same lot number as the sample tested. If field samples of 3 different lots of the same brand of explosive fail within a period of 5 years, that brand will be declared nonpermissible and removed from the list of permissible explosives

Refs: 1)(?)Mente & W.Will, JSCI **28**, 748 (1909) (Experiments with expls in regard to the expln of firedamp and coal dust) 2)W. Will, SS **4**, 323-27 & 343-49 (1909) (Contribution to testing of expls against firedamp) 3)C. Hall et al, USBurMinesBull **15** (1912) 171 & 173 (Testing of coal mining expls) 4)C.Hall & S.P.Howell, Ibid **66** (1913) (Tests of permissible expls) 5)C.G.Storm, Ibid **96** (1916) (Analysis of permissible expls) 6) Marshall **2** (1917), 584-91 (Testing in galleries of coal mining expls) 7)S.P.Howell & J.E. Tiffany, USBurMinesTechPaper **186** (1918) (Testing of mining expls in physical lab of BurMines) 8)Barnett (1919), 121-30 (Test galleries) 9)C.A.Taylor & W.H.Rinkenbach, USBurMinesBull **219** (1923) (Expls, their material, compn and analysis) 10)G.S.Rice et al, USBurMinesBull **268**, 1-168 (1927) & CA **21**, 2986 (1927) (Coal-dust expln test in the experimental mine from 1919 to 1924 incl) 11)Naoúm, NG(1928), 386-98 (Testing of coal mining expls in galleries) 12)J.F.Thorpe, Safety in Mines Research Board (Brit) Paper **51** (1929) (Testing of expls for use in fiery coal mines) 13)W.Payman, TransInstMining-Eng **80**, 11-23 (1930-31) & CA **25**, 1675 (1931) (Researches on coal-mining expls at the Safety in Mines Research Station, Bruceton, Pennsylvania) 14)C.E. Munroe & J.E.Tiffany, USBurMinesBull **346** (1931) (Physical testing of expls at the BurMinesExplsExptlSta, Bruceton, Pa) 15)H.C.Grimshaw & W.Payman, Safety in Mines Research Board Paper **69** (1931) (Gallery experiments on ignition of firedamp by coal mining expls) 16)Vennin, Burlot & Lécorché (1932), 231-51 (Essais des explosifs antigrisouteux) 17)Marshall **3** (1932), 186-90 (Testing galleries) 18) Stettbacher (1933), 248-52 (Schlagwetter -Versuchsstrecken) 19)G.S.Rice et al, USBurMinesBull **369** (1933) (Tests in exptl mine, 1925 to 1932 incl on coal mining expls) 20)C.E.Munroe & J.E.Tiffany, IEC **27**, 655-58 (1935) (Review of the work conducted at the BurMines in testing permissible expls) 21) Pepin Lehalleur (1935), 408-11 (Galleries d'essais) 21a)Beyling & Drekopf (1936), 3 & 107-09 (Ger testing galleries) 22)E. Audibert, AnnMines **12**, 278-98 (1937) & CA **32**, 3616 (1938) (A review of tests conducted in France since 1880 on the ignition of firedamp on firing expls) 23)B.Lewis & G. von Elbe, USBurMinesResInvest **3463** (1939) (Ignition of firedamp by expls) 24)S.L.Gerhard et al, Ibid **3464** (1939) (Ignition of firedamp by expls) 25)Thorpe **4** (1940), 108-14 & 557 -62 (Testing galleries); 558-61 (General investigation of conditions governing the safe use of coal-mining expls) 26)J. Formánek, HornikýVéstnik, **22**, 241-43, 256-60, 269-78 & 281-87 (1940) & CA **37**, 6462 (1943 (Detn of the explosibility of firedamp) 27)Pérez Ara (1945), 255-58 (Galerías de ensayo o de prueba); 258-64 (Diversos explosivos de seguridad) 28)I.Hartmann & H.P.Greenwald, ProcCoalMiningInstAmerica (Pittsburg) **1946**, 68-84 (Tests of permissible expls in the experimental coal mine) 29)H.P.Greenwald et al, USBurMinesTechPaper **716** (1949) (Tests on the ignition of natural gas-air mixts by permissible expls in exptl coal mine) 30)R. L.Grant et al, RevIndMinérale **32**, 486-90 (1951) & **33**, 884-92 (1952) (Research on the mechanism of ignition of firedamp by expls) 31)H.Ahrens, Sprengtechnik **1952**, 101-07, 131 -34 & 159-65 (Investigation of factors influencing the safety of permissible expls against firedamp) 31a)Belgrano (1952), 168 -71 (Galleria di prova degli esplosivi antigrisoutosi) 32)J.W.Gibson et al, Safety in Mines Research Establishment, Research Rept **47**, July 7-12 (1952) (Procedures for assessing the relative safety of expls and of coal dust/inert dust mixts by means of gallery experiments) 33)R.L.Grant & C.M.Mason, USBurMinesReptInvest **5049** (1954) (Studies of mechanism of ignition of firedamp by expls) 34)R.L.Grant et al, Ibid **5050** (1954) (A new method of measurement of the incendivity of expls to firedamp) 35)Bureau of Mines, Schedule **26A**, "Blasting Devices", Federal Register, Mar 22, 1955, vol 20, No 56 36) Yaremenko & Svetlov (1957) (Testing of per-

missible expls) 37)M.Taillandier & G. Thozeau, RevIndMinerale **39**, 591-96 (1957) & CA **51**, 17168 (1957) (Test method for the evaluation of the transmission of detonation of charges in blasting of coal) 38)K.Fischer, ZElektrochem **61**, 685-92 (1957) & CA **52**, 2410 (1958) (Studies of coal dust explns in exptl gallery 250m long) 39)W.Cybulski, PlaceGlównegoInstGórnictwa No **197**, (1957) & CA **52**, 4181 (1958) (Investigation of shooting some AN-based expls in galleries with settled coal dust) 40)McAdam & Westwater (1958), 34-9 (Testing of expls at the Safety in Mines Research Establishment's Testing Station at Buxton, England) 41)Taylor & Gay (1958), 33-43 & 86-96 (Gallery testing in GtBritain & other countries 42) Giua, Trattato **6**(1), 450-51 (Galleria di prova degli esplosivi antigrisoutosi) 43)Andreev & Belyaev (1960), 492-97 (Testing of coal mining expls in galleries and experimental mines) 44)R.L.Grant & R.W.VanDolah, US BurMinesReptInvest **5760** (1961) (Statistical analysis of gallery variables affecting the probability of ignition by expls) 45)Bureau of Mines Schedule **1-H**, Title **30** - Mineral Resources, Subchapter **C** - Explosives and Related Articles; Tests for Permissibility and Suitability", USGovtPrtgOff, Washington,DC (1961) 45a)StdMethodsChemAnalysis, **2B** (1963), Chapter 32 46)Dr. Omero Vettori, Chieri (Torino), Italy; private communication, July 20, 1964 47)Dr. R.W.VanDolah, USBurMines, Research Director, Explosives Research Center, Pittsburgh, Pa; private communications, July 31, 1964 and August 17, 1964 48)C.Giorgio, "Tecnica degli Esplosivi", DelBianco, Udine (Italy) (1964), 96-101

Coal, Nitrated (Nitrohouille in Fr). When powdered coal was treated with nitric acid of d1.40 to 1.48 a nitrated product of brown color was produced. As consumption of acid was very great, it was decided that prepn of nitrated coal is uneconomical
Refs: 1)Cundill (1889) in MP **5**, 308 (1892) 2)Daniel (1902), 556 (Nitrohouille)

Coal Oil. See under COAL PROCESSING

Coal Pitch, Nitrated. See under Coal Tar and Coal Tar Pitch

Coal Powders. These are black powders in which charcoal is replaced by coal, as for example, in compositions developed at Pic-Arsn by Varrato (Refs 1 & 2). Such powders burn slower than corresponding charcoal contg BkPdrs and for this reason are suitable for use in slow-burning safety fuses or in time fuzes. By blending them with suitable charcoal pdrs, various intermediate rates of burning may be obtd

The composition described in Ref 3 as "Powder, Black, Slow-burning", consists of K nitrate 70.0 ± 1.0, coal (semibituminous) 14.0 ± 1.0 & Sulfur 16.0 ± 1.0%. It has been used in time fuzes, such as "45sec Combination Fuze" and "M-54 Fuze"

US military requirements and tests for this powder are as following:
a)Granulation - 98% must pass thru No 140 US StdSieve
b)Moisture - max 0.7%. It is detd by heating for 4hrs a 2g sample in a tared weighing dish, cooling in a desiccator and weighing
c)Potassium nitrate is detd by washing a 10.0g sample, placed on a tared sintered glass crucible, with 10-15ml portions of distd w until nitrate is completely removed. After passing ca 200ml of w, its last portion is tested by adding concd sulfuric and a few crysts of DPhA. The absence of blue coloration indicates the completeness of washing. Then the crucible and contents are dried for 4hrs at 70°, cooled in a desiccator and weighed. The loss of wt, multiplied by 10 gives percentage of K nitrate
c)Sulfur is detd by extracting for 1 hour with carbon disulfide, the contents of the above crucible placed in a Soxhlet or Wiley extractor. Then the crucible is removed, rinsed with alcohol, followed by ether, dried for 1 hr at 100-105°, cooled in a desiccator and weighed. The loss of wt, from previous weighing, multiplied by 10 gives percentage of sulfur
d)Coal is detd by subtracting from 100 the sum of percentages of K nitrate and sulfur
e)Burning time is determined when fuzes are tested statically, using a chronograph. Burning time for powder in 45sec Combination Fuze at a setting of 45 should be not less

than 50 sec and for M-54 Fuze not less than 36 sec at a setting of 25. (See also "Coal Powder" in Vol 2, pB171-R of this Encycl)
Refs: 1)P.Varrato, "Study of Blending of Coal Powder and Fast Grade A7 Powder for Use in the 21sec Fuze", PATR **142** (1931) 2)P.Varrato "Development of 30sec Powder for 21sec Time Fuze", PATR **243** (1932) 3)US Joint Army-Navy Specification, JAN-P-540 (1947), Powder, Black, Slow-burning

Coal Processing for Obtaining More Valuable Products. (Kohlenveredlung in Ger). Under this title can be included the following processes:
a)Carbonization (Refs 1,2,4,5,7,8,9,10 &12)
b)Chemical treatments (such as with acids or alkalies) (Refs 2,5,8, & 11)
c)Extraction with solvents (Ref 3)
d)Gasification (Refs 1,2,8,10 &11)
e)Hydrogenation or liquefaction (Combination with hydrogen at high temp in presence of a catalyst) (Refs 2,6,8,10 & 13)

The purpose of these processes is to obtain products which are, in many cases, more valuable than coal itself

These products, called *by-products of coal*, include coal gas, coal oil, coal tar, coal tar pitch, coal tar resins and coke
Coal gas is obtd by destructive distillation of bituminous coal heated to high temps in fire-clay or silica retorts, or in special by-product ovens. The gas is used in domestic & industrial heating & lighting and as a source of coal-tar, ammonia, benzene, toluene, xyline and related items (Refs 1,2,8,10 & 11)
Coal oils and other liquids, such as crude oil, illuminating oil, gasoline, kerosene, etc can be obtained either by destructive distillation of bituminous coals or by hydrogenation of coal or coal tar (Refs 6,10 & 11)
Coal tar is described separately
Coal tar pitch is described separately
Coke is described separately
Refs: 1)Thorpe **5** (1941), 364 (Carbonization); 367-78 & 430-80 (Gasification) 2)H.H.Lowry, "Chemistry of Coal Utilization", Vols 1 & 2, Wiley, NY (1945) 3)H.H.Lowry & H.J.Rose, USBurMinesInfoCirc **7420** (1947) (Description of Pott-Broche process and the plant of Ruhröl GmbH, Bottrop-Welheim, Germany for extracting coal with solvents at elevated temps. This gave nearly ash-free coal suitable for manuf of high-grade electrode carbon or for hydrogenation in order to obtain liquid hydrocarbons) 4)Kirk & Othmer **3** (1949), 160-78 (Carbonization) 5)P.G.Wilson & J.H.Wells, Coal, Coke and Coal Chemicals", McGraw-Hill, NY (1950) 6)Kirk & Othmer **7** (1951) 701-05 (Coal hydrogenation) 7)E. Rammler & H. von Alberti, "Braunkohlenschwellung und Braunkohlenverkokung"(Low Temperature Carbonization and Coking of Brown Coal), VEB Vlg für Buch- and Bibliothekswesen, Leipzig (1956) 8)Ullmann **10** (1958), 183 (List of products obtd on processing coals); 213 & 229-30 (Low temp gasification of coals);241-458 (Gasification of coals); 483-569 (Hydrogenation of coal and of heavy oil); 581-88 (Chemical treatments of coal) 9)M.Gomez, IEC **52**, 717-20 (Aug 1960) [A summary of significant developments in coal carbonization (pyrolysis)] 10)CondChemDict (1961),219 (Carbonization of coal); 283 (Coal gas); 284 (Coal hydrogenation or liquefaction); 284 (Coal oil) 11)EncyclBritannica **5** (1963), 973 & 976-79 (Coal by-products) 12)EncyclBritannica **6** (1963), 36-8 (High temperature carbonization) 13)EncyclBritannica **11** (1963), 973-76 (Hydrogenation)

Coal Tar and Coal Tar Pitch. When a bituminous coal is heated at temps 900 to 1200°C, in the absence of air, the collected distillate consists of *coal tar* (ca 6% of wt of coal) and of solid residue called *coke*. Coal tar is usually a black, viscous, sticky, heavier-than-water liquid with a characteristic naphthalene-like odor and a sharp burning taste. The crude product has been used for waterproofing, pipe-coating, roofing, road paving, etc (Refs 2,7,8,9,11,12 & 13). It has also been used as a fuel binder in some compns such as in one of the cheddites (See Vol 1, pC157, table 2 of this Encycl) and in some pyrotechnic compns. For example, some Ital smoke-producing compns contained tar (catrame in Ital). Izzo (Ref 6, p238) lists the following black smoke mixture: K chlorate 31, coal tar 47, naphthalene 11 & red phosphorus 11%

Rotten patented in 1892 expls contg coal

tar oil, as for example: a)AN 77.1, naphthalene 6.2, tar oil 6.2 & varnish 10.5% b)K picrate 38.8, anthracene 38.8, tar oil 6.9 & varnish 15.5% (Ref 1, p695). Some current Rus mining expls, such as *Ammonite-goudronite* contain AN & coal tar (See Vol 1, pA310-L of Encycl & Ref 10)

Schultze patented in 1886 expl compns contg up to 15% of *nitrotar* obtd by adding, with stirring, 1 part of crude coal tar into 15 parts of nitric acid (d 1.42-1.46) and heating the slurry on a water bath until the disappearance of acid fumes. The resulting foamy-like expl product was used, without purification, in compns, such as: a)Mining explosive K nitrate 75, nitrated tar 15, NC 10 & sulfur 10ps andb)Sporting powder Ba nitrate 45, NC 40, nitrated tar 9 & K nitrate 6% (Ref 1, p708). The expl called **Emilite**, which was patented in 1887 by Audouin contained a product prepd by nitration of the fraction of coal tar boiling betw 185 & 200°(Ref 1, p256)

Distillation of coal tar yields: light oil (contg benzene, toluene & xylene), middle oil (contg phenol, cresol & naphthalene), heavy oil (contg impure cresol & other phenols) and green oil (contg anthracene) (Refs 4,9,11 & 13)

The distillation residue, amounting to 50-60% of the usual grades of coal tar, is a dark brown to black amorphous solid melting at ca 150°F (65.6°C) known as *coal tar pitch*. It has been used as a thermoplastic, in roofing and in road surfacing (Refs 9,11 & 12). Some commercial expls contain pitch which serves the same purpose as coal tar. For example the Rus mining expl *Ammonpek* consists of AN 95 & coal tar pitch (pek in Rus) 5% (See Vol 1, pA382-L of this Encycl & Ref 10)

Accdg to Daniel (Ref 1, p556) nitration of coal tar pitch (poix de houille in Fr) with weak nitric acid produced an expl which was patented in 1886 by Emile et al for use in mixts with K nitrate or chlorate in the manuf of some mining expls. Nitration of pitch with stronger acids proved to be too dangerous

Coal tar pitch has also been used in some pyrotechnic compns, as for example, in red light signal compn listed by Izzo (Ref 6, p 207), which contains K chlorate 76, Sr oxalate 8, pitch (pece in Ital) 9 & shellac 7%. Another Ital pyro compn contg pitch is the black smoke mixture: K chlorate 60 & Pitch 40% (Ref 6, p238)

Hydrogenation of coal tar is discussed in Refs 3,7 & 11

Refs: 1)Daniel (1902) 256 (Emilite); 556 (Nitrated coal tar pitch); 695 (Rotten's expls contg coal tar); 708 (Schultze's expls & proplnts contg nitrated coal tar) 2)Thorpe **3** (1939), 207-14 (Coal tar) 3)Thorpe **6** (1943) 371-2 (Coal tar hydrogenation to obtain gasoline & light oils) 4)Davis (1943), 129 (Coal tar) 5)E.Rhodes, USBurMinesInfoCirc **7409** (1947) (German high-temperature coal tar industry) 6)Izzo, Pirotecnia (1950), 207 & 238 7)Kirk & Othmer **7**, 698-701 (Coal tar hydrogenation) 8)H.Winkler, "Der Steinkohlenteer und seine Aufarbeitung", Glückauf-Vlg, Essen (1957) 9)Kirk & Othmer **13** (1954), 614-38 (Coal tar, under Tar and Pitch) 10)B.T.Fedoroff et al, PATR **2145** (1955), pRus2 11)Ullmann **10** (1958), 208-16 & 285-95 (Coal tars) 12)Cond-ChemDict (1961), 284 (Coal tar); 284 (Coal tar pitch) 13)Encycl Britannica **5** (1963), 976-79 (Coal tar)

Coating of Explosive, Propellant, and Pyrotechnic Composition. If an expl compn contains one or several hygroscopic ingredients (such as AN, Na nitrate, etc), the compn can be rendered moistureproof by coating hygroscopic ingredients with waterproofing agents. These agents may include wax, paraffin, stearic acid, lanolin , collod cotton, dextrin, gelatin, casein, bentonite clay, resins, protein, etc. Some coating methods for AN are described in Vol 1, pA334-L of this Encycl (See also Refs 8,9,10,14,18,21,23,24,25,28 & 29)

If it is desirable to increase the sensitivity to initiation of an expl compn, the particles of its insensitive ingredients (such as AN, Al, etc) can be coated with HE's (such as TNT, Tetryl, PETN or RDX) dissolved in volatile solvents (See Refs 3,14 & 18)

Some pyrotechnic compns contain ingredients (such as chlorates, picrates, etc) which in combination with powdered metals (such as Mg) are rather sensitive to mechanical action and dangerous to handle. These compns can be made less sensitive and at the same time more waterproof by coating the ingredients

with wax, paraffin, stearic acid, etc (See also Refs 11 & 19)

Composite proplnts contg hygroscopic ingredients (such as AN) can be treated in the same manner as is done in the case of expl compns (See also Ref 13)

Coating of homogeneous (single- and double-base) proplnts can achieve several purposes, such as slowing down the rate of burning, waterproofing and stabilization

In the prepn of progressive burning single-base proplnts, the flakes or single-perforated short cylinder are coated with a non-volatile, non-explosive solid or liquid, compatible with NC, dissolved in a volatile solvent, such as benzene or acetone. The disolved material and proplnt grains are tumbled together in a "sweetie barrel" or a similar apparatus and then the slurry is heated to evaporate the solvent, while the tumbling is continued. The resulting proplnt grains are more progressive burning than uncoated grains, because the coating slows down the initial rate of burning. The materials used for this type of coating are called **deterrents** in USA and **moderants** in GtBritain. The most important of these materials are Centralites (See Vol 2, ppC126ff of this Encycl). Other deterrents include DBuPh (dibutylphthalate), DMeDPhU (dimethyldiphenylurea), DNT, EtPh (ethylphthalate), MePh (methylphthalate), etc (Ref 1, pp319-22). To these may be added Acardites which are described in Vol 1, ppA7ff of this Encycl (See also Refs 2,5,7,12,15,26,30 & 31)

Large proplnt grains, such as used in rockets, may be coated with noncombustible materials in order to restrict burning to the desired surface (see Refs 16,17 & 27)

A method for prevention of diffusion of NG thru coating of DNT in double-base proplnts was patented by Hale (Ref 6); method for detn of completeness of coating with wax was proposed by Rowen & Phillips (Ref 8); and method of polarographic analysis of a coated US military proplnt was described by Ribaudo (Ref 12)

Refs: 1)Davis (1943), 319-22 & 328-29 (Coated proplnts and coating agents) 2)T.Thomson USP 2407967 (1946) & CA **41**, 865 (1947) (Coated progressive, single-base proplnt is obtd by tumbling its grains in an alcoholic soln of pentaerythritol tetraacetate) 3)E.G. Cockbain, BritP 574879 (1946) & CA **43**, 1984 (1949) (Desensitized expls are produced by coating particles of sensitive expls, such as PETN, with insolubilized protein or protein derivs) 4)Brevets Aéro-Méchaniques SA, SwissP 244840 (1947) & CA **43**, 6418 (1949) (Coating of fine particles of sensitive, water-insol expls with dil aqueous colloidal suspensions of dextrin, casein, gelatin, etc) 5) D.C.Lindsten, USP 2432578 (1947) & CA **42**, 7046 (1948) {Coating of single- or double base proplnt grains with w-insol ester of dicarboxylic aliphatic acid [such as di (butyl cellosolve) tartrate], which melts below 100°} 6)G.C.Hale, USP 2440267 (1948) & CA **42**, 5229 (1948) [To prevent NG in double-base proplnts from diffusing into the surface coating (such as DNT), the NG used in prepn of proplnt is saturated with the same material as used for coating] 7)J.Fauveau & R. Delpy, MP **31**, 161-66 (1949) & CA **46**, 11686 (1952) (Description of improvements in methods of coating of rifle proplnt grains and of ball powders) 8) J.W.Rowen & A.J.Phillips, USP 2531471 (1950) & CA **45**, 2670 (1951) (Completeness of coating of HE's grains with wax can be detd by examining them under UV radiation. The uncoated particles are purple colored whereas the coated material has a yel-white or a blue-white fluorescence) 9)A.LeRoux, MP **33**, 265-82 (1951) (Coating AN expls for waterproofing with ca 1% Ca stearate soln) 10)J.Whetstone, IEC **44**, 2663-67 (1952) (Description of several methods for coating AN particles for waterproofing and preventing them from caking) 11)M.Schliemann, GerP 873513 (1953) & CA **52**, 14172 (1958) (Safe & waterproof pyrotechnic compns can be obtd by coating their ingredients with protein-contg substances) 12)C.Ribaudo, "Polarographic Determination of Dibutylphthalate in Coated M-18 Propellant", PATR **2119** (1955) 13)C.A.Thomas, USP 2742672 (1956) & CA **50**, 11019 (1956) (Each grain of a blend consisting of a finely divided oxidizer and a solid fuel of a composite proplnt is coated with

a liquid thermosetting resin) 14)Dynamit AG, GerP 938594 (1956) & CA **53**, 1716-17 (1959) (Coating of hygroscopic expls, such as based on AN, with solns of aromatic or aliphatic nitrocompds or nitramines) 15)J.Cohen & E.D.Besser, USP 2779288 (1957) & CA **51**, 6159 (1957) (Grains of proplnts are kept for 2hrs at $<40^{\circ}$ in aq alc contg 10% Amm sulfide soln, followed by washing with w and drying. This treatment forms a flame-inhibiting coating consisting of regenerated cellulose) 16)R.L.Hirsch et al, USP 2780996 (1957) & CA **51**, 6160 (1957) (The burning of solid rocket proplnt is restricted to the desired surface by depositing an alkyd-resin coating over the sides of the cast proplnt stick) 17)G.W.Miller et al, USP 2813487 (1957) & CA **52**, 4183 (1958) (Coating of extruded rocket proplnt grains on selected surfaces by inhibiting substances, such as a mixt of Methyl- & Ethyl Cellosolve) 18)Dynamit AG, BritP 788311 (1957) & CA **52**, 7703 (1958) (AN based expls are coated with surface active agents, such as nitroisobutylglycerin; DNPh, Tetryl, TNT, hexamine, etc. Coating may either retard or promote the detonation) 19)R.H.Comyn, USP 2832704 (1958) & CA **52**, 14516 (1958) (Pulverized metals used in pyrotechnic compns can be protected from moisture by immersing them for 30mins in a 5% Na or K dichromate soln, drying and immersing in a soln of stearic acid) 20)R.S. Gow & J.F.Williamson, BritP 796709 (1958) & CA **52**, 19144 (1958) (The flow and antistatic props of PETN are improved by coating the discrete particles with a w-sol, nonionic, surface active agent, particularly one having a polyoxyethylene deriv of a higher aliphatic alc, an org acid amide, etc) 21)E.I.duPont de Nemours & Co, BritP 806278 (1958) & CA **53**, 7597 (1959) (Waterproof blasting expls are prepd by compressing their chief ingredients AN & Na nitrate into spherical or cylindrical pellets and coating them with a plasticized NC compn) 22)E.von Holt & G.Koch, GerP 1029275 (1958) & CA **54**, 12589 (1960) (Particles of an expl are coated with a layer of a lower melting expl at a temp betw the mp's of both components) 23)G.Boyd & A.McIntosh, Australian P 213095 (1958) & CA **54**, 16833 (1960) (Powdery expls contg grains of w-sol oxidizers are waterproofed by coating with lanolin) 24)P.Aubertein, FrP 1180530 (1959) & MP **41**, 475-77 (1959) (Prepn of waterproof and insensitive to mech action metal-contg expl compns by incorporating the metal previously coated with w-insol compd. For expampl in expl called **Hexal**, finely powdered Al particles are coated with wax or paraffin prior to their mixing with powdered RDX preheated to $60\text{-}70^{\circ}$) 25)E.I.duPont de Nemours, Brit P 810759 (1959) & CA **54**, 3958 (1960) (Waterproof blasting expls are obtained by coating the spherical pellets of AN & Na nitrate, 250 mils in diam, with a flexible coating 12 mils thick. The coating consists essentially of PVC in a tetrahydrofuran soln) 26)A. Darche, GerP 1057513 (1959) & CA **55**, 10891 (1961) (Coating the outer surface of tubular molded cannon proplnts with a colloidal, viscous soln of Centralite) 27)W.E.Campbell Jr & L.H.Brown, USP 2958288 (1960) & CA **55**, 5958 (1961) (Coating of rocket proplnt grains to restrict the burning surface, by dipping in a bath composed of bentonite clay, alkyd resin styrene mixture with 0.5% of ter-Bu hydroperoxide as polymerization activator) 28)S.D.Rolle, USP 2982642 (1961) & CA **55**, 20436 (1961) [Coating of pellets of expl compns consisting of carbon, sulfur and K nitrate or of carbon and K perchlorate by lead (using the vacuum-sputtering, film forming techniques) in order to raise their autoignition temp] 29)A.Bertmann & E.Ludolphy, GerP 1092357 (1961) & CA **55**, 25255 (1961) (Coating of w-sol ingredients of expl mixts with stearic acid dissolved in ethanol or by other substances) 30)W.G. Stanley, USP 2987388 (1961) & CA **55**, 25259 (1961) (Coating of proplnt grains with a mixt of cellulose acetate & Ac-tri-Me citrate) 31)A.Gey, USP 2994597 (1961) & CA **55**, 27893 (1961) (Slow burning proplnt is obtd by coating

its grains with cured polysulfide polymer)

COBALT AND ITS COMPOUNDS

Cobalt, Co, at wt 58.95, steel-gray metal, d 8.71 at 20°, mp 1495°, bp 2880°; readily sol in nitric acid and less sol in dil hydrochloric or sulfuric acids. It is obtained from ore concentrates by roasting, followed by thermal reduction by Al or other methods. The principal use of the metal is in alloys, especially Co steels for permanent supermagnets, Co-Cr high-speed tool steels, cemented carbides and high temperature-resisting alloys for jet engins. For a description of its alloys see Refs 1, 2, 3, 4, 5, 6 & 8. *Cobalt Bomb* is briefly discussed under Bombs in Vol 2, pB239-R

Many Co salts and Co complexes are known; some of them are explosive.
Refs: 1)Gmelin, Syst Nr **58** Teil A, (1932) & Teil B (1930) 2)Mellor **14** (1935), 419 -859 3)Thorpe **3** (1939), 214-22 4)R.S. Young, "Cobalt", Reinhold, NY (1948), ACS Monograph No 108 5)Kirk & Othmer **4** (1949), 189-215 6) Ullmann **9** (1957), 609-26 7)Sax (1957), 491-98 (Toxicity of Co compds) 8)Gmelin, Syst Nr **58**, Teil A, Ergänzungsband (1961) 9)Cond-ChemDict (1961), 284-89 10)P.Queneau "Extractive Metallurgy of Copper, Nickel and Cobalt", Interscience, NY (1961), 5 5 475 **Cobalt 60.** (Co^{60}). An artificially obtained radioactive isotope, Co of mass number 60, emmitting both gamma & beta rays. Its gamma rays have about the same penetrating power as those from radium. Half life is 5.2-5.3 years. Its radiotoxicity is moderate. It has been used in medicine, in research, for radiographic testing of welds, as a source in portable radiation units and in activation (radioactivation) analysis (See Vol 1, pA99)
Refs: 1)Kirk & Othmer **11** (1953), 480-93 (Radiochemistry) 2)Sax (1957), 491 3)H. Etherington, Edit, "Nuclear Engineering Handbook", McGraw-Hill, NY (1958), Section 14 (Isotopes) 4)Gmelin, Syst Nr **58**, Engänzungsband, Teil A (1961), 213 5)CondChem-Dict (1961), 284-85

Cobalt, Analytical Procedures. See Vogel, InorgAnalysis (1961), 528-32 and StdMethods-ChemAnalysis **1** (1962), 377-94

List of Cobalt Compounds

Cobalt Acetylide. See Vol 1, pA72-R

Cobaltammine Complexes. Thirty complexes are described in Vol 1, ppA278 to A281, under Ammines
Addnl Refs: A)U.Alvisi, Gazz **31** II, 281-301 (1901) & JCS **82** II, 24 (1902) B)R.Salvadori, gazz **40** II, 9-18 (1910) & JCS **98** II, 959 (1910) C)Gmelin, Syst Nr **58**, Teil B (1930) D)Mellor **14** (1935), 688 E)W.R.Tomlinson, Jr, PATR **1364** (1943) F)W.R.Tomlinson, Jr & K.G. Ottoson, PATR **1632** (1946) G)W.R.Tomlinson et al, JACS **71**, 376 (1949)

Cobalt Azide. See Cobalt Triazide in Vol 1, pA531-L

Cobalt Azide Complexes. See Vol 1, p531-R

Cobalt(III)bis(diethylenetriamine) Perchlorate or Bis(diethylenetriamine) Cobaltic Perchlorate, $\{Co[(NH_2.C_2H_4)_2\}(ClO_4)_3$. This complex was prepd during WWII at the Univ of Illinois and then investigated at PicArsn. No method of prepn is given. The following props are reported: expln temp 325°; sand test value 39,4g (vs 42.0 for TNT); impact sensitivity 21cm, BurMines app, 2kg wt; requires 0.07g LA for complete deton; 100° heat test-loss of wt 1st 48hrs 0.30%, 2nd 48hrs 0.0% and no expln in 100 hrs; 100° vacuum stability test 0.16ml/1g/40hrs
Refs: 1)W.R.Tomlinson Jr & K.G.Ottoson, PATR **1632** (1946) 2)W.R.Tomlinson et al, JACS **71**, 376 (1949)
Note: Not found in Mellor, Gmelin or Beil

Cobalt Chlorate. See Cobaltous Chlorate in Vol 2, pC187-R

Cobalt Chromate. See under Chromates in this Vol, p C279-R

Cobalt-Chromiun Acetoacetonate, $CoCr(CH_3.CO.CH_2COO)_5$(?). This compd is listed by Swotinsky without giving its formula, method of prepn, props or refs) as one of the ingredients of experimental rocket proplnts, as a ballistic modifier to replace

Pb stearate. For example: NC(13.25%N) 58.5, NG 22.5, DNT 2.5, triacetin 8.5, Et-Centr 8.0 & CoCr acetoacetonate 9.5%
Refs: 1)Beil - not found 2)CA's (1907-1960) - not found 3)J.M.Swotinsky, PATR **1806** (1951)

Cobalt Compounds for Smoke Production. DeMent (Ref) proposed to use the following Co ion contg mixts as smoke-producing pyrotechnic compns: a)Co stearate 2.0, K chlorate 10.0 to 12.0 & Amm acid fluoride 4.0 to 8.0 parts. This compn, when ignited by a flame, produced a heavy, voluminous fine white smoke b)When using monochloroacetic acid in previous mixt, instead of fluoride, the color of smoke produced was blue-white c)Equal parts of Co chloride, K bromate, K bromide, sulfur, K chlorate & K acid sulfate burned with the emission of a green to green-brown smoke d)Equal parts of Co nitrate, K bromate, K bromide, sulfur, K chlorate & K acid sulfate burned with the emission of grn to grn-brn smoke e)A mixt of Co chloride 1.0, K chlorate 2.0, K iodate 1.0, K iodide 1.0 & sulfur 1.5 parts burned with evolution of a heavy voluminous deep violet smoke
Ref: J.DeMent, USP 2995526 (1961), p8

Cobalt(III)dichlorodiethylenediamine Chlorate and **Cobalt(III)dichlorodiethylenediamine Perchlorate.** Thes two compds are listed by Tomlinson et al (Ref) without giving their formulas, methods of prepn or props, except some expl props as detd at PicArsn. The props are for the 1st compd: expln temp 320° & sand test value 13.7g (vs 42.0g for TNT) and for the 2nd compd: expln temp 300°, sand test value 9.9g and impact sensitivity (BurMines app, 2kg wt) 85cm (vs 95cm for TNT)
Ref: W.R.Tomlinson, Jr et al JACS **71**, 376
Note: If these compds are salts of Cobalt-(III)dichlorobis(ethylenediamine), their formulas should be $[Co(en)_2Cl_2]ClO_3$ and $[Co(en)_2Cl_2]ClO_4$

Cobalt(III)dinitratobis(ethylenediamin) Nitrate [called Dinitratodiäthylendiaminkobalt(III) nitrat in German]. $[Co(en)_2(NO_2)_2]NO_3$; N 26.84%; mp - decomp explosively above 129°. Can be prepd by treating $[Co(en_2(H_2O)].(NO_3)_3.2H_2O$ with malonic acid or by other methods
Refs: 1)A.Werner & C.Kreuzer, Ann **351**, 67 & 79 (1907) 2)W.Schramm, ZAnorgChem **180**, 170 (1929) 3)Gmelin, Syst Nr **58**, Teil B (1930), 227

Cobalt(III)dinitrobis(ethylenediamine) Nitrates [called Dinitrodiäthylendiamin kobalt (III)-nitrat in German], $[Co(en)_2(NO_2)_2]NO_3$; N 29.43%. Prepn and some props (except explosive) of 1, 2- and 1, 6-dinitro isomers are given in Refs
Refs: 1)A.Werner & E.Humphrey, Ber **34**, 1721 & 1727 (1901) 2)A.Werner, Ber **44**, 2452 (1911) 3)Gmelin, Syst Nr **58** Teil B (1930), 217-18 & 220-21

Cobalt(III)dinitrobis(ethylenediamine) Nitrite, [called Dinitrodiäthylendiaminkobalt(III)nitrit in German] $[Co(en)_2(NO_2)_2]NO_2$; N 30.96%. Prepn and some props (except explosive) of 1, 2- and 1, 6-dinitro isomers are given in:
Refs: 1)A.Werner & E.Humphrey, Ber **34**, 1720 & 1726 (1909) 2)Gmelin, Syst Nr **58**, Teil B (1930), 217 & 220

Cobalt(III)dinitrobis(ethylenediamine) Perchlorate, [called Dinitrodiäthylendiamine-kobalt(III) perchlorat in Ger] $[Co(en)_2(NO_2)_2]ClO_4$; prisms or plates, , mp - not given. Can be prepd by treating corresponding nitrate with concd perchloric acid soln. Its expl props were not detd
Refs: 1)A.Werner, Ber **44**, 2453 (1911) 2) Gmelin, Syst Nr **58**, Teil B (1930), 218-19

Cobalt(III)hexahydroxylamine Nitrate, $[Co(NH_2OH)_6](NO_2)_3$; N 28.4%; golden yel crysts, mp -decomp explosively on heating. Was prepd by adding dropwise concd nitric acid (free from N oxides) to ice cold soln of cobalt(III)hexahydroxylamine chloride
Refs: 1)A.Werner & E.Berl, Ber **38**, 897-88 (1905) 2)W.R.Tomlinson, Jr et al, JACS **71**, 376 (1949)

Cobalt(II)hydrazine Chlorate, $Co(ClO_3)_2.2N_2H_4$; col crysts, mp - expl at 85-90°; expl also on the slightest percussion or friction. Can be prepd either by treating

$Co(ClO_3)_2$ with hydrazine hydrate in alc soln or by other methods. Because of its extreme sensitivity only a small quantity should be prepd at a time
Refs: 1)R.Salvatori, Gazz **40** II, 9-18 (1910) & CA **5**, 1568 (1911) 2)W.Friederich & P.Vervoorst, SS **21**, 105 & 124 (1926) 3)Gmelin, Syst Nr **58**, Teil B (1930), 23

Cobalt(II)hydrazine Nitrate or Cobaltous Trihydrazine Nitrate, $Co(NO_3)_2.3N_2H_4$; brownish pdr; mp - expl ca 210°; exlp also on impact, friction or contact with concd sulfuric acid; decomp in presence of moisture. Can be prepd by treating cobaltous hexammine nitrate with 50% hydrazine hydrate or by other methods. It is difficult to ignite
Refs: 1)H.Franzen & O.von Mayer, ZAnorgChem **60**, 247 & 274 (1908) 2)Gmelin, Syst Nr **58**, Teil B (1930), 12 3)Mellor **14** (1935), 827 4)L.Médard & J. Barlot, MP **34**, 160 & 162-64 (1952); CA **48**, 6125 (1954)

Cobalt(II)hydrazine Nitrite, $2Co(NO_2)_2.3N_2H_4$; dull red pdr, explodes on heating or on contact with concd sulfuric acid. Was obtained by interaction of ice-cooled solns of Co acetate with Na nitrite
Refs: 1)P.Rây & B.K.Goswami, ZAnorgChem **168**, 331 & 337 (1927) 2)Gmelin, syst Nr **58**, Teil B (1930), 10

Cobalt(II)hydrazine Perchlorate, $Co(ClO_4)_2.3N_2H_4$(?); col crysts (from alc), hydrolyze in air turning pink; mp - expl ca 215°. Can be prepd by treating, in alc soln, $Co(ClO_4)_2$ with $N_2H_4.H_2O$. Only a small quantity of complex should be prepd at a time, because it is very sensitive to impact or friction. Was patented by Friederich for use in initiating compns
Refs: 1)W.Friederich, GerP 341063 (1920) 2)W.Friederich, & P.Vervoorst, SS **21**, 86, 104 & 125 (1926) 3)Gmelin, Syst Nr **58**, Teil B (1930), 24

Cobalt Hydroxides. See under Cobalt Oxides & Hydroxides

Cobalt Naphthenate, approx formula $Co(RCOO)_2$, where R is largely a mixt of trimethylcyclopentane alkane radicals; Co content ca 6%, mp ca 125° d ca 0.96 and flash p ca 100°(in open cup); insol in w; sol in benz, alc, eth & oils. Can be prepd by treating cobaltous hydroxide or acetate with one of the naphthenic acids. These acids, derived from some petroleums, are mixts of various cyclopentane monocarboxylic acids, contg on the average ca 12 carbon atoms (Refs 1 & 4) (See also Ref 3)

Specification requirements for Co naphthenate for US Government uses are given in Spec No **TT - D - 643** and in Ref 4, p103; properties of "Cobalt Naphthenate DMR" (Cobalt 6%), manufd by the Nuodex Products Co, Elizabeth, NJ, are listed in Ref 4, p104

Co naphthenate has been used in some pyrotechnic compositions as a catalyst with "Laminac 4116" and "Lupersol DDM" (Ref 4, pp104, 157 & 183) (See also Ref 2). It has also been used as an accelerator, when it was required to increase the rate of polymerization of some rocket proplnt fuels (See Vol 1, pA108-R of Encyclopedia)
Refs: 1)E.R.Littmann & J.R.M.Klotz, ChemRevs **30**, 97-11 (1942) (Naphthenic acids; manuf, props & uses) (129 refs) 2)A.R. Lusardi & J.Wingler, PATR **2516** (1958), Table 1 3)CondChemDict (1961), 287 & 777-78 4)Anon, "Properties of Materials Used in Pyrotechnic Compositions", US Army Materiel Command Pamphlet, **AMCP 707-187**, Washington, DC, 20315 (1963), 103-4 (Co naphthenate); 157-8 (Laminac 4116) and 183-4 (Lucidol)

Cobalt(II) Nitrate or Cobaltous Nitrate, $Co(NO_3)_2.6H_2O$; mw 291.05; red monocl crysts; mp 57°, decomp at higher temps; sol in w, alc or acetone. Can be prepd by the action of nitric acid on Co hydroxide. This salt is an oxidizer dangerous in contact with organic materials; has been used in prepn of Co catalysts, Co pigments, etc
Refs: 1)Gmelin, Syst Nr **58**, Teil A (1932), 253-56 2)Mellor **14** (1935), 821-22 3)Kirk & Othmer **4** (1949), 206 4)Sax (1957), 496 5)CondChemDict (1961), 287 6)Gmelin, Syst Nr **58**, Teil A, Ergänzungsband (1961), 515-17

Cobalt(II)nitratonitroethylenediamine Nitrate,

$[Co(en)_2(NO_2)(NO_3)]NO_3$; N28.1%, lt yel pdr, mp - not given; was prepd by treating 1, 2-chloronitrodiethylenediamminocobalt with Ag nitrate. Its expl props were not detd
Refs: 1)A.Werner & L.Gerb, Ber **34**, 1744 (1901) 2)Gmelin, Syst Nr **58**, Teil B (1930) 259

Cobalt Nitrides. The following Co-N compds are described in the literature: Co_3N_2, Co_4N_2, Co_3N, Co_2N and CoN. Of these only the *cobaltous nitride*, Co_3N_2, mw 204.84, N13.72%, is expl. It was obtd by Bergstrom (Ref 2, p2632) by heating cobaltous amide, $Co(NH_2)_2$, in a vacuum above 120°; dissolves in dil H_2SO_4 with decompn. Its expl props were not investigated
Refs: 1)G.T.Beilby & G.G.Henderson, JCS **79**, 1251 (1901) 2)F.W.Bergstrom, JACS **46**, 2632-33 (1924) 3)Gmelin, Syst Nr **58**, Tl A (1932), 250-51 4)Mellor **14** (1935) - not found 5)Gmelin, Syst Nr **58**, Teil A, Erganzungsband (1961), 511-12

Cobalt Nitrites. *Cobaltous Nitrite*, $Co(NO_2)_2$, red crysts, mp-decomp on slight heating; was first prepd in 1848 (Ref 1) on treating an aqueous suspension of Co pdr with Ag nitrite soln. Several other methods of prepn are listed in Ref 2. There are also known *basic cobaltic nitrite* and *basic cobalto-cobaltic nitrite* (Ref 2), but no cobaltic salt, $Co(NO_2)_3$
Refs: 1)W.W.Fischer, PoggAnn **74**, 121 (1848) 2)Gmelin, Syst Nr **58**, Teil A (1932) 252

Cobalt Nitrite Complexes. When KNO_2 is added in excess to a soln of $Co(NO_2)_2$, acidified with AcOH, a yel ppt of *potas sium hexanitrocobaltiate (potassium cobaltinitrite)*, $K_3[Co(NO_2)_6]$ forms. This compd is also known as Fischer's Salt or as a pigment Cobalt Yellow. Formation of above complex can serve as the basis of a volumetric detn of Co ion. When a sol K salt is added to *sodium cobaltinitrite*, $Na_3[Co(NO_2)_6]$, a yel cryst ppt *potassium-sodium cobaltinitrite* $K_2Na[Co(NO_2)_6].H_2O$ is formed. This complex, being practically insol in w, can be used as the basis for detection of K ion
Refs: 1)Gmelin, Syst Nr **58**, Teil A (1932), 400, 409 & 411 2)Kirk & Othmer **4** (1949) 210-11 3)Gmelin, Syst Nr **58**, Teil A, Ergänzungsband (1961), 513-15 4)CondChemDict (1961), 288

Cobalt Oxides and Hydroxides. The follwoing compds are known: Cobaltous Oxide CoO & its Hydroxide, $Co(OH)_2$; Cobalto-Cobaltic (or Cobaltosic) Oxide, Co_3O_4 & its Hydrate, $Co_3O_4.xH_2O$; Cobaltic Oxide, Co_2O_3 and its Hydrate $Co_2O_3.3H_2O$. There are also some oxides and hydroxides of Co whose compns have not definitely been established. Oxides and hydroxides can be used for prepn of Co salts, as pigments in paints & ceramics and as catalysts. Prepn & props of oxides and hydroxides are described in refs
Refs: 1)Gmelin, Syst Nr **58**, Teil A (1932), 224-49 2)Mellor **14** (1935), 558-600 3)Kirk & Othmer **4** (1949), 206-08 4)Sax (1957), 493 & 495-96 5)CondChemDict (1961), 285-88 6)Gmelin, SystNr **58**, Teil A, Ergänzungsband (1961), 476-511

Cobalt Perchlorate or Cobaltous Perchlorate, $Co(ClO_4)_2$, mw 257.85, red ndls, d 3.327; sol in w, alc or acet. It is an oxidizing compd. No method of prepn is given in Refs 2 or 3. Accd to Ref 4, p581, it cannot be prepd by dehydration of its hexahydrate, $Co(ClO_4)_2.6H_2O$, or of dihydrate, $Co(ClO_4)_2.2H_2O$. "Cobaltic perchlorate, $Co(ClO_4)_3$, exists only in solns (Ref 4, p583)
Refs: 1)Mellor **14** (1935) - not listed 2)Sax (1957), 496 3)CondChemDict (1961), 288 4) Gmelin, Syst Nr **58**, Teil A Ergänzungsband (1961), 581-83

Cobalt Picrate or Cobaltous Picrate, $Co[C_6H(OH)(NO_2)_3]_2$; brown pdr (Refs 1, 3 & 4) or dk red crysts (from alc) (Refs 2 & 5); mp expl with moderate violence at 320-25°. Can be obtained on heating to ca 150° any of its hydrates, such as with 2-, 6- or 9.5-H_2O (Refs 1 & 3). Tetra - and pentahydrates are also reported (Ref 1). Bernardi & Piacentini (Refs 2 & 5), reported prepn of anhydrous salt by treating picric acid in ethanol with Co hydroxide

Refs: 1)Beil **6**, 278 2)Beil **6**, [264] 3)O. Silberrad & H.A.Phillips, JCS **93**, 487-88 (1908) 4)Colver (1918), 332 5)A.Bernardi & G.Piacentini, Gazz **56**, 130 (1926) & CA **20**, 2296 (1926)

Cobalt Resinate or Cobaltous Resinate, $Co(C_{44}H_{62}O_4)_2$; mw 1368.81, brn-red pdr, spontaneously flammable in air; reacts with oxidizing materials; insol in w, sol in oil. Can be prepd by cautiously heating a cobaltous salt and rosin oil
Refs: 1)Sax (1957), 496 & 497 2)CondChemDict (1961), 288

Cobalt Stearate or Cobaltous Stearate, $Co(C_{18}H_{35}O_2)_2$; pink amorph pdr, mp 73-5o (Refs 1 & 2); Lawrence (Ref 3) gives 200° as solidification point. Its solubilities in various solvents are given in Ref 2, pp354-56. Can be prepd by the methods similar to prepn of Cr stearate (Refs 2 & 3). It was investigated at PicArsn (Ref 4) as an additive to semi-plastic RDX compns, such as Comp A-3 & Comp A-4 (See under *Chromium Stearate*)
Refs: 1)Beil **2**, (172) & [1011] 2)A.E. Koenig, JAC **36**, 952-56 (1914) 3)A.S.C. Lawrence, TrFaradSoc **34**, 665 & 667-71 (1938) 4)A.L.Forchielli, PATR **1787** (1950)

Cobalt Sulfates. Both cobaltous and cobaltic sulfates are known, of which the *cobaltous sulfate*, $CoSO_4$, or its hydrates are used in industry, such as in baths for Co plating, ceramics, pigments, glazes and in catalysts. Its prepn & props sulfates are described in refs
Refs: 1)Gmelin, Syst Nr **58**, Teil A (1932), 324 & 336 2)Mellor **14** (1935), 750 & 787 3)Kirk & Othmer **4** (1949), 208-09 4)Sax (1957), 493 & 497 5)CondChemDict (1961), 288 6)Gmelin, Syst Nr **58**, Teil A, Ergänzungsband (1961), 628-41

Cobalt Tetracarbonyl (Dicobaltocarbonyl), $Co_2(CO)_8$, mw 342.0, mp 51°, bp-decomp at higher temp with evoln of poisonous CO; d 1.78; insol in w, sol in alco or eth. Can be prepd by treating finely divided Co with CO under pressure; used in high anti-knock gasoline. It can react with oxidizing materials
Refs: 1)Sax (1957), 498 2)CondChemDict (1961), 289

Cobalt(III)tris(ethylenediamine), Chlorate, $[Co(en)_3](ClO_3)_3$. This complex was prepd during WWII at the Univ of Illinois and then investigated at PicArsn. No method of prepn is given. The following props were reported: expln temp 215°; sand test value 39.3g, vs 42.0g for TNT; impact sensitivity 7 cm, BurMines app, 2kg wt; stability was not detd.
Refs: 1)Gmelin, Syst Nr **58**, Teil B (1930) - not found 2)Mellor, **14** (1935) - not found 3)W.R.Tomlinson Jr & K.G.Ottoson, PATR **1632** (1946), Tables I & II

Cobalt(III)tris(ethylenediamine) Nitrate, $[Co(en)_3](NO_3)_3$; dk red or brn-red crysts; mp - not given; d 1.709 at 25°/4°. Can be prepd by heating Co hexammino nitrate (or nitropentammino nitrate) with an aq soln of ethylenediamine monohydrate. Its impact sensitivity test is 61cm, BurMines app, 2kg wt and sensitivity to initiation > 0.4g Tetryl
Refs: 1)Mellor **14** (1935), 832 2)W.R.Tomlinson Jr & K.G.Ottoson, PATR **1632** (1946), Table I 3)W.R.Tomlinson et al, JACS **71**, 376 (1949)

Cobalt(III)tris(ethylenediamine) Perchlorate, $[Co(en)_3](ClO_4)_3$; br-red crysts, mp - 335°, d 1.88 at 25°/4°. Can be prepd by treating cobalt(III)tris(ethylenediamine) bromide with Ag chlorate (Ref 1). Another method of prepn is given in Ref 2, p4. Its impact sensitivity is 17cm, BurMines app, 2kg wt; sand test value 40.1g, vs 42.0g for TNT; can be initiated with 0.03g LA; 100°heat test - no loss in wt in 48+48hrs and no expln in 100hrs; 120° vacuum stability test 0.33ml/5g/40hrs
Refs: 1)Gmelin, Syst Nr **58**, Teil B (1930), 76 2)W.R.Tomlinson & K.G.Ottoson, PATR **1632** (1949), Tables 1 & 2

Cobalt(III)tris(ethylenediamine) Peroxysulfate, $[Co(en)_3]_2(S_2O_8)_3$. It was reported by Beacom, that this compd can be ignited by flame and exploded by exposing it to UV radiation (3650Å), followed by heating to 120°. Its method of prepn was not given by Beacom; and we could not find this compd listed in

the alphabetical indexes of CA's (except the listing indicated in Ref 1
Note: en is abbreviation for ethylenediamine
Refs: 1)S.E.Beacom, Nature **183**, 38-9 (1959) & CA **53**, 11087 (1959) 2)Anon, C&EN **37**, No 15, p70 (April 15, 1959)

Cobra. Designation of a wire-guided A/T missile with a shaped-charge warhead
Ref: OrdTechTerm (1962), 75

COC. Abbr for Cleveland Open Cup, a type of flash point test

Cochise No 1. One of the dynamites developed at Apache Powder Co, Benson, Arizona, ca 1925: NG 18.0, drip oil 2.0, Na nitrate 66.0, woodpulp (of low absorption value) 4.0, coal dust 9.0 & marble dust 1.0%. Its strength by ballistic pendulum 83% TNT and gap test 36+"
Ref: Dr. I.A.Grageroff, Keystone Ordnance Works, Meadville, Pennsylvania; private communication (1944)

Cocking, Allen T. Brit expls specialist associated for many years with the Kynoch Ltd, Essex, England. His numerous patents on expls and proplnts date from 1896 to 1922. Following are some of his patents: a)A.T. Cocking & Kynoch Ltd, EngP 18034 (1896) & JSCI **16**, 825 (1897) (Improvements in manuf of NG) b)Ibid, EngP 22190 (1896) & JSCI **16**, 760 (1897) (Apparatus for drying Cordite and for recovery of solvents) c)Ibid, EngP 22717 (1896); JSCI **16**, 934 (1897) & Daniel (1902), 148 (An improved method for mixing and conveying acids used in manuf of NG and other expls) d)Ibid, EngP 28889 (1896); JSCI **16**, 1039 (1897) & Daniel (1902), 398 (Safety expl contg NG 25-27, woodmeal 40-43, Ba nitrate 30-36, Amm oxalate 0.5 10 & Na carbonate 0.5 (ormore)%) (This expl was called, accdg to Daniel, **Kynite**) e)Ibid, EngP's 28890 & 28891 (1896); JSCI **16**, 1040 (1897) & **17**, 182 (1898) (Improvements in manuf of NG) f)Ibid, EngP 19650 (1897); JSCI **17**, 949 (1898) & Daniel (1902), 149 (Proposed the placing of wads impregnated with concd AN soln in the bore-hole above and below the expl chge. This arrangement cooled the gases of expln (due to the absorption of heat by the volatilized NH_4Cl & H_2O) to below the temp of ignition of firedamp)
Note: A similar idea was employed in the so-called *Flameless Cartridge Case*, also invented by Cocking. It consisted of a wool bag filled with expl chge and inpregnated with Amm chloride soln [Daniel (1902), 150] g)A.T.Cocking & Kynoch Ltd, EngP 22965 (1901) & JSCI **21**, 1412 (1902) (Hardening of granulated smokeless proplnts by action of solvents, such as acetone, in the form of a vapor) h)Ibid, EngP 22966 (1901) & JSCI **21**, 1556 (1902) (Reduction of bulk of mining expls by replacing woodmeal with starch. Eg: NG 25, starch 40.5, Ba nitrate 33 & Ca carbonate 0.5%) i)Ibid, EngP 457 (1903) & JSCI **22**, 1366 (1903) (Improvement in manuf of Cordite) j)Ibid, EngP 15053 (1904) & JSCI **24**, 814 (1905) (Manuf of "bulk" powder, using 12-12.5% N-NC dissolved in 4-5 parts of acetone, followed by adding double the bulk of water at 65°) k)Ibid, EngP's 12892 (1905), 15564 (1905), 15565 (1905), & 15566 (1905); JSCI **25**, 337, 497 & 609 (1906) (Improvements in manuf of smokeless proplnts) l)Ibid, Eng P 21779 (1905) & JSCI **25**, 953 (1906) (Manuf of " bulk" powder) m)Ibid, EngP 25081 (1905) & CA **25**, 1175 (1906) (Solvents for NC used in manuf of Cordite) m)Ibid, EngP 16667 (1906); JSCI **26**, 717 (1907) & CA **1**, 2197 (1907) (Process of manufg NC) n)A.T.Cocking, USP, 903958 (1908) & CA **3**, 717 (1909) (Smokeless proplnt contg NC 50-60, NG 30-40, vaselin 2.5, olive oil 2.5, Ba tartrate 4.1 & K tartrate 0.9%) o) A.T.Cocking & Kynoch Ltd, BritP's 2836 (1911) & 1722 (1911); CA's **6**, 1992 (1912) & **7**, 417 (1913) (Nitration of glycerin to which sugar is added) p)A.T.Cocking, BritP 22311 (1911) & JSCI **7**, 1101 (1913) (Manuf of expls by incorporating NC with aqueous emulsions of nitrocompounds, such as TNT or TNB, followed by adding NG and heating with steam; H_2O is removed by decantation, draining, pressing & heating) q)Ibid, BritP's 28177 & 28178 (1911); CA **7**, 1975 (1913) (Process for manufg plastic expls, such as blasting gelatins) r)Ibid, USP 1164170 (1915) & CA **10**, 391 (1916) (Blasting gelatin prepd by mixing NG with wet NC, heating and removing H_2O under reduced pressure) s)A.T.Cocking & Kynoch Ltd, BritP 130038 (1918) & CA **14**, 210

(1920) (Concn of dil nitric acid obtained from spent acids in manuf of expls) t)A.T.Cocking & C.H.Lilly, GerP 472870 (1921) & CA **23**, 3300 (1929); USP 1425838 (1922) & CA **16**, 3364 (1922) (Manuf of glycerin by fermentation)

Cocoa Powder, also known as *Chocolate Powder* or *Brown Powder.* See Vol 2, pB173-L, under Black Powder Modifications. Marshall, Dict (1920) 23 gives the following compn: K nitrate 79, straw charcoal 18 & sulfur 3%

Coconut Fiber Dynamite. It was obtd in 1905 by Gonsalves by soaking coconut fibers in NG. It was claimed that this dynamite was not affected by low temps
Ref: Clift & Fedoroff **2** (1943), pC12

Coefficient d'absorption de NC. In this test, developed by J.Desmaroux, there is detd the amt of liquid such as ether-alcohol mixt, which can be absorbed by NC
Procedure: Digest for 24 hrs in a glass tube 1.5g NC with 25ml of eth-alc such as 60% eth & 40% alc of 95.2% strength. Centrifuge for 15 mins at 2100rpm and decant the liquid into a small graduate. Calculate the coeff from the formula (25-n)/1.5, where n is vol of decanted liquid, in ml
Ref: (?)Thomas, MP **25**, 118-19 (1932-33)

Coefficient d'échange (Fr) (Exchange Coefficient). In order to det if a NC is sufficiently stabilized, Vieille detd titrimetrically the amt of acid (expressed as mg of H_2SO_4) removed from each 100g of NC in each hour of washing with w or dil alkali solns. He found that as soon as the coeficient drops to ca 5 (from original of about 150), the stabilization may be considered as completed. This method was improved by using pH meters for detn of acidity
Refs: 1)P.Vieille, MP **22**, 307-19 (1926) 2) A.Koeler & M.Marqueyrol, MP **22**, 320-25 (1926) 3)Vennin, Burlot & Lécorché (1932), 399-400

Coefficient de gélatinisation or Pouvoir gélatinisant (Fr) (Gelatinization Coefficient or Gelling Power). In order to determine the amts of various substances required to cause gelatinization of 100g of NC, the following tests were developed in France:

a)*Test of Marqueyrol & Florentin* (Ref 1), modified by Davis (Ref 3): Add to a 0.2g sample, 1ml of a 2% alc soln of the gelatinizer under study, provided it is nonvolatile. Evap the alc at 60° and examine the sample. If it is not completely gelatinized, add another 1ml of alc soln of gelatinizer and evap the alc. Repeat these opns until the sample is completely gelatinized. The coefficient is given as the parts of the gelatinizer required for complete gelatinization of 100g sample, such as NC (see also Ref 4)
b)*Test of Ab der Halden.* It is briefly described under Ref 3, pC116-L, Vol 2 of this Encyclopedia
Refs: 1)M.Marqueyrol & D.Florentin, MP **18**, 151-67 (1921) 2)J.Desmaroux, MP **18**, 168-82 (1921) 3)Davis (1943), 320-21 4)J. Tranchant, MP **32**, 288 (1950)

Coefficient de gonflement or Pouvoir gonflant (Fr) (Swelling Coefficient or Swelling Power). Its definition and test method are briefly described in Vol 2 of Encyclopedia, pC116-R under Ref 14

Following are some coefficients for CP_1 (NC of 12.9 to 13.4% N), as detd by Thomas:

Liquid for Swelling	Grams per 100g of CP_1	Coefficient of Swelling
Water	10 to 100	0
Ether	10 to 100	0.65
Alcohol	20	4.4
(95.2%)	30	6.4
	40	6.8
Alcohol	20	7.0
(85.3%)	30	7.5
	40	5.9
Alcohol	20	6.75
(75.6%)	30	5.45
	40	4.3

Ref: (?)Thomas, MP **25**, 115-20 (1932-33)

Coefficient de rangement (Fr) (Arrangement Coefficient). This term was introduced by Tavernier, who investigated the relationship betw the geometrical forms of various proplnts (such as balls, cords, strips, tubes, etc) and their loading densities. For detailed info on this subject, see the Ref
Ref: P.Tavernier, MP **31**, 199 (1949)

Coefficient de self-excitation (CSE); Transmission de la détonation à distance or Aptitude de transmettre la détonation à distance (Fr) (Coefficient of Excitation; Transmission of Detonation at a Distance or Ability to Transmit the Detonation at a Distance). This property known also as *Sympathetic Detonation* or as *Detonation by Influence* is detd in the US by the so-called *gap test*

The French test is conducted in the following manner: Two cartridges of expl to test, 30mm diam and each weighing 50g, are placed upon two lead plates, supported by two steel vertical cylinders. The cartridges are placed a certain distance apart (say 5 to 20cm) in line with axes coinciding. Then one of the cartridges is detonated from the end not facing the 2nd ctge and if the other ctge is not detonated by influence, the distance btwn the ctges is reduced by a few cm. After several attempts, the max distance is determined at which there are 3 successive detonations of the 2nd ctge. After this, the minimum distance is determined at which there are three successive failures to deton the second ctge. The mean of the two distances is the CSE (Refs 1-3)

Following are some CSE values, as reported in Ref 3:

Explosive	Density g/cm²	CSE cm
Picric Acid	1.0	13.5
" "	1.40	7.5
TNT	1.0	7.0
Tetryl	1.0	16.0
"	1.40	12.0
"	1.70	6.5
RDX	1.20	21.0
"	1.60	20.0
"	1.78	9.5
PETN	1.20	21.0
PETN & 7.5% wax	1.0	14.0
PETN 50 & TNT 50%	1.10	14.0

Refs: 1)E.Burlot, MAF **9**, 799 (1930) 2)M. Dutour, MP **31**, 74 (1949) 3)L.Médard, MP **33**, 342-44 (1951)

Coefficient d'utilisation pratique (CUP or cup) or Travail spécifique relatif (FR) (Coefficient of practical utilization or Relative specific work). This property of expls, which may be considered to express relative power or strength is detd in France by modified Lead Block or Trauzl Test. The Trauzl Test was considered by Davis (Ref 2, p25) to determine largely the brisance, but for expls of substantially equal brisance it gives some indication of their relative power. A brief description of the method is given in Vol 1, ppIX-X of this Encycl, where six refs are given. A detailed description of the method is given in Ref 1 & 3 listed here. It is claimed by Médard (Ref 3), that Fr method gives reliable results with aluminized expls, although with original Trauzl test the results are higher than reasonably expected (Ref 2, p25). This test is called by Stettbacher (1933), 363 "Französisch Bleiblockausbachung Methode"

Following are some CUP values given by Médard (Ref 3, p351): PA 100; Amm picrate 81; DEGDN 130; DNCB 99; m-DNB 88.5; DNPh 70; 2,4-DNT 69, EGDN 160; HNDPhA 104; HNDPhSulfide 100; Methyl Nitrate 157; NG 150; Nitroisobutanetriol Trinitrate 157; PETN 146.5; RDX 135; 1,2,3,5-TeNA 110.5; Tetryl 115.5; TNAns 97, TNB 103.5, TN-m-Cresol 88, TNN 70; TNPhenetole 86, TNResorcinol 95.5, TNT 94 & TN-m-Xylene 82

Refs: 1)E.Burlot, MAF **13**, 113 (1934) 2) Davis (1943), 24-5 3)L.Médard, MAF **22**, 597-605 (1948) (See also Refs in Vol 1, p X of this Encycl)

Coefficient de viscosité des nitrocelluloses industrielles, called also **Unité MB**, was proposed in 1934 by General Goujon as a value for comparing viscosities of industrial NC's. It is the angular coefficient of the tangent at the origin of the curve: log z=f(c), when c approaches zero. In this equation (based on the work of Staudinger), z is relative viscosity at 20° of collodion (prepd by dissolving in butyl acetate the NC to test) and c is the wt of NC in 1g of collodion

Ref: N.Moreau & Y.Lacroix, MP **32**, 443, Footnote (1950)

Coefficient de vivacité des poudres (Quickness coefficient of propellants). Accdg to Vennin et al (Ref 1, p100), it is the value dP/dt, where P is the pressure in tons

developed on burning of a proplnt in a closed bomb and t is duration of burning in seconds. Pepin Lehalleur (Ref 2, p86) defines the coefficient as the quotient $(dp/dt\ max)/P^2 max$, where (dp/dt max) is the angular coeffecient which can be detd from the pressure -time curve obtained during closed bomb examination of burning of a proplnt, on a graph paper attached to rotating drum of registering manometer. The pressure Pmax is measured by a crusher gage placed inside the bomb during the test. For description of test see Refs 1 & 2
Refs: 1)Vennin, Burlot & Lecorché (1932), 100 & 102 2)Pepin Lehalleur (1935), 86-7

Coefficient of Gelling (Gelatinization) of Soler & Vian. Its definition and test method are briefly described in Vol 2, pC117-L under Ref 37

Coefficient of Thermal Conductivity or Specific Heat Conductivity (λ) is the quantity of heat transmitted per second thru a plate of material 1cm thick and $1cm^2$ in area, when the temp difference between the two sides of the plate is one degree centigrate. Some values are given in Ref and under individual compds described in this Encyclopedia
Ref: Clift & Fedoroff, Vol 2 (1943), Table of Physical Constants of Compounds Used in Explosives Industry and Definition of Terms Used in Table of Physical Constants [See also S. Nagayama & Y.Mizushima KKK **21**, 8-11 (1960); CA **55**, 9877 (1961) & Explosivst **1964**, 21]

Coefficient of Thermal Expansion is the ratio of the increase in length (linear), area (superficial), or volume (cubical) of a body for a given rise in temperature (usually from 0° to 1°C), to the original size. Some values are given in Ref and under individual values a

given in Ref and under individual compds described in this Encyclopedia
Ref: Same as in previous item

Coefficient of Thermal Expansion (Linear) of 47 plastic materials used in US Ordnance Corps items, were detd over a temp range from -30° (-22°F) to +30° (86oF) at Pic-Arsn by R.W.Kuchkuda and listed in PATR **2025** (1954)

Cohete (Span). Rocket

Cohete guiado (Span). Guided Rocket (Guided Missile)

Coke (Coke in Fr or Ital; Cok or coque in Span; Koks in Ger or Rus). Coke is the solid residue which remains when certain types of bituminous coals are heated in retorts or special ovens (such as "behive" type) to high temps out of contact with air until practically all of the volatile constituents are removed. Coke consists principally of C and of small amts of S, H, N, O & mineral matter present in the original coal. It is a hard, cellular form, porous substance, ranging in color from silvery gray to dull black. Its true density 1.85 -1.90, is higher than that of coal, but its bulk d is lower because of porosity

The name coke is also applied to solid residues obtained from various other carbonaceous materials, such as petroleum, shale oil, copal, tar, etc (Refs 1, 2, 3, 6, 7, 8, 10 & 11)

The process of prepg coke is known as carbonization or coking

Accdg to Kirk & Othmer (Ref 2, pp158-60), the carbonization process can be subdivided into:
High temperature process in which final coke temps ranging from 1500 to 2000 °F are obtained, the average being 1900°F (1037.8°C), when carbonization is completed. This process is the one most commonly used
Low temperature process in which the final coke temp averages 1000°F (537.8°C) and
Medium temperature process in which final coke temps ranging from 1200 to 1300°F (648.9 to 704.4°C) are attained
See also Carbonization in Vol 2, pC62-L of this Encycl

The types of coke and by products (such as gas & tar) differ with the type of carbonization process. The coke obtd from high temp process is the least reactive and has the greatest structural strength. For these reasons, it is suitable for use as blast-furnace and cupola -furnace fuels. The coke obtd from low temp process contains considerable amt of liquid ingredients, but produces little smoke on burning. It is considered very suitable as a domestic fuel, because it is easy to ignite and, when

dampered, it holds the fire much better than high temp coke. The medium temp coke has props intermediate betw high & low temp cokes. It is used as domestic fuel

Finely pulverized cokes have been used as ingredients of expls or pyrotechnic compns. In the numerous compns listed in Vol 2, pp C55 to C59, carbon can be replaced by coke and the same may be applied to expls contg coal (See Coal Dust and Its Uses)

In some compns, coke is specified as an ingredient as, for example, in Black Dynamite described in Vol 2, pA165-L of this Encycl. Other examples are the following **Dahmenits** which are listed in Ref 1, p28: a)Neu-Dahmenit A AN 68, K nitrate 2, vegetable meal 2.5, coke 2, TNT 10 & alkali chloride 15.5% and b) Neu-Dahmenit B AN 65, K nitrate 2, vegetable meal 0.5, coke 7, TNT 8 & alkali chloride 17.5%

Giua (Ref 9) lists the expl of **Golovine**, patented in 1905, which contained 3% coke, the rest being K chlorate 62, K borate 29, Amm chloride 3 & glycerin or petroleum 3. One of the older US primary mixts contained coke. Its compn and tests were described in Specification **50•11•21** (1923), which is not available now

Refs: 1)Marshall, Dict (1920), 28 1a)Thorpe **3** (1939), 258-76 (Coke manuf and the recovery of by-products) 2)Kirk & Othmer **3** (1949), 158-60 & 165-67 (Coke manuf) 3)P.G.Wilson & J. H.Wells, "Coal, Coke and Coal Chemicals," McGraw-Hill, NY (1950) 4)Kirk & Othmer **6**, (1951), 899 (Coke as a fuel) 5)H.Gesner & H.R.Zbinden, SchweizVerGas - und WasserfachMonatsBull **32**, 45-52 (1952) & CA **47**, 7196 (1953) (Tests with coke dusts have shown that it is practically impossible to ignite their mixts with air. A weak ignition was observed only once in 15 tests) 6)W.Gluud, "Handbuch der Kokerei," K.Knapp, Düsseldorf (1953) 7)O. Grosskinsky, "Handbuch der Kokereiwesens," K.Knapp, Düsseldorf, vol 1 (1955) & vol 2 (1958) 8)Ullmann **10** (1958), 241-55 & 269-85 (Coking of coal) 9)Giua, Trattato **6** (1) (1959), 398 (An explosive contg coke) 10)Merriam-Webster's (1961), 441 (Coke) 11)Encycl Britannica **6** (1963), 35-8 (Coking, coke and high temperature carbonization)

Coke, Analytical Procedures. Most of procedures are similar to those used for coal and they are described in Refs 1 & 2

Refs: 1)ASTM Stds **1961**, Pt 8: Method **D141•48**, pp1361-63; Method **D271•58**, p1238; Method **D294•50**, pp 1366-67; Method **D346•35**, pp 1349-54 and methods listed under Coal, Analytical Procedures 2)StdMethodsChemAnalysis, vol **2A** (1963), 1149 (Sample prepn for coke); 1151 (Moisture detn); 1152 (Ash detn); 1158 (Sulfur); 1159 (Phosphorus in ash); 1160-65 (Carbon & hydrogen); 1165-68 (Nitrogen) and 1168/70 (Oxygen)

Cold Extrusion of Metals. See under Cold Working or Shaping of Metals

Cold Working or Shaping of Metals. This general process of metal fabrication is concerned with the plastic deformation of a metal, without the application of heat, into useful forms by such mechanical means as hammering, pressing, rolling, extrusion & shearing of the metal. The *Cold Extrusion or Cold Flow* (Kaltspitzen in Ger) is a process developed in Germany ca 1935 by the "Kabell und Metallwerke Neumeyer AG", at Nürnberg and applied before and during WWII to manuf of gun barrels, artillery shells and various articles of ammunition. In 1942 the Neumeyer firm worked out the application of this process to manuf articles other than of ammunition (Refs 1 & 2)

In the cold extrusion process a piece of metal (such as steel) is forced to flow, at room temperature, under high, rapidly applied pressure, thru an orifice of the die. This produces a tubular object of the desired size

The steel used for this opn should be homogeneous and of low carbon & silicon contents

General descriptions of the cold working process are given in Refs 3, 4, 5, 6 & 7. Its modification known as *impact extrusion* has been used mostly for shaping nonferrous metals (Ref 11), but can be used also for iron or steel

Application of cold extrusion process to manuf of artillery shells is discussed in Refs 8, 9, 10, 12, 13 & 14 and its application to manuf of gun-barrels is briefly discussed as the *Auto-frettage* or *Self-hooping* in Vol 1, p A510-R of this Encycl

Refs: 1)Heinz Manufacturing Co, Philadelphia Penna, PB Reports **39371** (1946); **96704** (1948) & **96704s** (1948) (Description of Ger cold extrusion process) 2)Heinz Co Reports on cold

extrusion listed in the article of W.deVore (See Ref 14) 3)T.Lyman, Edit, "Metals Handbook," AmSocfor Metals, Cleveland, Ohio (1948), 4 & 353 4)A.H.Allen, Iron Age, **1953**, (August), 65-8 (Cold shaping of steel by compression reaches commercial application) 5)H.J.Pessl & H.H.Hautmann, Iron Age, **1953** (August), 69-73 (Factors influencing cold extrusion of steel) 6)T.Bishop, Iron Age **1953** (August), 74-7 (Tools lubricants and steels for cold extrusion) 7)J. G.Henderson & J.M.Bates, "Metallurgical Dictionary", Reinhold, NY (1953), 77-8 8)W.C. Tucker, Ordn **38**, 458 (1953) (Examples of ammunition articles manufd at present by the cold extrusion process) 9)Battelle Memorial Institute, "Shaping of Steel by Extrusion Method", Frankford Arsenal, Philadelphia, Pa, 31 July, 1954 10)G.E.Whitlock, Ordn **38**, 694-96 (1954) (Cold extrusion process by the method developed in 1947 by Whitlock at the Liberty Plant of the Mullins Manufacturing Corp, Salem, Ohio) 11)J.L.Everhart, Impact (Cold) Extruded Parts, Materials & Methods **1955** (August), 111-26 (Description of the process and discussion on sizes, shapes, limitations and applications of parts produced from Al & its alloys, Mg & its alloys, other nonferrous metals and steels) 11a)B.T.Fedoroff et al, PATR **2510** (1958), pGer 28 (Cold extrusion process) 12)Battelle Memorial Institute, "Methods of Manufacture Artillery Shells," Frankford Arsenal, Philadelphia, Pa, 10 May, 1959 13)S.Valencia, Ordn **44**, 806-07 (1960) (Cold forging known as "Swaging process") 14W.deVore, Ordn **48**, 316-24 (1963) (A German method of cold extrusion as improved and expanded by the Kelsey-Hayes Co, Romulus, Mich, so that artillery shells of large caliber can be mass produced quickly and economically)

Colinite Antigrisouteuse. An older Belg, coal mine permissible expl: NC 25, collod cotton 1, AN 20, TNT 12, K chlorate 6, cellulose & flour 29 & $MgSO_4.7H_2O$ 7%
Refs: 1)Marshall 1 (1917), 397 2)Marshall, Dict (1920), 23 3)Naoúm, NG (1928), 464

Collado (No initials given). An Italian ballistician of 16th century, who published in 1586 in Venice the book entitled "Manuel Pratique de l'Artillerie"
Ref: V.Montojo, MAF **13**, 765 & 790 (1934)

Collapsible Tube Method for Depositing Ignition and Explosive Charges in Electrical Blasting Caps. In this method the colloided soln (or suspension) of an expl (such as a mixt of Pb styphnate with NC lacquer), loaded in a collapsible tube (similar to those used for tooth paste), is squeezed thru a nozzle to be deposited in desired quantity around bridge wire of the igniter

It has been claimed that this method is an improvement over the previously employed methods, such as dipping of wire into a suspension of charge in NC lacquer
Ref: L.A.Burrows & C.A.Woodbury, USP 2205081 (1940) & CA **34**, 7112 (1940)

Colliard Boosters. Accdg to Hopper (Ref) a patent was granted to P.L.Colliard (not found in CA's 1907 to 1956) on the method for increasing the efficiency of expls by the use of special booster charges. These consisted of powdered Al or Mg mixed with one or more metallic oxides, chlorates or nitrates in such a proportion as to provide for complete combustion which would result in detonation, with the liberation of large amt of radiant energy. Colliard based his invention on the theory that detonation is propagated by the action of radiations emitted by the atoms and claimed that velocity of detonation of expls, and hence their brisance, is substantially increased when HE's are caused to explode by detonation of mixts emitting radiant energy

The following Colliard mixts were prepd and tested at PicArsn as boosters for HE's, such as TNT and Tetryl:
a)$Pb(NO_3)_2 + 4Al$ b)$3KClO_3 + 8Al$ c)$3BaO_2 + 4Al$ d)$3PbO_2 + 4Al$ and e)$KClO_3 + 3Mg$

Preliminary tesfs have shown that the brisance of TNT, initiated by primary mixts alone, was not increased when any of the above booster mixts was placed betw primer and TNT. When Tetryl was used in lieu of TNT, a slight increase in brisance was observed. This increase was, however, of little practical significance, since the above booster charges, being relatively insensitive to initiation, required abnormally large priming charges
Ref: J.D.Hopper, PATR **861** (1937)

Collieries, Blasting in. Under the title "Blast-

ing in Collieries," McAdam & Westwater (Ref) divide the work with expls in collieries (coal mines), into the following five main categories: a)Coal blasting b)Ripping blasting c)Drifting d)Shaft sinking and e)Waste blasting
Ref: R.McAdam & R.Westwater, "Mining Explosives," Oliver & Boyd, Edinburgh (1958) 95-135

Colliery Cheddite. See Vol 2 of Encyclopedia, pC158-L, item C, under CHEDDITES or STREETITES

Colliery Explosives. Explosives suitable for use in coal mines. See Coal Mine Explosives

Colliery Steelite. See under Colophony - Starch Nitrated Mixture

Collision Theory of Reaction. A theory to account for observed kinetics of reaction in terms of the molecular behavior of the reacting systems. For interaction, this theory requires that the molecules must collide and, in addition, have sufficient energy to be activated (See also Absolute Rate Theory in Vol 1 of Encyclopedia, pA4-R)
Refs: 1)C.Pruttons & S.Maron, "Fundamental Principals of Physical Chemistry", Macmillan, NY (1951), 637-42 2)A.A.Frost & R.G. Pearson, "Kinetics and Mechanism," Wiley, NY (1953), 57-72 and Second Edition 1961

Collodin. A smokeless proplnt suitable for small arms, patented in 1870 and 1871 by F. Volkmann of Austria. It was prepd by nitrating purified wood with concd nitric acid (in the manner similar to the method of Schultze), followed by treatment of the resulting "nitrolignin" with ether-alcohol. This gave partly a gelatinized material which could be shaped in any desirable form. Collodin was slower burning than Schulze's powder, because in the latter powder nitrated wood was not gelatinized but only impregnated with nitrates, such as K or Ba
(See also under Bulk and Condensed Powders In Vol 2)
Refs; 1)Marshall **1** (1917), 48 2)Davis (1943); 287 3)P.Tavernier, MP **32**, 242-43 (1950)

Collodion. A soln of collodion cotton (NC ca 11-12%N) in 2:1 ether-alc. A pale-yel syrupy liquid used for over 100 years for coating wounds; it is also used for making photographic films, varnishes, adhesives, etc (Ref 2). A.Nobel obtained (ca 1870) a gummy, elastic, highly expl mass by allowing a mixt of collodion and NG to dry. Later he prepd a similar expl, which is now known as *Blasting Gelatin* by warming (to 60-70°) a mixt of NG and collodion cotton alone (Ref 1)
Refs: 1)Naoúm, NG (1928), 9 2)CondChemDict (1961), 293

Collodion Cotton (abbr **CC**) or **Collodion Nitrocotton.** See Vol 2, ppC103 & C105, under CELLULOSE AND DERIVATIVES

Collodion Cotton – Nitroglycerin Gel Tests. The ability of a CC to bind NG into a homogeneous solid mass (like in Blasting Gelatin invented by A. Nobel in 1870) was detd in Germany before WWII either by the test of Escales (Ref 1, p294) or by the following test: Finely screened CC (2.5g) was well stirred (using a horn spatula) with 97.5g of cold NG, in a small procelain casserole having a handle. Then the casserole was heated on a water bath at 65° for 20 mins, while the mass was kneaded with the same spatula. If, after cooling, the mass separated readily from the porcelain as a coherent, cohesive gelatin, the CC was considered as suitable for manufg Blasting Gelatin. If, on the contrary, the mass was sticky or oily, it was considered unsuitable because of the possibility of exudation of NG from dynamite, especially on prolonged storage (Ref, p294)

It was reported that in some US dynamite factories, suitability of CC has been detd by actually making Blasting Gelatin on a small scale in a model mixer (Ref, p295, footnote)
Ref: Naoúm, NG (1928), 294 & 295

Collodion Smokeless Powder of Volkmann. See Collodin

Collodion Wool. One of the names for Collodion Cotton

Colloidal Dispersions or Solutions (Sols) and Colloids. Colloidal solutions (or rather "pseudo solutions"), also called *sols* (or in case of liquids *hydrosols)* are heterogeneous systems consisting of a "dispersion medium" (mostly a liquid) and a "dispersed" or "suspended medium" known as a "colloid". Colloidal particles are invisible under ordinary microscope but detectable by the ultramicroscope. Their size ranges from ca 1 x 10^{-7} to 1 x 5^{-5}mm. If the dispersion is a viscous, sticky, transparent liquid, it is what is generally known as a "colloidal solution". As examples of this may be cited a soln of gum-arabic in water and solns of NC in acetone, ethyl acetate or ether & alcohol. When "solns" are dialized, most of the colloidal particles do not pass thru the membrane. This is their principal distinction from "crystalloids", which are substances like Na chloride, etc. If part of the volatile liquid (dispersing medium), is evaporated the resulting tacky, jellylike substance is known as a *gel.* This jelly (or paste) may also be obtained on treating a colloid with a smaller amt of dispersing medium. Fruit jelly is a typical example of such gels. These gels are used in manufg expl & proplnt compns (See Colloidal Dynamites and Colloidal Propellants)

If the dispersed medium is a solid,immissible with dispersion liquid, it is called "suspensoid" and the combination a "suspension" (Eg: colliodal gold or ferric oxide in water), and if the dispersed medium is a liquid immissible with dispersion liquid, it is known as "emulsoid" and the combination as "emulsion" (Eg: milk, rubber latex, etc)

There are also colloidal "sols" in which dispersed and dispersion mediums are solids (Eg: alloys, plastics, glass, some minerals, etc)
Refs: 1)J.Alexander, "Colloid Chemistry, Theoretical and Applied", 6 volumes, Van Nostrand, NY (1926-1946) 2)W.K.Lewis, L.Squires &G.Broughton, "Industrial Chemistry of Colloidal and Amorphous Materials", Macmillan, NY (1943) 3)H.B.Weiser, "Colloid Chemistry", A Textbook, Wiley, NY (1949) 4)A.E.Alexander & P.Johnson, "Colloid Science", Oxford UnivPress, Vols 1 & 2 (1949) 5)Kirk & Othmer **4** (1949) 223-41 ("Colloids" by S.Glasstone) 6)B.Jirgensons & M.Straumanis, "Kurzes Lehrbuch der Kolloidchemie", Springer, Berlin (1949) 7)E.K.Fisher, "Colloidal Dispersions", Wiley, NY (1950) 8) J.W.McBain, "Colloid Science", Heath, Boston (1950) 9)H.R.Kruyt, "Colloid Science", Elsevier, NY, Vol 1 (1952) and Vol 2 (1949) 10) E.Hauser, "Colloidal Phenomena", MIT, Boston (1954) 11)Ullmann **10** (1958), 588-615 (Kolloide) 12)Encycl Britannica **6** (1963), 61-8

Colloidal Dynamites and Other Colloidal Explosives. In the true colloidal compns all ingredients are dispersed in such a manner that the mass is homogeneous and no individual particles are visible under ordinary microscope. Any colloidal compn contg as ingredients jellies, made by treating NC with expl liquid organic nitrates (such as NG, NGc, DEGDN etc), may be considered as a colloidal dynamite or explosive. But if substances non-dispersable in jellies (such as AN, KNO_3, sawdust, woodmeal, charcoal, etc) are incorporated in the jelly, the resulting mixts are not colloidal and may be defined as "gelatinous"

As an example of a purely colloidal dynamite may be cited "Blasting Gelatin", described in Vol 2, pB211-R of this Encyclopedia. Another example of "colloidal explosives" may cited Ballistite contg 60% NG & 40%NC. It was used by the Italians during WWII, under the name "Balistite al 60%", as a bursting chge for 37mm HE & HEAP shells (Vol 2, pB9-L, under BALLISTITE). This compn is too erosive (due to high NG content) to be used as a propellant

Following Refs are examples of expls listed in CA's as "colloidal", although some of them are not completely homogeneous compns:
Refs: 1)K.Bergl & J.Reitstötter, GerP 541468 (1926) & CA **26,** 2320 (1932) (Colloidal expl prepd by grinding its ingredients to colloidal fineness in a slowly rotating ball mill) 2)A. Hough, USP 18220290 (1931) & CA **25,** 5992 (1931) (Colloidal expls based on jellies obtd by colloiding vegetable parchment with NG) 2a)A.F.York, USP 1978070 (1934) & CA **29,** 346 (1935) (Highly nitrated NC, insol in eth-alc can be colloided by methanol at 0°) 3)R.W. Lawrence, USP 2595960 (1952) & CA **46,** 7332 (1952) [Highly efficient colloidal explosive shaped charges are prepd by pouring into a shaped container, first NG (precooled to 0-5°), and then dry, pulped NC and a stabilizer, using as little stirring as possible. Then the container is heated until the mass becomes homogeneous and complete colloiding of NC is

achieved. The mass solidifies on cooling] 4)K.Hino, JapP 2247 (1953) & CA **48**, 4840 (1954) (Colloidal dynamite contg NG 28.0, NC 1.2, AN 43.3, starch 1.0, urea 6.0, soft wax 0.5 & NaCl 20.0%) 5)K.Hino & J.Sato, JapP 6196 (1953) & CA **48**, 11790 (1954) (Colloidal dynamite contg NG 9.0, NC 1.0, AN 52.5, urea 8.0, urea nitrate 24.2 & guanidine nitrate 5.3%) 6)S.Mizuma & Y.Nemoto, JapP 3500 (1954) & CA **49**, 6610 (1955) (Colloidal dynamite: NG-NC gel 23.0, AN 64.85, paste of 60/40-polyvinyl acetate/ethanol 1.0, o-dibutyl ester of phthalic acid 0.25, woodmeal 1.0, K nitrate 5.0, starch 1.5, naphtalene 2.4 & water 1.0%) 7)Hispano Suiza SA, Tanger, DAS 1031700; Anmeldetag: 10 Februar 1956; Ausgabe der Auslegeschrift: 4 Juni 1958; listed in CA **54**, 16833-34 (1960) as GerP 1031700. This patent application deals with propellants (See Addnl Ref K under COLLOIDING AGENTS AND COLLOIDAL PROPELLANTS) and also with brisant explosives. The expls are obtained by kneading in the presence of water, a mixt of NC (not less than 25% in the final product on the dry basis), NG, crystalline HE (such as PETN, RDX or TNT) and a water-insol soap. The soap is prepd by the reaction of fatty acids with w-sol salts of Ba, Ca, Sr, Mg or Al or with compns contg nitrogen atoms capable of coupling, such as aniline, hydrazine, urea, etc. The resulting slurry is centrifuged to remove the bulk of water and then passed thru "endless screw press" (Schneckenpresse in Ger) to have the blend contg 8-10% of w. The next operation of rolling (passing the material betw two heated rolls) serves to dehydrate the material, to gelatinize NC and to form sheets. During this operation, and before complete removal of water, some powdered oxidizing agents, such as Amm or K perchlorate may be incorporated. The resulting sheets, similar to "solventless propellant", are either cut into grains of desired shapes or pressed hydraulically thru dies to form cords

Accdg to Dr. H.Freiwald of Saint-Louis Laboratory, Germany (Private communication 25 Sept 1962) the expls patented by Hispano-Suiza were developed by Dr. E. von Holt (who died in an automobile accident in summer 1962) and are now known under the name of **Holtex**

Following are expls listed in DAS issued in 1958. The same expls are listed in Patentschrift 1031700, issued in 1962

Example 1.

A)Mixture of NC 54, NG 16, TNT 18, PETN 8, H_2O-insol soap 2.8, and product of reaction betw oleic acid and aniline 1.2%. It is blended under water and the resulting slurry is centrifuged, passed thru endless screw press and then thru heated rolls. The resulting sheets are cut into strips, or passed thru press to form cords. The insol soap consists of Mg oleate 70, Ca oleate 20 & Ca stearate 10%

B)Mixt of NC 40, NG 36, PETN 17.5 & Ca oleate 3.5% is blended under w and then treated as above

C)Mixt of NC 45, NG (or DEGDN) 33, PETN 9, Amm or K perchlorate 10 & Ca oleate 3% is treated as above

Example 2:

Mixt of NC 46.5, NG (or DEGDN) 33.5, PETN (or RDX) 16, & w-insol soap 4% is blended under w and centrifuged to lower the w-content to ca 30%. The soap consists of Ca oleate 88, Mg stearate 10 & Al stearate 2%. The blend is then treated as in Example 1A, but not to complete removal of w. Then, in the middle of rolling operation, some powdered oxidizer (such as Amm or K perchlorate) is incorporated and the material is continued to be rolled to completion of dehydration and gelatinization. The amt of oxidizer is 5 to 45% of dried sheet

Example 3:

Mixt of NG 60 & NC 40% is blended under w and centrifuged to a w-content ca 20%. To the resulting slurry are added: w-insol soap 3-15, TNT 5-25 & mixt of Ba nitrate + Al powder 20-55% and the mass is blended, gelatinized and dehydrated by passing thru heated rolls etc] 8)Wasag-Chemie AG & H.Schlueter, DAS 1095722 (1960) & CA **57**, 10097 (1962) [Gelatinous expls prepd by mixing NGc, NC, MNT (or NG), AN (or Na nitrate), sawdust & > 5% Fe_2O_3. The last ingredient gives adhesiveness making these expls suitable for use in mud-capping] 9) Wasag-Chemie AG & E.von Holt DAS 1110562 (1961) & CA **57**, 10098 (1962) [Expls of high power & brisance can be obtd by gelatinizing NC with TeNMe either alone or in the presence of NG (or DEGDN) and/or other nitrocompds; other expls without gelatinizing props, such as RDX, may be added; also may be incorporated metal dusts, stabilizers & gelatinizers. Eg: mixt of 100 parts of wet NC

(12.2%), 40ps TeNMe, 20ps DEGDN & 0.5p of stabilizer possess, after being processed in the manner used for manuf of "solventless" proplnts, the following props: heat of expln 827cal/g, gas vol evolved on expln 903cc/g, Trauzl test value 320cc (TNT ca 300) & impact sensitivity 25cm with 2kg wt) 10)Hispano-Suiza SA, Tanger, DBP Patententschrift 1031700, Ausgabe der Patentschrift 30 August 1962 (Herstellung von Brisantzsprengstoffen). These expls, known as **Holtex**, are described under Ref 7 11)K.Böhlen, "Holtex-ein neuer Sprengstoff?" in Explosivst **1962**, 156-58 [Props of an expl prepd by Dr. E. von Holt and known as *Holtex* are given w/o revealing its exact compn. It is only stated that Holtex is a gelatinized mixt of NG, NC, PETN & water -insol soap prepd in the manner of solventless proplnt (POL-Pulver ohne Lösung in Ger). Its props are similar in some respects to those of **Nipolit** which was also developed by E. von Holt. Compns of two Nipolits manufd during WWII in Germany are given in PATR **2510** (1958), pGer 117

Following are props of Holtex: Gray-black rubbery substance; d 1.63; brisance by Cu compression test 4.9mm; chemical stability - satisfactory; detonation rate 7800 - 8000m/sec; energy (f) 11900 $l \times p_o/kg$; explosion temp 180°; gas volume at NTP 830 l/kg; heat of expln 1230cal/g; heat of formation 480cal/g; impact sensitivity with 2kg wt 120cm -no expln; power by Trauzl test 380-450cc; rifle bullet test - no expln when hit with a bullet from Swiss carbine cal 7.5mm at a dist of 15m at muzzle velocity 780m/sec; temp of expln 3650°K

Holtex is nonhygroscopic and water-insol. It can be shaped not only by the process of hot rolling used in manuf of solventless proplnts but also by cutting, boring, sawing, milling, lathe-turning, etc]

Colloidal Fuel. It is a colloidal suspension of finely pulverized coal in a fuel oil. Such a mixt, although more expensive than coal, possesses the advantage (particularly useful for marine purposes) that it is more readily handled and permits more economical utilization of bunker space. The density of such a suspension is higher than that of fuel oil and it produces more BTU per cu ft than either coal or oil. For example, the mixt of coal (d 1.35 & BTU 145000 per lb) 40 and fuel oil (d 0.90 & BTU 19000 per lb) 60% has a potential heat content 11.2 thermes per cu ft, as compared with 10.7 for oil and 7.2 for coal alone.

Refs: 1)Thorpe **5** (1941), 364 2)D.Brownlie, SteamEngr **13**, 3-5 (1943) & CA **38**, 1089 (1944) (Colloidal fuels in US - reasons for renewed interest)

Addnl Refs: a)Annon, IronAge **103**, 824 (1919) & CA **13**, 1253 (1919 [Atomizable fuel, called "Fliesskohle" in Ger, can be prepd by suspending coal dust in mineral oil, using as suspending agent ca 1% of substance resembling an axle grease in appearance and consistency) b)L.W.Bates, BritP 149306 (1919) & CA **15**, 431 (1921); CanChemJ **4**, 40-7 (1920) & CA **14**, 1027 (1920); CanP's 204259 (1920); 204260 (1920; 204266 (1920); 204267 (1920); CA **14**, 2313 (1920) (Various colloidal fuels consisting of coal dust & oil, suitable for marine use) c)F.Gramme, Challeur et Industrie **Special No**, 202-12 (1924) & CA **19**, 1188 (1925) (Review of prepn of colloidal fuels) d)H. Plauson & P.Schröde, USP 1647471 (1927) & CA **22**, 313 (1928) (Stable liq fuels consisting of a suspension of finely divided coal, coke or peat in mineral oil) e)T.Itakura, JSocChem-Ind, Japan **40**, Suppl binding, pp280-81 (1937) & CA **31**, 8880 (1937) (Suspensions of coal in oil which can be stored w/o sepn for more than 450 days) f)V.F.Kustov et al, KhimReferatZhur **1940**, No10-11, 95 & CA **37**, 1240-41 (1943) (Colloidal fuels consisting of coal dust suspensions in "mazut", which is the residue remaining after the distn of gasoline and kerosene from Rus petroleum. These fuels have been used in industrial furnaces) g)E.Boye, Chem-Ztg **68**, 187-8 (1944) & CA **40**, 3865 (1946) [Review with 34 refs of colloidal fuels known in Germany as Fliesskohle (fluid coals)] h) R.M.Bridgewater, Petroleum (London), **8**, 2 & 4 (1945) & CA **40**, 4870 (1946) (Brit research on petroleum substitutes: colloidal fuels) i)R. S.Voose, USP 2423913 (1947) & CA **41**, 6387 (1947) (Homogeneous liq prepd by heating fuel oil with finely divided coal at temp above 66°, but below cracking temp of oil) j)K.A.Spencer & D.W.Machin, USP 2430085 (1947) & CA **42**, 1042 (1948) (Colloidal coal fuel) k)C.S.Reed, USP 2453641 (1948) & CA **44**, 2734 (1950) (Col-

loidal coal fuel)

Colloidal Propellants. See next item.

Colloiding Agents and Colloidal Propellants. *Colloiding (or gelatinizing) agents* are substances, which upon mixing (either alone or dissolved in a volatile solvent) with organic compns, such as NC, cellulose acetate, etc, produce homogeneous transparent (or semitransparent) gels which usually harden either in storage or after evaporation of the volatile solvent. The most important gels used in the explosives industry are those based on NC *Colloiding Agents for NC's.* As mentioned above, NC's can be colloided by means of some volatile solvents, and this was discussed more fully (giving numerous refs) in Vol 2, p C115-R of this Encyclopedia. Since nearly all of the volatile solvent is removed on drying, there is no change in the properties on NC, except a decrease in its rate of burning. If a colloiding agent is non-volatile (or only slightly volatile), liquid or solid explosive (such as NG, EGDN, DEGDN, TEGDN, NGu, DNT, DNEtBz, DNX oil, Nitrotoluidine, TNT, etc), its use for the gelatinization of NC not only decreases the rate of burning, but also increases its explosive characteristics. See under CANNON PROPELLANT in Vol 2, pp C33-R & C36-L

If a nonexplosive, nonvolatile (or slightly volatile), colloiding agent is used (such as DBuPh, DEtPh, Substituted Ureas, Urethans, Ethyl Sebacate, Rosin, Camphor, Triacetin, etc) alone not only the rate of burning of NC is decreased but also its expl characteristics. Davis (Refs 2 & 12) gives tables listing various colloiding agents and parts by wt necessary for the complete gelatinization of 100ps of Pyrocellulose

Colloidal Propellants. If a non-colloided NC is used as a proplnt, it burns rapidly developing extremely high pressure in a short interval of time. Such material can be used as a proplnt only in smooth-bore arms (such as shotguns) without danger of damaging their barrels, but cannot be used (even in highly compressed form) in rifled weapons becuase of the danger of damaging (or even bursting) their barrels. Although the fact that NC can be colloided with ether-alcohol mixt (but not with either of these solvents alone) was known since 1847 (discovery of Maynard), no practical application of this property was made until 1870-71, when Volkmann of Austria patented his partly gelatinized NC proplnt called "Collodin" (qv). Although this substance was considerably slower burning than any proplnt previously made, it was not suitable for cannons but only for small arms. The first successful colloidal smokeless proplnts suitable for both small arms and cannons were prepd independently and nearly at the same time at Rottweil, in Germany by M. von Duttenhofer (1883-84) (Rottweiler Cellulose-Pulver, abbr *RCP*), and in France by P.Vieille (1884) (*Poudre V*, later named *Poudre B*) (See Vol 2, pB1-L). These proplnts were prepd by treating NC with a volatile solvent and consisted, after drying, of hard, colloided NC with only traces of solvent. As they are based on only one explosive ingredient (NC), the term "single-base propellant" is applied to them now. In order to render them stable in storage, a small amt of a stabilizer was incorporated

Slightly later (1891) and independently from Fr & Ger inventions (which were kept secret from the rest of the world), D.I.Mendeleyev of Russia developed a single-base propeln named *Pirokollodion.* It contained NC of ca 12.44% N colloided with eth-alc. This proplnt served as a prototype for *Pyrocellulose,* abbr as *Pyro,* introduced by Lt. J. B. Bernadou (See Vol 2, pB104-L) to US Navy and then later to US Army. The NC used in Bernadou's proplnt contains ca 12.6% Its prepn and props are given in Vol 2, pC105-L)

At present there are in US, NC's with lower and higher than 12.6%N content, such as: *Collodion Cotton* (also called Pyroxylin) with 11-12% N (See Vol 2, pC103-R), *Guncotton* with ca 13.4%N (See Vol 2,pC106-R), and *Blend* with N ca 13.15% (prepd by blending Pyrocellulose with Guncotton) (See Vol 2, pC106-L)

Compositions and characteristics of current US single-base proplnts for small arms, recoilless rifles and some cannons are given in Vol 2, ppC34 & C35. Compositions of Ger single-base proplnts used during WWII are given in Ref 23, pGer 141. Some of these proplnts contained large amts of PETN and may be considered as "double-base". Some

Rus single-base proplnts are listed in Ref 19, pp Rus 17-18

Single-Base Propellants, Manufacture of. In the prepn of these proplnts it is essential that NC is completely (or nearly so) colloided by 2:1 eth-alc mixt in order to obtain a homogeneous mass. This can be achieved by the following operations, which have been practiced in US(Ref 10, pp21-27, Ref 11, pp 20-33; Ref 12, pp299-306 & Ref 20, pp252-53) a)The pulped NC received from *wringers* (See Vol 2, pC108-L, item VII, Wringing) contains 25 to 31% H_2O. As wet NC cannot be successfully colloided with eth-alc mixt, it is necessary to remove the bulk of water. This is achieved by compressing the wringed material for a few mins in a hydraulic press at ca 250psi b)The pressure is released and a predetermined amt of 95% denatured alcohol is pumped thru the NC block in order to remove the remaining water as completely as possible and to replace it with alc. During this opn the pressure is gradually increased to 3500psi. This opn, called *dehydration,* results in the formation of a block consisting of 25lbs of dry NC and about 9 lbs of 90% alc d)The block is removed from the press, placed in a covered container and transferred to another bldg where the *Breaking operation* is conducted. This is usually done in a block breaker which consists of a rotating drum equipped on the inside with a wire screen and iron prongs. The drum upon revolving throws the block against the prongs thus breaking it into small lumps e)The lumps are placed in closed containers and transferred into the *mixer,* which consists of a covered water-cooled tank in which two shafts carrying curved blades rotate in opposite directions, giving a kneading motion to the material in the mixer. During this agitation, the calcd amt of ether (ca 70ps per 100ps of NC), contg a stabilizer (1p of DPhA per 100ps of finished product) is poured in rapidly in order to minimize loss of solvent by evapn. The other substances (such as plasticizers, DBuPh, Triacetin, DNT, etc) may also be dissolved in ether previous to their addn to NC. If K sulfate is required to be incorporated, it is added separately following the ether f)The cover is closed and the blades are rotated until the mass becomes fairly homogeneous. This opn, which requires about 1hr, is known as *mixing* or *incorporating.* g)The resulting partially colloided NC, (brownish in color and soft enough to be deformed on pressing betw the fingers), is transferred to another hydraulic press, called *preliminary blocking press,* where it is compressed at 3000-3500psi h)The *preliminary block* is transferred to the *macaroni press,* where it is squeezed, at 3000-3500psi, first thru a series of steel screens (to remove any uncolloided NC and most of mechanical impurities) and then thru plates with round perforations i)The material emerging from the press in a form resembling macaroni and contg nearly completely colloided NC, drops directly into the cylinder of the *final blocking press.* Here macaroni is squeezed, during 1 or 2 mins at 3500psi, into a compact, transluscent, amber colored, cylindrical block of the size to fit the *graining press* j)Here the block is squeezed by hydraulic pressure thru dies by which it is formed into single-perforated or multiperforated cords. k)By means of a *cutting machine,* the cord is cut into pieces of desired size. This gives *green powder,* which contains completely colloided NC with a fairly large amt of volatile solvent (ether-alcohol) l)The finished product, ready for loading in ammo, is obtained by removal of volatile solvent from the green powder. This is required not only because the finished product should not give off vol solvent under changing atmospheric conditions, but also because it is economical to recover the bulk of the solvent for future use m)Removal of volatile solvent, with consequent shrinkage of the grains to their final dimensions, is accomplished by the following three operations: 1)*Solvent recovery.* The green pdr is placed in a large tank and warm air is passed thru the material to pick up the solvent vapors; and this vapor-laden air is then forced over cold coils to condense the vapors. This is continued until the volatile content is reduced to ca 6%. This takes 6 to 14 days 2)*Water-drying.* The partially dried proplnt is placed in w at 25° and, while constantly stirring, the temp is gradually raised to 55° and maintained so for several days. This reduces residual solvent to 0.3-5%, depending upon the grain size

3)*Air-drying*. The wet proplnt is transferred to the continuous air dryer where w is removed at 55-65° n)The final opn before packing is to blend the proplnt in a lot which may vary betw 50 and 500 thousand pounds. This gives a uniform product

Double-Base Propellants for Cannons. The discovery of colloidal proplnts based on two expl ingredients (NC & NG), dates from 1887-88 when A. Nobel of Sweden developed *Ballistite* (See Vol 2, pB8-L). This proplnt, as well as *Cordite* (See this Vol), developed slightly later (1889) in England, is now known as *double-base propellant*

There are two methods used for their prepn: the "solvent" and "solventless"

In the *solvent* method, such as used in some early Ballistites (Ref 1, p301), NC of ca 12%N was treated in cold with NG to which a volatile solvent, such as benzene, was added. The presence of vol solvent was necessary because without it and in cold, NG does not gelatinize but only "swells" NC's. A similar method was used for some *Cordites*. Here NC of high N content (Guncotton) was treated with NG and acetone with an added small amt of mineral jelly. Both acet & jelly were used to facilitate the gelatinization of NC with NG. The resulting paste was extruded in the form of cords and dried to remove the acetone (Ref 1, pp304-08 & Ref 12, p295)

The manuf of US *solvent double-base propellants* is similar to that described for US single-base proplnts, except that the mixt of alc & acet is used as the volatile solvent, and the solvent recovery procedure is omitted because of the hazard involved in handling the vapors contg NG (Ref 20, p255)

In the original *solventless* (or *nonsolvent*) method, patented in 1889 by Lundholm & Sayers, the soluble NC & NG were stirred in hot water by means of compressed air. The dough thus produced was passed betw two rolls heated to 50-60°, whereby the bulk of water was pressed out and the NC-NG gel made into a sheet. This was folded over and passed thru the hot rolls again, and the opn was repeated until the material was converted into a uniform colloid, and all the water removed by evaporation. The sheet was then cut into square flakes, generally coated with graphite, and these were blended with flakes obtained from other sheets (Ref 1, p302)

In the US, the *solventless process* is used when NG, plus any other colloiding agents for NC, constitutes ca 40% of the composition. In this process the wet NC (usually of 13.25% N) is removed from the wringer (See Vol 2, p C108-L) and is mixed first with NG and then with EtCentr. The slurry thus obtained is centrifuged to remove the bulk of the excess water and the resulting paste is put into cotton bags, which are subjected to heated air currents to reduce the moisture content of the paste. If a proplnt is intended for use in cannons, such as compns M2 & M5, listed in Vol 2, pC34, Table V, the two other ingrediens :Ba nitrate & K nitrate are then blended with the partly dried paste. If a proplnt is intended for use in rockets such as compn T8 listed in Ref 20, p259, then DNT, Pb stearate and Triacetin are incorporated at this time. These operations are followed by repeated passing of paste between two heated steel rolls in order to remove the rest of the water and to complete the colloiding of NC. The thickness of the resulting sheet is controlled carefully and varies with its use (Ref , pp255-57)

Compositions of some current US double-base proplnts, both "solvent" and "solventless" types, are given in Vol 2, ppC34 & C35 and compns of German double-base NC-NG proplnts used during WWII are listed in Ref 23, pGer142. Some Rus proplnts used during WWII are listed in Ref 19, ppRus 17 & 18

Germans also used during WWII a "cool" double-base proplnt, known as "G" Pulver, which was obtained by substituting DEGDN or TEGDN for NG. Its prepn is described in Ref 23, p Ger70 and some typical compns are given on pp Ger 143-46. This proplnt was developed under direction of Gen U.Gallwitz in 1934-35

Some US proplnts listed as "single-base" (such as M1, MIAI & M6 may also be considered as "double-base" because they are based on two expl ingredients: NC & DNT. The same reasoning applies to some Ger proplnts which contd NC & PETN (Ref 23, pGer 141)

Double-Base Propellants for Rockets. Compns of some US rocket proplnts, such as M7, M13, T2, T5 & T8 are given in Ref 20, p259 and of proplnt JPN in Ref 24, p9. The manuf of

"solvent-type" (extruded) and of "solventless" (in sheets or extruded) is similar to those processes used for cannon proplnts. For a more detailed description of the manuf of solid rocket proplnts, see Ref 24, pp43-58

There are also *cast rocket* proplnts, such as **OV** described in Ref 24, p9. The process consists essentially of filling a plastic mold (or beaker) with granular proplnt material [such as in case of OV proplnt, the mixt of NC (12.6% N) 89.0, DNT 10.0 & EtCentr 1.0%, with added carbon black 0.2%], adding the "casting solvent" (in case of OV proplnt, the mixt of NG 70.9, dimethylphthalate 28.1 & EtCentr 1.0%), and curing at elevated temp to obtain a homogeneous solid grain. This method permits the obtaining of proplnt grains of considerably larger size than is possible by extrusion. More detailed description of method of prepn of cast rocket proplnts is given in Ref 24, pp58-60

Ball Powder. This unique type of "solvent" proplnt was developed by F.Olsen of Western Cartridge Co (now called Olin-Mathieson Chem Corp). For its prepn, props and uses, see Vol 2, ppB11 to B16 and Ref 24, pp9 & 48-57

Triple-Base Propellants. These proplnts, based on three expl ingredients, one of them NGu, are even "cooler" then double-base proplnts based on NC & DEGDN. A composition based on NC, DEGDN & NGu, known as "Gudolpulver", was developed ca 1937 in Germany by the Dynamit AG. It is described in Ref 23, p Ger 81; and some compns used during WWII are listed in Ref 23, pp Ger 143, 145 & 148-49. The triple-base cannon proplnts based on NC, NG &NGu are manufd now in US (See proplnts MI5, MI7, T20, T29, M30 & M31 in Vol 2, pC34 of Encyclopedia). Some Rus proplnts examined during WWII at PicArsn contained NC, NG & DNT (or TNT) and may be considered as *triple-base proplnts* (Ref 19, pRus 18)

In the manuf of Ger or Amer triple base proplnts, powdered NGu is gradually added to the blend of NC-DEGDN (or NC-NG), contained in a mixing machine, and kneaded until the mass becomes fairly homogeneous. Then the resulting dough is rolled betw two heated rollers in the same manner as described under "double-base" proplnts (Ref 23, pGer 81-R)

Composite Propellants. These are non-homogeneous mixts of a finely ground oxidizer (such as Amm perchlorate or nitrate) in a matrix of plastic, resinous or elastometric material (Ref 24, pp10-11 & 34-40). As these proplnts are not based on colloided NC, they are not considered as "Colloidal Propellants", but are described separately as "Composite Explosives" (See also Coefficient de gélatinisation and Coefficient de gonflement)

Refs: 1)Marshall **1** (1917), 289-321 (Slow-burning smokeless powders); 322-35 (Fast-burning smokeless powders) 2)T.L.Davis, IEC **14** 1140-41 (1920) (Colloiding agents for NC) 2a)F.L.Nathan et al, USP1339142 (1920) & CA **14**, 1898 (1920) (A gelatinous proplnt is formed of NC 50, NG 40, p-nitromethylacetanilide 7.5 & β-naphthylether 2.5%) 3)H.Brunswig, "Das Rauchlose Pulver", W. de Gruyter, Berlin (1926) 4)M.Guia, "Le Polvere Senza Fumo", Subalpina, Torino (1930) 5)R.Molina, "Esplodenti", Hoepli, Milano (1930), 206-44 (Fulmicotone); 380-430 (Polvere senza fumo) 6)Marshall **3** (1932), 85-96 (Smokeless powders) 7)Vennin, Burlot & Lécorché (1932), 575-638 (Poudres sans fumée) 8)Stettbacher (1933), 186-205 (Die rauchschwachen Pulver) 9)Pepin Lehalleur (1935), 289-323 (Les poudres colloïdales) 10)Hayes (1938), 21-27 (Manuf of smokeless powder) 11)Anon, "Military Explosives", WarDept TM **9-2900** (1940), 20-33 (Manuf of smokeless powder) 12)Davis (1943), 287-330 (Smokeless powders); 320-22 (Gelatinizing agents for NC) 13)Pérez Ara (1945), 401-56 (Pólvoras sin humo) 14) Mangini, Esplosivi (1947), 233-35 (Polvere senza fumo) 15)Vivas, Feigenspan & Ladreda **3** (1948), 105-204 (Pólvoras sin humo de nitrocellulosa) 16)Stettbacher (1948), 39-49 (Die rauchlosen Pulver) 17)Stettbacher (1952), 49-64 (Pólvoras sin humo) 18)Belgrano (1952), 108-14 (Polveri colloidali o Polveri senza fumo) 19)B.T.Fedoroff et al, PATR **2145** (1955), ppRus 17 & 18 (Russian proplnts of WWII) 20)Anon, "Military Explosives", TM **9-1910** (1955), 218-66 (Propellants) 21)C. Bignotti "Nitrocellulose", Cya, Firenze (1956) 22)Gorst (1957), 131-48 (Rus colloidal powders) 23)B.T.Fedoroff et al, PATR **2510** (1958), pp Ger 70-1, 81 & 140-51 (Ger proplnts of WWII)

24)Warren (1958), 6-68 (Rocket proplnts) 25) Guia, Trattato **6** (1959), 346-68 (Polveri senza fumo) 26)Berthmann (1960), 62-3 (Rauchschwache Pulver) 27)A.M.Ball, "Solid Propellants", Ordnance Corps Pamphlet ORDP **20-175**, Part 1 (1961)

Addnl Refs: A)H.J.Tattersall, USP 2188322 (1940) & CA **34**, 3918 (1940) (Pb salt of dimethylphosphoric acid is incorporated in colloidal proplnts to reduce their flammability) B)B. Troxler, Brit P 568721 (1945) & CA **41**, 4312 (1947) (Flashless colloidal double-base proplnt) C)T.Thomson, BritP 578372 (1945) & CA **41**, 1843-44 (1947) (Flashless, colloidal double-base proplnt) D)D.Fenson & S.Fordham, TransFaradSoc **43**, 538-42 (1947) & CA **42**, 2771 (1948) (Colloiding of NC's with NG was followed microscopically using crystal violet base to make the process visible by ordinary light. With NC's lower than 11.4%N and higher than 12.6%N, only limited swelling occurred; but with NC's betw 11.4 & 12.6%N gels were formed which could be seen to diffuse at a speed dependent on the vicosity) E)W.R.Moore, TransFaradSoc **43**, 543-52 (1947) & CA **42**, 3174 (1948) (Swelling of NC's in binary mixtures) F)I.Jullander et al, JPolymerSci **3**, 804-11 (1948) & CA **43**, 8673-74 (1949) (Gelling of solns of NC's can be brought about by addn of small amts of certain substances, which form cross links betw unesterified OH groups of adjacent NC chains. The properties of such gels were studied by means of the ultracentrifuge and a consistometer) G)W.R.Moore, JTextileInst **40**, T731-33 (1949) & CA **44**, 836 (1950) (Microscopic examination of the swelling and soln of fibrous NC of high N content) H)H.Maisner, USP 2712989 (1955) & CA **49**, 14325-26 (1955) (Rocket proplnts obtd by colloiding NC with nitroparaffins, such as $MeNO_2$. They can also be used as expls. Ignition of these compns can be made easier by incorporating up to 3% chromium acetylacetonate) (See Vol 1, pA53-R and Vol 3, under Chromium) I)W.O.Baker, USP 2780997 (1957) & CA **51**, 7014 (1957) (Manuf of colloidal proplnts by treating NC with acetone, followed by incorporating NG, nitrate salts, etc. The resulting gelatinous mass is extruded at high sheer from a die in the form of a sheet, ribbon, filament, etc and then the material is dried while being maintained under tension along the line of extrusion) J)A.Kraus, Farbe u Lack **64**, 538-39 (1958) & CA **53**, 3687 (1959) (Cooling of solns of colloidal cotton to temps as low as -70° induces the formation of gels) K)Hispano Suiza SA, Tanger, DAS 1031700; Anmeldetag : 10 Februar 1956; Ausgabe der Auslegeschrift: 4 Juni 1958; listed in CA **54**, 16833-34 (1960) as GerP 1031700. This DAS deals with prepn of a smokeless propellants and brisant explosives, now known as **Holtex** (See Ref 7 under Colloidal Explosives). All of these compositions were invented by Dr. E. von Holt (Dr. H. Freiwald, private communication, 25 Sept 1962)

Prepns of proplnts are similar to the methods used in the manuf of ordinary solvent and solventless proplnts, except that some water-insol metallic soap is incorporated to facilitate the gelatinization of NC and to serve as a stabilizer

Following are examples of proplnts developed by Dr. Holt

Example 1:

A)Single-base proplnt is prepd by gelatinizing 90 parts of NC with a small amt of 2:1-ether-alcohol, in presence of 10 parts of w-insol metallic soap (which consists of Mg oleate 65, Al stearate 20 & Ca stearate 15%). The resulting gel is extruded by hydraulic pressure into cords and these are cut to desired lengths and dried B)Single-base proplnt is prepd by treating 92 parts of NC with 2:1-ether-alc in presence of 4 parts of w-insol coap (Mg oleate 60, Ca stearate 20 & Ba palmitate 20%) and 4 parts of product of reaction betw oleic acid, urea & DPhA. Further operations are the same as in A)

Example 2:

Double base, solvent-type proplnt is prepd by blending,with cooling, in presence of acetone, a mixt of NC 60 & NG 40% (contg ca 8% H_2O) to which are added: 5% of w-insol soap (Ca oleate 85 & Mg stearate 15%) and 2% of product of reaction betw stearic acid, diethanolamine & aniline. Further treatment is the same as in A)

Example 3:

A)Double-base, solventless proplnt is prepd by blending, under water a mixt of NC 65, DEGDN 28, w-insol soap (Ca oleate 70, Mg oleate 18 & Ca stearate 12%) 5 and a product obtd by treating oleic (or stearic) acid with alpha-naphthylamine & urea 2%. The resulting slurry is centrifuged (to remove all but 30% of w)

and then passed several times thru heated iron rolls. The resulting rubber-like material is either cut into grains of desired sizes and shapes or pressed hydraulically thru dies to form cords B)Double-base solventless proplnt is obtd by treating the mixt of NC 57, NG 39 & w-insol soap 4% in the same manner as above. The soap consists of Ca oleate 80, Ca palmitate 10 & Al stearate 10%
Note: Dr. E. von Holt was also the inventor of solventless colloidal proplnt known as **Nipolit.** Two types of this proplnt were manufd during WWII at the Kaiburg plant of the Deutsche Sprengchemie GmbH: a)Nipolit tubes - NC (12.6 - 12.7%N) 34.1, DEGDN 30.0, PETN (unwaxed) 35.0, stabilizer 0.75, MgO 0.05 & graphite 0.1% and b)Nipolit sticks - NC (12.6 - 12.7%) 21.9, DEGDN 20.0, PETN (unwaxed) 50.0, stabilizer 0.75, MgO 0.05 & graphite 0.1%. Their prepn and some props are described in PATR **2510** (1958), p Ger 117, which includes 4 refs. L)Wasag-Chemie AG, DAS 1030748 (1958) & CA **54**, 16833 (1960) (A base chge of colloidal smokeless proplnt is incorporated with cryst expls, such as PETN, or NGu) M)Wasag-Chemie AG & G. Knöffler, DAS 1056989 (1959) & CA **55**, 6868-69 (1961) [Colloidal proplnts with low heat of expln may be obtd by gelatinizing NC with nitrated phenylethyl alcohols. Example: NC 56.75, 2-(dinitrophenyl)ethyl nitrate 41.00, K sulfate 2.00, Mg oxide 0.10 & graphite 0.15%]
Note: Many Refs on colloiding of NC are given in Vol 2, ppC116-C118, under Cellulose Nitrate, Action of Solvents

Colloxyline or Kolloksilin. Rus designation for NC with N content 11.5 to 12.%. It is sol in eth-alc mixts; has been used in prepn of dynamites, some smokeless proplnts, celluloid, artificial leathers and in lacquer. NC's with higher N content are known in Russia as *Piroksiliny*
Ref: Gorst (1957), 134

Cologne Powder or Colonia Powder. An older expl mixt consisting of black powder 65-70 & NG 35-30%. It was manufd by Wasserfuhr Bros at Köln (Cologne), Germany
Refs: 1)Daniel (1902), 150 2)CondChemDict (1942), 288 (Not found in later editions)

Cologne Rottweiler Safety Powder (Köln-Rottweiler-Sicherheits-Sprengpulver). An expl compn safe for use in gaseous or dusty coal mines, manufd since 1897 at Cologne by the Vereinigte Köln-Rottweiler-Pulver-fabriken. Its compn was: AN 93.0, Ba nitrate 0.9, vegetable oil 4.9 & sulfur 1.2%
Refs: 1)Daniel (1902), 150 & 395 2)Cond-ChemDict (1942), 288 (Not found in newer editions)

Colombia (or Columbia) Powder. An expl mixt, patented in 1891, prepd by intimately mixing 82.6ps of powdered K chlorate with 8.7ps sulfur and coating the grains with 8.7ps of paraffin
Refs: 1)Daniel (1902), 151 2)Giua, Trattato **6** (1959), 395

Colophony or Rosin (Colophane or Résine in Fr; Kolophonium in Ger; Colofonia in Ital & Span and Kanifol' in Rus). A translucent, pale-yel or amber to dk red, brittle & friable resin, d ca 1.08 & mp 100-150°; gives off flammable vapors at higher temps. Its chief constituents are resin acids of the abietic and pimaric types of general formula $C_{19}H_{29}COOH$, and having a phenanthrene nucleus. It is insol in w; but sol in alcohols, ether, benz, AcOH & dil solns of K & Na hydroxides. Can be obtd as a residue from the distillation of turpentine oil, oleoresin, or by other methods. It has been used in the manuf of varnishes & lacquers, linoleum, adhesives, rosin soaps, binding materials, in soldering and for rosining bows of string instroments (Refs 1, 4, 5, 6 & 7)

Rosin has also been used in some industrial expls either as a fuel or as a binder (when dissolved in alcohol or other solvent). Examples of using rosin in expls are given by Davis (Ref 3, pp333, 334, 336, 350 & 360). They include: *Dynamite with Active Base of Nobel* (1869) - Ba nitrate 70, NG 20 & rosin 10%; *Dynamite with Active Base of Nobel* (1873) - same as above but paraffin, wax or ozokerite is incorporated to render the mixt nonhygroscopic (Ref 3, p333); *Pyrodialythe* - K chlorate 85 & rosin 15% mixed with 2 parts of alcohol, followed by drying; *Silesia No 4* - K chlorate 80 & rosin 20%; *Silesia IV 22* - K chlorate 70, NaCl 22 & rosin 8% (Ref 3, p360). In Ref 2 is listed *PSE No 15 Powder* which consisted

of Amm perchlorate & rosin
Refs: 1)Thorpe **3** (1939), 294-95 (Colophony) 2)CondChemDict (1942), 290 (Not found in later editions) 3)Davis (1943), 333, 334, 336, 350 & 360 4)Kirk & Othmer **11** (1953), 779-810 (Rosin and Derivatives) 5)Ullmann **8** (1957), 400-17 (Kolophonium under Harze, naturliche) 6)CondChemDict (1961), 988 7)Merriam-Webster's (1961), 1975 (Rosin)

Colophony, Nitrated (Nitrorésine in Fr). Accdg to Daniel (Ref 1, p708), Schultze patented in 1886 expls and proplnts contg nitrated rosin, (or nitrated tar). For nitration of rosin it was pulverized and heated on a water bath, while stirring, with 15 parts of nitric acid d 1.420 to 1.460. Eg: *Mining Explosive* - NC 10, K nitrate 75, sulfur 10 & nitrorosin 15%; *Sporting Powder* - NC 60-80, Ba nitrate 60-80, K nitrate 8-10 & nitrorosin 12 parts (Ref 1, p708)

In Ref 2 is listed *Silesia A* as contg K chlorate 75 & nitrated resin 25%
Refs: 1)Daniel (1902), 568 (Nitrorésine); 708 (Schultze) 2)CondChemDict (1942), 291 (Not found in later editions)

Colophony–Starch Nitrated Mixture (called by Davis **Oxidized Rosin** and by its inventor E. Steele **Résidée**). It is a solid prepd by treating an intimate mixture of finely pulverized colophony 90 & starch 10% with cold 42 Bé nitric acid. A homogeneous solid, formed after several mins, was used as an ingredient in the prepn of potassium chlorate expls, known as *Steelites*. For their prepn, finely pulverized and dried K chlorate & résidée, in desired propns, were slightly moistened with methyl alcohol and intimately mixed in a wooden vat using a wooden paddle. During this operation, alc evaporated by itself. The following compns were prepd by this method: *Steelite No 3* - K chlorate 75 & résidée 25%; *Steelite No 7* - K chlorate 87.5 & résidée 12.5% and *Colliery Steelite* - K chlorate 72.5-75.5, résidée 23.5 - 26.5 & castor oil 0.5-1.0%. The last expl was, at one time, on the Brit "permitted list" (Refs 1, 2 & 3)

The *Steelite No 5* - K chlorate 83.33, résidée 16.67 & Al powder 5.0% was rejected by CSE as being too dangerous due to the presence of Al (Ref 1)
Refs: 1)CSE (Commission des Substances Explosives), MP **15**, 182-85 (1909-1910) 2) Marshall **1** (1917), 388 3)Barnett (1919), 111 4)Davis (1943), 360

Color. Many US Military Specifications for expls and materials used in their manuf have color requirements. For example: AN, Amm Chlorate, Amm Perchlorate, EDD, EDNA, HMTeA, LA, PE, PETN & RDX should be white; DNB & DNT pale yellow; DNPh, HNDPhA, MNN, PA, TNAns & TNB yellow; AmmP golden orange; and TNT light tan (buff)

Because of this, it is appropriate to list here some references on color, color designation and color measurements
Refs: 1)Thorpe **3** (1939), 295-306 2)A.H. Munsell, "Munsell Book of Color", Munsell Color Co, Baltimore, Md (1942) 3)W.D.Wright, "The Measurement of Color", A.Hilger, London (1944) 4)R.M.Evans, "An Introduction to Color", Wiley, NY (1948) 5)G. & G. Plochere, "Plochere Color System", Los Angeles, Calif (1948) 6)Kirk & Othmer **4** (1949), 242-51 (Color measurements) 7)E. M.Graves, "Color Fundamentals", McGraw-Hill, NY (1952) 8)Anon, "Color Index", Society of Dyers & Colourists, London and American Association of Textile Chemists & Colorists, Durham, NC, 2nd edition in 4 vols (1955-1959) 9)Anon, "Reinhold Color Atlas", Reinhold, NY (1962) 10)D.B.Judd & G.Wyszecki, "Color in Business, Science and Industry", Wiley, NY (1963)

Colored Clouds.
Colored Lights.
Colored Signals.
Colored Smokes.
Colored Stars.
} Intended to be described under PYROTECHNIC COMPOSITIONS AND DEVICES

Colorimetry, Colorimeter and Colorimetric Analysis. *Colorimetry* is the branch of physics that deals with determining and specifying colors. The instrument used in colorimetry is called *colorimeter;* it determines and specifies colors by referring to other colors. This process will permit,by the proper selection of suitable reagents, the identification of principle ingredients in the sample tested when it is in solution. The depth of color, measured by the colorimeter, gives the quantity of material present in a solution. This is known as *colorimetric analysis* (See also Color

Reactions and Color Reagents)
Refs: 1)F.D.Snell & C.T.Snell, "Colorimetric Methods of Analysis", Van Nostrand, NY: Vol **1** (1948) (Theory, instruments, pH detn); Vol **2** (1949) (Inorganic compds); Vol **2A** (1959) (Including photometric methods); Vol **3** (1953) (Organic compds, 1st book); Vol **3A** (1961) (Including photometric methods); and Vol **3** (1954) (Organic compds, 2nd book) 2)Kirk & Othmer **4** (1949), 267-76 (Colorimetry & fluorimetry) 3)A.Weissberger, "Physical Methods in Organic Chemistry", Interscience, NY, Vol 1, Part 2 (1949), pp1399-1490 (Colorimetry, Photometric Analysis and Fluorimetry, by W. West) 4)G. Charlot & R.Gauguin, "Dosages Colorimetriques, Principes et Méthodes", Masson, Paris (1952) 5)B.Lange, "Kolorimetrische Analyse", Verlag Chemie, Weinheim (1956) 6)N.L.Allport & J. W.Keyser, "Colorimetric Analysis", Chapman & Hall, London (1957) 7)D.F.Boltz, "Colorimetric Determination of Nonmetals", Wiley (1958) 8)E.B.Sandell, "Colorimetric Determination of Traces of Metals", Wiley, NY (1959) 9)Tintometer Ltd "Colormetric Chemical Analytical Methods", The Author, Salisbury, England (1959) 10)Vogel, InorgAnalysis (1961) 738-837 (Colorimetric and spectrophotometric analysis; description of various colorimeters) 11)Pamphlets and catalogs of A.H.Thomas, Will Corp, Central Scientific Co, Fisher Scientific Co. etc which describe various colorimeters, etc.

COLOR REACTIONS AND COLOR REAGENTS. When a test sample (comprising a single compd or its mixt with other ingredients) is treated with an appropriate reagent, a coloration is produced which is characteristic of the compd tested. The reaction, so produced, is known as a *color reaction* and it may serve for identification of the sample. A reagent which produces the coloration is known as a *color reagent*

Color reagents are used extensively for identification of explosives, primary materials used in their manuf, and of various ingredients in explosive, propellant and pyrotechnic compositions

The following *colorimetric reagents* are commonly used in explosives laboratories:
A. *Alkali Hydroxide Reagent* is prepd by dissolving 5 or 10g of KOH (or NaOH) in 100ml of alcohol or water. The 10% aq NaOH soln is known as the *Janovsky's Reagent* (Ref 21,p 805).For testing, dissolve a small amt (ca 0.05g) of the unknown material in a small amt of acetone placed in an indenture of a white, glazed porcelain spot-test plate (or in a small crucible), add 1-2 drops of the reagent and observe the color produced. Many aromatic nitro -compds produce color reactions. For example: 2,4,6-TNT gives blood red color, which turns to brown on standing; 2,3,4,-TNT dark green; 2,4-DNT deep blue turning to black and then fading; 1,3,5-TNB deep red, etc (Refs 1 & 5). Ovenston(Ref 18a) used 6N aq NaOH soln as streak reagent in chromatographic analysis
B. *Ammonium Hydroxide Reagent.* Colver (Ref 1) and Marshall(Ref 5) tabulated colors produced by alcoholic ammonia, but did not describe the prepn of the reagent. The reagent prepd by mixing equal vols of Amm hydroxide and alcohol was used during WWII at Keystone OdnWorks for testing expls

For testing, use the same procedure as described under Alkali Hydroxide Reagent. Many aromatic polynitro-compds produce colors. For example: 2,4,6-TNT gives a red color, turning violet on standing and 1,3,5-TNB gives a yellow-red which persists on standing(Refs 1 & 5)(See also Charts A & B given further in this section)
C. *Ammonium Sulfide Reagent* is prepd by mixing 1 volume of $(NH_4)_2S$ with 1 vol of alcohol

For testing, use the same procedure as described under Alkali Hydroxide Reagent. Many aromatic polynitro- compds produce colors. For example: 2,4,6-TNT gives deep red; 2,3,4-TNT greenish-yel; 2,4,5-TNT indigo blue; all MNT's and 2,4-DNT produce a light yel color(Ref 1,p 235)
D. *Dichromate-Sulfuric Acid Reagent* is prepd by dissolving 0.5g of $K_2Cr_2O_7$ in 100ml of 60% sulfuric acid. It was proposed by Ovenston for use in chromatographic analysis of proplnts, for the location of substituted ureas and urethanes which have been employed as stabilizers and plasticizers(Ref 18a)
E. *Diphenylamine Reagent* is prepd by dissolving 1g of $C_6H_5.NH.C_6H_5$ in 100ml of concd reagent-grade sulfuric acid

For testing, place ca 0.05g of the unknown material in an indenture of a white porcelain spot-test plate, add 3-4 drops of the reagent,

stir and, after 1 min, note the color. A blue color is obtained with substances contg NO_3 or ONO_2 groups(such as KNO_3, BkPdr, NC, NG, DEGDN, etc)(Ref 27, pp 1347-48)(See also Ref 18a and Charts A & B given further in this section). A **blue ring test**, which is similar to brown ring test described under Reagent G may be used

F. *Ethylenediamine Reagent* is a 65-68% aq soln of $H_2N.CH_2.CH_2.NH_2$. For testing, place a small amt(ca 0.05g) of the unknown material in an indenture of a white porcelain spot-test plate, add 2-3 drops of the reagent, stir and note the color. Many expls produce colors. For example: TNT gives a maroon color, Tetryl red, PA & Explosive "D" orange, etc (Ref 27,pp 1347-48)(See also Chart A)

G. *Ferrous Ammonium Sulfate Reagent.* Dissolve 39.214g of reagent grade $Fe(SO_4).(NH_4)_2SO_4.6H_2O$ in 1:9 sulfuric acid(previously boiled and cooled) and make up to 1 liter. This is 0.1N soln and can also be used for quantitative analysis. Brownish coloration is produced by nitric acid or nitrates(Ref 13,p 726). Ferrous sulfate soln(prepd by dissolving $FeSO_4.7H_2O$ in w & 60% H_2SO_4 as described in Ref 9, Vol 1, Chap I, p 14), may be used in lieu of ferrous ammonium sulfate. The **brown ring test** for nitric acid and nitrates is conducted as follows: Pour into a test tube 2ml of distd w, followed by 1ml of soln to be tested. Place the tube in an inclined position(about 45° angle) and pour $FeSO_4$ reagent slowly in such a manner that it will run down the wall of the tube without mixing with the contents of the tube. The reagent, being heavier than the diluted sample will accumulate as a lower layer in the tube. If a NO_3 group is present in an appreciable amt, a brown ring will form on the border of two layers. If no ring forms, but the presence of NO_2 group is suspected, use the DPhA reagent(Reagent D)(Ref 9, Vol 3, Chap IX,p 5)

H. *Griess' Reagent,* modified by Ilosvay is prepd in the following manner: Soln 1. Dissolve 1g of CP sulfanilic acid in 300ml of soln prepd by diluting 125 ml glacial AcOH with NH_3 - free distd w; Soln 2. Add 0.2g of alpha-naphthylamine to 100ml of hot distd w. Cool and filter into 200ml of dil AcOH. When both solns have cooled mix them and transfer to a brown glass-stoppered bottle. Protect the soln from air; can be used as long as the reagent remains colorless. Red color is produced with compds contg N & O (Ref 26,p 164 & Ref 27,p 1556). It has been used in Webster's Test for detn of TNT in urine(See **R**)

I. *Ilosvay's Reagent* is prepd in the following manner: Dissolve 1g of $Cu(NO_3)_2.5H_2O$ in ca 30ml distd w contained in a 50ml volumetric flask. Add 4ml of concd NH_4OH soln and 3g of hydroxylamine hydrochloride, $NH_2OH.HCl$. Shake until the mixt becomes colorless and dilute to volume. If the reagent is protected from oxidation by a layer of mineral oil, or if a piece of Cu wire is added, it will retain its reactivity for a long time. When acetylene or its derivatives are bubbled thru the reagent, red coloration or ppt is produced(Ref 18,p 516)

J. *Janovsky's Reagent.* See under Alkali Hydroxide Reagent

K. *Liebermann's Reagent* is prepd by mixing phenol with concd sulfuric acid. The test consists in warming a small amt of the unknown substance with few drops of the reagent and observing the color produced. Aromatic nitroso compds and aliphatic nitroso- and isonitroso compds produce a red color which turns blue on addn of alkalies(Ref 25,pp 164-65)

L. *Muraour's Reagent* is prepd by dissolving in 100ml of distilled w, 5g of K dichromate, 100ml of glacial AcOH & 50ml of concd sulfuric acid (Ref 17,p 329). For testing with this reagent, Tavernier & Lamouroux placed 1ml of 1% acetonic soln of sample on a watch glass and added 1-2 drops of the reagents. Twenty six substances, suitable as ingredients of proplnts, were tested and results are given in Ref 24

M. *Nessler's Reagent* can be prepd by dissolving 5g of KI in a minimum quantity of cold distd w, and adding a saturated aq soln of HgI_2 until a faint ppt is formed. Then 40ml of 50% aq KOH soln is added and the mixture allowed to stand until clarified by settling. After diluting to 100ml with distd w, the mixt is allowed to settle once more and then the clear liquid is removed by decantation

For testing, place ca 0.05g of the unknown sample in a 5-ml beaker, add 2-3ml of distd w, stir for 5 mins and observe the color. Dip one end of a strip of universal pH indicator paper, and note any change in color. Add a

drop of Nessler's reagent and note the color of any ppt formed. Explosive "D", AN, Picratol & Ammonal give brown ppt, while NGu gives white ppt(Ref 27,pp 1347-48)(See also charts A & B given further in this section)

N. *Pyridine Reagent.* See Ref 23

O. *Schiff's Reagent* can be prepd by dissolving 0.2g of fuchsin(rosaniline hydrochloride) in hot distd w(120ml), cooling and adding this soln to a 200ml volumetric flask contg the soln of 2g Na bisulfite in 20ml of distd w. Finally 2ml of concd HCl is added to the mixt and it is diluted to 200ml mark. The reagent is then placed in a refrigerator where it should remain for at least 24hrs before use. It should be stored in an amber colored bottle and protected from air. Used for detection of methanol(blue or violet color) or of formaldehyde(dark violet color)(Ref 13,p 720 & Ref 18, pp 616 & 671)

P. *Tetramethylammonium Hydroxide Reagent.* See Ref 21

Q. *Thymol Reagent* is prepd, right before use, by mixing crystalline thymol with concd H_2SO_4. For testing an unknown substance place ca 0.05g sample in an indenture of a white porcelain spot-test plate, add an equal amt of thymol and 3 drops of concd H_2SO_4. Stir the mixt and, after allowing it to stand for 5 mins, note the color. Green color is produced with many expls, as can be seen from Charts A & B(Ref 27,pp 1347-48)

R. *Webster's Reagent and Improved Test for TNT in Urine.* The reagent is a freshly prepd 5% alcoholic soln of KOH. For the test, place 10ml of urine in a test tube, add ca 10ml of 20% H_2SO_4 soln, shake and bring to a boil. Cool under tap w, add ca 10ml of ether, stopper and shake. After allowing to stand for 10 mins pour some of the supernatant ether into the 2nd tube, add 2ml of the reagent and note the color. If brown color appears and persists, no TNT is present, but if reddish-purple color develops TNT is present. The same test may be used for detection of Tetryl. In this case, straight ether extraction is substituted by 1:3-acetone -ether mixt(Refs 7 & 9). Another modification of Webster's test was proposed by Elvove (Ref 2). He used the Griess' reagent in lieu of 5% KOH soln

CHART A
Changes of Color on Addition of Reagents

Explosive	A. Alkali Hydroxide	B. Ammonium Hydroxide	E. Diphenyl-amine	F. Ethylene-diamine	M. Nessler's Reagent	Q. Thymol Reagent
Amatol	Same as	for TNT	Dirty grn	Maroon	No ppt	Grn
Ammonal	—	—	Dirty grn	Maroon	Brn ppt	Grn
Ammonium Nitrate	No color	No color	Dirty grn	No color	Brn ppt	Grn
Ammonium Picrate	Yel	Yel	—	Orn	Brn ppt	
Aniline:						
MNA's	No color	No color	—	—	—	—
2,4-DNA	Reddish	No color	—	—	—	—
2,4,6-TNA	Red-orn	No color	—	—	—	—
1,2,3,5-TeNA	Red-orn	No color	—	—	—	—
Anisole:						
MNAns	No color	No color	—	—	—	—
2,4-DNAns	Pink	No color	—	—	—	—
2,4,6-TNAns	Red	Orn	—	—	—	—
2,3,5,6-TeNAns	Red	Orn	—	—	—	—

Note: See end of Chart A for the meaning of abbreviations used

CHART A
Changes of Color on Addition of Reagents (cont)

Explosive	A. Alkali Hydroxide	B. Ammonium Hydroxide	E. Diphenyl-amine	F. Ethylene-diamine	M. Nessler's Reagent	Q. Thymo Reagent
Benzene:						
MNB(coml)	No color	No color	—	—	—	—
DNB(coml)	Viol to deep wine	No color	—	—	—	—
TNB(coml)	Dk red	Deep red	—	—	—	—
TeNB	Orn	Red turning to orn	—	—	—	—
Black Powder	No color	No color	Blue	—	—	Grn
Cellulose Nitrate	—	—	Blue	—	—	Grn
Composition A-3, B & C-3	See under Tests for Composite Explosives					
Cresol:						
3-N-p-Crs	Yel	No color	—	—	—	—
3,5-DN-p-Crs	Yel-orn	Yel-orn	—	—	—	—
2,4,6-TN-m-Crs	No color	No color	—	—	—	—
Cyclotri-methylenetri-nitramine(RDX)	See Chart B					
Diethanol-nitramine di-nitrate(DINA)	No color	No color	See also Chart B			
Diethylene-glycol di-nitrate(DEGDN)	—	—	Blue	—	No ppt	Brn
Dinitro-compounds		See under corresponding compds, such as Aniline, etc				
Diphenyl-amine(DPhA):						
2,4-DNDPhA	Red-orn	No	—	—	—	—
2,4,6,3'-TeNDPhA	Red	Red	—	—	—	—
2,4,6,2',4',6'-HNDPhA	Red	Red-orn	—	—	—	—
EDNA	See Ethylenedinitramine					
Ednatol	—	—	Intense blue	Maroon	No ppt	Orn
Ethylenedia-mine dinitrate (EDD)	See Chart B					
Ethylenedi-nitramine (EDNA or Haleite)	See also Chart B		Blue	No color	No ppt	Orn

CHART A
Changes of Color on Addition of Reagents (cont)

Explosive	A. Alkali Hydroxide	B. Ammonium Hydroxide	E. Diphenyl-amine	F. Ethylene-diamine	M. Nessler's Reagent	Q. Thymol Reagent
Glycerin nitrate(NG)	—	—	Deep blue	—	No ppt	Grn
Guanidine, Nitro(NGu)	Lt yel	Lt yel	Blue	—	Wh ppt	Grn
Haleite	Same as EDNA					
Mannitol Hexanitrate (HNMnt)	No color	No color	—	—	—	—
Mesitylene, Trinitro (2,4,1'-TNMes)	Pink	No color	—	—	—	—
Mononitro-compounds	See under parent compds					
Naphthalene:						
α-MNN	No color	No color	—	—	—	—
DNN	Orn	Lt pink	—	—	—	—
TNN	Scarlet	Orn-red	—	—	—	—
TeNN	Orn-red	No color	—	—	—	—
Pentaerythri-tol tetranit-rate(PETN)	—	—	Dirty grn	No color	—	Grn
Pentolite	Same as for TNT		Dirty grn	Maroon	No ppt	Grn
Phenetole:						
2,4-DNPht	Viol	No color	—	—	—	—
2,4,6-TNPht	Red	Yel-orn	—	—	—	—
Phenol:						
MNPh	Lt yel	Lt yel	—	—	—	—
DNPh	Lt yel	Lt yel	—	—	—	—
2,4,6-TNPh (Picric Acid)	Yel-orn	Lt yel becoming sl reddish	—	Orn	No ppt	—
Picratol	Same as for TNT		—	Maroon	Brn ppt	—
Resorcinol, Trinitro (2,4,6-TNR)	Lt yel	Lt yel	—	—	—	—
Starch Nitrate(NS)	No information at our disposal					

Note: See end of Chart A for the meaning of abbreviations used

CHART A
Changes of Color on Addition of Reagents (cont)

Explosive	A. Alkali Hydroxide	B. Ammonium Hydroxide	E. Diphenyl-amine	F. Ethylene-diamine	M. Nessler's Reagent	Q. Thymc Reagent
Tetryl	Deep red	—	Blue	Red	—	Grn
Tetrytol	Same as for TNT		Blue	Maroon	No ppt	Grn
TNT	See under Toluene					
Toluene:						
o-MNT	Very sl yel	Very sl yel	—	—	—	—
m-MNT	No color	No color	—	—	—	—
p-MNT	No color	No color	—	—	—	—
2,4-DNT	Deep blue turning to bk	No color	—	—	—	—
DNT(coml)	Same as above					
2,4,6-TNT	Deep red-viol turning to brn	—	No color	Maroon	—	—
2,3,4-TNT	Dk grn ppt, which redisolve	Brillant yel, on standing	—	—	—	—
2,4,5-TNT	Jet bk assuming viol tinge on standing	Indigo-blue on standing	—	—	—	—
TNT(coml)	Deep red-brn	Viol increasing on standing and then changing to red-brn	—	—	—	—
Torpex	—	—	Intense blue	Maroon	No ppt	—
Tridite	—	—	—	Orn	No ppt	—
Trimonite	—	—	Red	Orn	No ppt	—
Trinitro-Compounds	See under corresponding parent compds					
Tritonal	—	—	No color	Maroon	—	—
Xylene:						
2,3-&2,4 -DN-p-X	Lt pink	No color	—	—	—	—
4,6-DN-m-X	Grn	No color	—	—	—	—
2,4,6-TN-m-X	Dk blue	No color	—	—	—	—
3,4,5-TN-o-X	Blue-bk	No color	—	—	—	—
3,4,6-TN-o-X	Blue	No color	—	—	—	—
2,3,5-TN-p-X	Red	No color	—	—	—	—

Abbreviations used in Table A: bk - black; brn - brown; dk - dark; grn - green; lt - light; orn - orange; ppt - precipitate; sl - slight; viol - violet; wh - white; yel - yellow

Analytical Procedures Based on Color Reactions Rapid Method of Identification of Common Explosive Compounds and of Explosive Mixtures

During WWII, when many foreign expls were found in the field by US Ordnance personnel, it became evident that a simple, rapid method of testing should be developed. On the request of Delaware Ordnance Depot a simple procedure was developed in 1944 at Picatinny Arsenal (Ref 10a) and this procedure was taken as a basis of work conducted under Shriner for NDRC (Ref 14)

Following procedures may be considered as a summary of procedures described in Refs 9, 10a, 14, 22a, 26 and in some PicArsnChemLab Repts

I. *Preliminary Examination of an Unknown Material.* Observe the color & odor and whether it is crystalline or amorphous. If the sample appears to be nonhomogeneous, examine it under a magnifier, or microscope. Det its sensitivity to flame by touching a few grains of substance with a flame of gas burner or of a lighted match. BkPdr, colloidal proplnts and some initiating expls do not explode but just flash. Most solid non-initiating expls burn without expln. Sensitivity to impact is detd by striking with a hammer a few grains of material placed on a steel plate, and sensitivity to friction is detd by rubbing cautiously a few grains of material in a porcelain mortar. If the material explodes or deflagrates, it is considered sensitive to mechanical action. All initiating expls are sensitive to impact or friction and most initiating expls are sensitive to impact, but not all of them are very sensitive to friction. If the material seems to be an initiating expl, no attempt should be made to det its mp, but otherwise it is advisable to det mp using either Fisher-Johns apparatus or capillary method. If the material melts sharply and its mp corresponds to one of the known HE's (such as 80.5° for TNT, 122° for PA, 66° for TNAns, 128° for Tetryl, 141° for PETN, 177° for EDNA, 202° for RDX, etc), it can be assumed that it is not a mixt. If, however, the material does not melt sharply, and its mp does not approximate that of any of the common HE's, it can be assumed that it is to be a mixt

It is also very useful to see if the sample is sol in water(See item XVI), carbon tetrachloride(See item XVII), and acetone(See item XVIII). Other solvents, such as alcohol, ether, benzene, etc may also be used

II. *Treatment with Sulfuric Acid.* Place ca 0.05g of unknown material in an indenture of a white porcelain spot-test plate, add 3 drops of concd H_2SO_4, stir and observe if any bubbles of gas rise to surface of liquid. Most expls are not affected, but EDNA decomposes(Ref 10a)

III. *Treatment with Thymol Reagent.* See item **Q** under colorimetric reagents and Charts A & B given in this section

IV. *Treatment with Ethylenediamine Reagent.* See item **F** under colorimetric reagents and Chart A

V. *Treatment with Diphenylamine Reagent.* See item **E** under colorimetric reagents and Charts A & B. A yel color indicates the presence of one of the expls: TNT, TNAns, HNDPhA, PA, AmmPic or DNPh; viol, blue or black color indicates DNT, DNB, DINA, Tetryl, PETN, RDX, EDNA, EDD or AN; red color indicates α-MNN

VI. *Treatment with Diphenylbenzidine Reagent.* Place a few small crysts of unknown in an indenture of a white porcelain spot test, add 2 drops of diphenyl-benzidine reagent(a satd soln of $C_6H_5.NH.C_6H_4.C_6H_4.NH.C_6H_5$ in glac AcOH), and then 1 drop of concd H_2SO_4. Observe the color after 1 min. A blue color indicates one of the following expls: TNT, TNAns or HNDPhA; yel color or no color -PA, AmmPic or DNPh (See also Chart B)

VII. *Treatment with Benzylamine Reagent.* Add ca 0.1g of sample to ca 5ml of benzylamine reagent(a 5% soln of $C_6H_5.CH_2.NH_2$ in 95% alcohol) in a test tube. Note the color developed and see if any change takes place after placing the tube in boiling w. A viol color indicates the presence of TNT and orn color TNAns or HNDPhA(See also Chart B)

VIII. *Treatment with Potassium Chloride.* Heat on a steam bath a test tube contg a 0.1g of the unknown, together with a 3ml of 1% Na_2CO_3 soln. After allowing to cool and settle, transfer a few drops of the clear supernatant liquid to another test tube and add an equal vol of satd KCl soln. A heavy orn-red ppt indicates HNDPhA and no reaction indicates TNAns (See also Chart B)

IX. *Treatment with Benzaldehyde Reagent.* Dissolve ca 5 mg of the sample in a 5ml of

alcohol and add 2 drops each of benzaldehyde and piperidine. Warm the soln for 1 min in boiling w, then cool and note the color. Add 5 drops of concd H_2SO_4 and note any change in color. An orn-red color which lightens after acidification indicates the presence of TNAns (See also Chart B)

X. *Treatment with Modified Griess' Reagent.* Dissolve in a test tube ca 0.1g of the sample in ca 3ml of 1% of dioxane, and add ca 3ml of 10% NaOH soln. Heat the tube in boiling w for 1 min(shaking occasionally), cool and add glac AcOH to make thesoln neutral or slightly acidic. Place one drop of the soln on a white spot test plate and add 1 drop of a 0.5% soln of sulfanilic acid in 50% AcOH and 1 drop of a 0.5% soln of dimethyl-α-naphthylamine in 50% AcOH. Allow to stand for 30 secs and note the color. No color indicates DNPh. Confirm it by next test. A red color indicates PA or AmmP. Confirm it by Nessler's test(See also Chart B)

XI. *Treatment by Meyer-Drutel Method.* Prepare 15ml of satd aq soln of the sample, filter the soln and transfer 10ml of the filtrate to a test tube. Add 2ml of glac AcOH & 0.2g Zn dust, shake the mixt, allow to stand 15 mins and note the color of liquid. Filter and add to the filtrate 2 drops of 0.1% $K_2Cr_2O_7$ w/o shaking. A pale pink color which turns, on addn of $K_2Cr_2O_7$, to deep red rose in the upper part of soln, confirms the presence of DNPh(See also Chart B)

XII. *Treatment with Nessler's Reagent.* Prepn of reagent is described as item M under colorimetric reagents. For test, prep a satd soln of sample by shaking ca 0.1g with ca 3ml of w. After allowing to settle, decant ca 1ml into a test tube contg ca 1ml of Nessler's reagent. A brn ppt confirms the presence of AmmPicrate and no ppt confirms the presence of PA(See also Chart B)

XIII. *Treatment with Alkali-Acetone Reagent.* Dissolve in a test tube ca 0.1g of the sample in ca 5ml of acetone, add ca 2ml of 10% NaOH soln and shake the mixt. After allowing to stand for 1min, note the color. A blue color indicates DNT and its presence may be confirmed by M-D(Meyer-Drutel) Test, which would give purple-red. A viol color indicates DNB which would give red-brn by M-D test. A light red indicates PETN, which would give with benzylamine no color(See item VII). A deep red indicates Tetryl, which would give with benzylamine a red color. No color might indicate the presence of EDD, EDNA, RDX, AN or DINA. A cream ppt with Nessler's reagent confirms the presence of EDD, a brn ppt the presence of AN and no ppt, the presence of either EDNA or RDX. If no color is produced with modified Griess' Reagent (See item X), it is EDNA and if red color is obtained, the presence of RDX is indicated(See also item XV, Chart B & Tests for Composite Explosives described in this section)

XIV. *Treatment with Franchimont Reagent.* Mix in a test tube ca 0.1g of the sample with ca 5ml of glac AcOH and add 2 drops of dimethyl-α-naphthylamine. Add ca 0.2g of Zn dust and allow to stand for 2 mins. A brn-red color confirms the presence of DINA(See also Chart B)

XV. The presence of RDX may be confirmed by one of the following tests:

a) *Formaldehyde Test.* Add ca 0.5g of the sample to ca 5ml of 80% H_2SO_4 contained in a test tube, heat the mixt at ca 100° for 3mins and cool. Dilute to 20ml with w and add 10% NaOH until the soln is neutral or faintly acidic. Filter and add to the filtrate 5 drops of *Methone Reagent*(which is a 10% soln of 1,1-dimethyl-cyclohexan-3,5-dione, $(CH_3)_2C_6H_6O_2$, in 95% alcohol). A cloudiness settling in ca 5 mins to a fine ppt, is a positive test for RDX

b) *Sodium Pentacyanoaminoferroate(SPAF) Test.* Dissolve ca 1mg of the sample in ca 5ml of acetone contained in a test tube and add 5ml of w. Dissolve in this liq a piece of SPAF, ca 2mm across and add 2drops of 10% NaOH soln. A blue-grn color indicatesthe presence of RDX

c) *Thymol Test.* Use the procedure described under item Q. Thymol Reagent. A deep red color is a positive test for RDX

Quantitative tests for RDX are described in this Volume under Cyclotrimethylenetrinitramine (RDX), Analytical Procedures

XVI. *Alkali Solubility Test.* Shake ca 0.5g of the sample for 2mins in a test tube with 5ml of 10% NaOH soln, filter and acidify the filtrate with concd HCl. Cool the tube under tap w, and rub the inside of the tube with a glass rod. A white ppt, appearing after about 5mins, is a positive test for EDNA and its presence can be confirmed by treating the sample with Thymol Reagent - a brown color is a positive test for EDNA (See also Chart B)

Testing of an Unknown Sample by Means of Solvents

XVII. *Treatment with Water.* Stir in a small test tube ca 0.2g of unknown subst with ca 2ml of w and observe whether the sample dissolves. A white subst which is nearly or completely insol, can be one of the following: TETN, RDX, DINA or EDD. A wh sol subst can be AN, Amm or K perchlorate, etc and a wh subst which is appreciably sol in w can be EDNA. A tan subst, practically insol in w, is usually TNT. A yel subst appreciably sol, can be PA or AmmPic, while a yel subst which is practically insol, can be DNPh, HNDPhA, MNN, Tetryl or TNAns; DNB & DNT are pale yel & nearly insol. If a sample of any color is only partially sol, it is usually a mixt, such as Amatol, Tridite, BkPdr, etc. Test the aqueous solns by color reagents and compare the colors with those given in Charts A & B

XVIII. *Treatment with Carbon Tetrachloride.* Shake in a small test tube ca 0.2g of ground unknown subst with ca 5ml of CCl_4 and allow to stand for 5mins. If the sample is completely sol it is usually TNT. Confirm its presence by treating the soln with one or more colorimetric reagents listed in Charts A & B. If the sample is only partially sol in CCl_4, remove the liquid portion and test it for TNT

XVIX. *Treatment with Acetone.* If the subst is not completely sol in CCl_4 proceed as follows:

a)Shake in a testtube ca 0.5g of the sample with ca 10ml of acetone and allow to stand. If the sample is completely soluble, it might consist of one or more of the following: DNB, DNPh, EDNA, HNDPhA, PETN, PA, RDX, Tetryl, TNAns, TNT, Comp B, Cyclotol, Ednatol, Pentolite, Picratol, Tetrytol, Tridite or Trimonite. If a residue remains, decant the liquid, divide it among three test tubes and save the residue for tests described further under operation e

b)Add to one of the test tubes contg the soluble portion, ca 20drops of aq 2% NH_4OH soln, stopper the tube, shake and allow to stand for 5 mins. Note the color: pink indicates the presence of Comp B; red permanent, HNDPhA and if the color is intense red(which has changed from light red), Tetryl is present(See further under Tests for Composite Explosives)

c)If the soln in the 1st tube remains colorless on addn of 2% NH_4OH, add to the 2nd test tube ca 10drops of aq 2% KOH soln and note the color: orange indicates PA, and no color change, either PETN or RDX

d)If the soln in the 2nd tube remains colorless, add rapidly to the soln in the 3rd test tube an equal vol of Diphenylamine Reagent(See item E) and observe the color: deep blue indicates PETN and no color change indicates RDX

The presence of RDX may be confirmed by the Thymol Test(See item Q). A pink or rose color indicates the presence of RDX

e) If theunknown substance is only partially soluble in acetone, treat the solid(left after opn a) with an aq 2% NH_4OH soln, shake and observe the color. If it is pink Amatol or Torpex may be present and if no color is produced AN, AmmPic, Comp A, or Comp C may be present(See further, under Tests for Composite Explosives)

f)If the color produced with NH_4OH is pink (See operation e) shake thoroughly a new portion of the unknown with acetone and add to the resulting slurry an equal vol of Diphenylamine Reagent (See items E & V), allowing it to run slowly down the inside surface of the tube. If the soln turns deep blue particularly around the residue at the bottom of the tube, the expl is Amatol. If there is no color change, the expl may be of the Torpex type(See further, under Tests for Composite Explosives)

g)If no color is produced with NH_4OH(See opn e) shake a new portion of the unknown with acetone and add drop by drop the Diphenylamine Reagent(See items E & V). If a white voluminous ppt forms, which redissolves upon the further addn of Reagent E, the presence of AmmPic is indicated. If on addn of a few drops of Reagent E no wh ppt forms, add quickly an excess of Reagent E and, if a deep blue color appears, the expl is AN; and if no color is produced, the expl may be either Comp A-3 or Comp C-3. Comp A-3 can be distinguished from Comp C-3 by the difference in physical props or by tests described further under Tests for Composite Explosives. Comp A is a cryst subst which crumbles to a powder, while Comp C is soft and plastic

Tests for Composite Explosives

Amatols, which are mixts of AN & TNT(See Vol 1,p A158 of this Encycl), give brown ppt in Nessler's Test(See items M & XII) and a deep red color in theAlkali-Acetone Test (See item XIII)

Chart B
Color Tests for United States Military Explosives

Explosive	Alkali Acetone	Alkali soly test(after acidification)	Benzaldehyde		Benzylamine		Diphenyl-amine
			Original	After acidification	Cold	Warming	
AN	No reaction	No ppt	No reaction		No reaction		Grey-bk
AmmPic	Orn	Pale yel ppt	No reaction		No reaction		Partial yel soln
DINA	No color	No ppt	No color		No change		Blue-bk
DNB	Deep viol	No ppt	No reaction		No reaction		Faint viol
DNPh	Deepened yel	Pale yel ppt	No reaction	Yel which disappears	No reaction		Dissolves yel
DNT	Deep blue	No ppt	No reaction		No reaction		Viol
EDD	No color	No ppt	No color		No reaction		Grn-bk
EDNA	No reaction	Wh ppt	No reaction		No reaction		Vigorous reaction; deep blue
HNDPhA	Deepened red	Yel ppt	Orn-red	Deep orn	Orn-red	Becomes less orn	No reaction; yel
MNN	No reaction	No ppt	No reaction	Very faint yel	No reaction		Deep blood red
PA	Orn soln	Pale yel ppt	No reaction		No reaction		Partial yel soln
PETN	Lt reddish	No ppt	Orn	No reaction	No reaction		Bk-grn
RDX	No reaction	No ppt	Faint yel	Color lightens	No reaction	Ppt	Deep grn -blue
Tetryl	Deep red	No ppt	Red	Color lightens	Red	No reaction	Bk-grn with yel soln above
TNAns	Deep red	No ppt	Orn-red	Color lightens	Orn	No reaction	Yel soln
TNT	Very deep red-viol	No ppt	Viol	Becomes reddish	Viol	Color deepens	Partial soln yel

Abbreviation used in Table B: AN-Ammonium nitrate; AmmPic-Ammonium picrate; bk-black; brn-brown; EDD - Ethylendiamine dinitrate; EDNA - Ethylenedinitramine; grn - green; HNDPhA - Hexanitrodiphenylamine; ppt - precipitate; RDX - Cyclotrimethylenetrinitramine; soln - solution; soly - solubility; TNAns -

Chart B
Color Tests for United States Military Explosives

Diphenyl-benzidine	Franchi-mont's	Griess' Modified	Meyer-Drutel's After re-duction	Meyer-Drutel's After addg $K_2Cr_2O_7$	Nessle rs'	Thymol (after 2 mins)
Deep blue	Clear red	No change	No color	Very pale yel	Brn ppt	Grn-brn
No reaction	Lt grn	Deep red	Tan	Blue	Brn ppt	Yel
No color	Brn-red	Deep red	No change		No ppt	Grn
No reaction	Faint brn	No change	Red-brn	Red-brn	No ppt	Yel
No reaction	Clear red	No change	Pale pink	Deep rose red	No ppt	Yel
No reaction	Faint brn	No change	Purple	Purple-red	No ppt	Yel
Deep blue	No col	Faint pink	No color	Very pale yel	Cream ppt	Deep grn-bk
Deep blue	No reaction	No reaction	No color	Very pale yel	No ppt	Brn
Faint blue	Lt brn-red	Deep red	Pink	Red-brn	No ppt	Yel
Deep blue	No reaction	No change	No color	Pink	No ppt	Yel
No reaction	Deep grn	Deep red	Yel to brn	Blue-bk	No ppt	Very pale yel
Deep blue	Clear orn-red	Pink	No color	Very pale yel	No ppt	Grn
Deep blue	Lt brn	Deep red	No color	Very pale yel	No ppt	Deep red
Deep blue	Brn	Deep red	Brn	Grn-brn	No ppt	Grn
Lt blue	Lt brn	Deep red	Brn	Brn	No ppt	Yel
Lt blue	Brn	Deep red	Brn	Brn	No ppt	Yel

DINA - Diethanolnitramine; dk-dark; DNB - Dinitrobenzene; DNPh - Dinitrophenol; DNT - Dinitrotolune; lt-light; MNN - Mononitronaphthalene; orn-orange; PA - Picric acid; PETN - Petaerythritol tetranitrate; Trinitroanisole; TNT - Trinitrotoluene; viol - violet; wh - white; yel - yellow

For more definite identification, use the procedures described in Vol 1,p A164-L of this Encycl

Ammonals, which are mixts of AN, TNT & Al (See Vol 1,p A287), give a brn ppt with Nessler's Reagent(See items M & XII) and a deep red color with Alkali-Acetone Reagent(See item XIII).These tests do not indicate, however, if Al is present

For more definite identification of components, digest ca 0.2g of the expl with 3ml of distd w in a 10ml beaker and decant the liq thru a filter to an evaporating dish. Evap to dryness and test for AN(See Charts A & B). Digest the insol portion with a three 3ml portion of acetone decanting thru a filter and examine the residue, for Al, under microscope. Evap the filtrate to dryness by warming gently, and test the deposit for TNT by one or more colorimetric tests listed in Charts A & B

Quantitative detn of ingredients in Ammonals is described in Vol 1,p A292-R of this Encycl

Composition A-3, which is a blend of RDX with a small amt of wax(See this vol, under Composition A Type Explosives),gives positive reactions for RDX, when using colorimetric tests described under item XV. These tests do not, however, identify wax

Complete qualitativeanalysis is conducted as follows: Warm ca 0.1g of the unknown with 2-3 drops of acetone in a 10ml beaker and allow to stand for 5 mins. Evap the acetone by gently warming on a water-bath, cool and add 2ml of CCl_4. Cover the beaker and warm the contents, occasionally swirling the beaker. Cool the mixt, allow to settle and decant the supernatant liquid into a 5ml beaker. Evap to dryness and see if the residue resembles a wax(and not a tarry substance). Dry the material insol in CCl_4, and examine it for RDX, as described under item XV

Quantitative analysis is described in this volume under Composition A Type Explosives, Analytical Procedures

Composition B, which is a blend of RDX & TNT with ca 1% of wax(See this vol under Composition B Type Explosives and Cyclotols) gives positive reactions with colorimetric reagents used for inentification of RDX or TNT by warming

For conducting the complete test, digest, by warming, ca 0.2g of the unknown, with 2-3ml of chloroform in a 10ml beaker covered with a watch glass. After 10 mins, of warming, with occasional swirling, decant the supernatant liquid thru a paper filter and evap the filtrate to dryness. The resulting solid is a mixt of TNT & wax. The residue remaining on filter is impure RDX. Purify it by washing with three 3ml portions of chloroform, followed by drying. Apply any of the tests given under item XV. Treat the deposit(obtd on evapn of chloroform from the filtrate), with acetone and test the sol portion forTNT. The insol portion is wax

Quantitative analysis is described in this volume under Composition B Type Explosives and Cyclotols, Analytical Procedures

Composition C-3, which is a plastic blend of RDX, plasticizer & NC(See this vol under Composition C Type Explosives), can be qualitatively analyzed in the following manner:

Place ca 0.2g sample of the unknown in a 10ml beaker, add 5ml benzene and digest for 10mins, crushing any lumps present. Decant the supernatant liquid thru a filter paper, evaporate the benzene with gentle heating and observe if any dark, tarry substance remains. This subst is a plasticizer, which usually consists of DNT, TNT, MNT & Tetryl. Wash the material insol in benz directly on filter paper with three 3ml portions of 2:1 ether-ethanol mixt and collect the washings in a beaker. Dry the residue on the filter and test it for RDX, as described under item XV. Add to the filtrate ca 15ml of distd w and heat the beaker on a steam bath to evaporate the ether-ethanol. The presence of white ppt indicates NC. Collect it on a filter paper and confirm the presence of NC by colorimetric tests with Diphenylamine Reagent(See item E), which gives a blue color and with Thymol Reagent (See item Q), which gives a green color

Quantitative analysis ofComp C-3 described in this volume under composition C Type Explosives, Analytical Procedures

Cyclotols, which consist of RDX and TNT (See Cyclotols in this vol), can be tested in the same manner as Composition B, omitting the test for wax

Quantitative tests are described in this volume under Composition B Type Explosives and Cyclotols, Analytical Procedures

Ednatol, which consists of EDNA(ethylenedini-

tramine) and TNT, produces a red color with Alkali-Acetone Reagent(See item XIII), and a grn-brn with Thymol Reagent(See item Q). A final proof of its compn is given by an Alkali Solubility Test(See item XVI) which produces a wh ppt

Pentolite, which is a blend of PETN with TNT, produces a red color with Alkali Acetone Reagent(See item XIII) and a red color with Franchimont's Reagent(See item XIV)(See also Charts A & B)

Picratol, which consists of AmmPic & TNT, can be identified after separating the ingredients. This can be done by treating it with one of the solvents for TNT, such as acetone and then testing the soln for TNT and the residue for AmmPic(See Charts A & B)

Tetrytol, which consists of Tetryl & TNT, gives a red alkali-acetone test, a deep grn thymol test and a grn-bk DPhA test. For more definite identification, the ingredients are sepd and then tested individually. Cabron tetrachloride, which is a much better solvent for TNT than for Tetryl, can be used for sepn of *Torpex,* which consists of RDX, TNT & Al, can be identified in the following manner:

Place ca 0.2g of the unknown in a 10ml beaker and extract with three 5 ml portions of acetone. Dry the residue and observe (under magnifier) if it has characteristic appearance of metallic Al. Evap the extract and treat the residue with three 3ml portions of benz. Test the soln for TNT and the residue for RDX(See also Charts A & B)

Tridite, which consists of PA & DNPh, gives colors characteristic for PA, such as orn color with ethylenediamine & deep red with the Griess Test(Modified)(See Charts A & B). None of the known colorimetric tests will detect DNPh in the presence of PA, because the colors produced by DNPh with some reagents (See Chart B) are always obscured by colors produced by PA

Trimonite, which consists of PA & MNN, gives a deep red Griess' test(characteristic for PA) and a deep blood-red DPhA test(characteristic for MNN)(See Chart B)

Tritonal, which consists of TNT & Al, produces with reagents colors characteristic for TNT (See Charts A & B). For identification of Al, extract a 0.2g sample with three 5ml portions of acetone, dry the residue and examine it for metallic Al under a magnifier

Tests for Initiating Explosives

A good description of tests for Lead Azide (LA), Mercuric Fulminate(MF), Diazodinitrophenol(DADNPh), Lead Styphnate(LSt) & Tetracene are given in Ref 22a, pp 267-69. The same tests are described in Ref 27,pp 1345-47

These tests are not repeated here but will be given, together with other tests under individual compds

A more detailed description of tests for LA is given in Vol 1,pp A580-R to A587-R of Encycl

Tests for Propellants

Since std colloidal proplnts are mixts of colloided NC with explosive(such as NG, NGu, DEGDN, etc) and nonexpl ingredients(such as DPhA, EtCentr, DBuPh, etc), no simple colorimetric tests are known for identification of components without preliminary separation of them. The separation can be done by solvent extractions, fractionation of the extracts or by chromatographic separation

The tests will be described under Propellants Analytical Procedures(See also Cannon Propellants, Analytical Procedures in Vol 2, p C38-L of this Encycl)

This section on Color Reactions and Color Reagents was reviewed by Mr. E.F. Reese of Picatinny Arsenal

Refs: 1)Colver(1918), 234-36(Color reaction with NaOH or NH_3 solns of nitroderivs of benzene, toluene, naphthalene, etc; a table) 2)E. Elvove, IEC **11**, 860-64(1919) [Modification of Webster's test for colorimetric detn of TNT in urea, described in BritMedJ **2**, 845(1916). Instead of using Webster's KOH soln, Elvove proposed to use *Griess' Reagent*(qv)] 3)H.W. van Urk, ChemWeekblad **21**, 169-71(1924) & CA **18**, 2142(1924)(Discussion on formation of colored compds by reaction of alkali alcoholates with polynitrocompds of the aromatic series) 4)L. Desvergnes, AnnChimAnalyt **13**, 321-22 (1931) [Color reactions of various nitrocompds were detd by prepg two 0.05% solns, one in 96% alcohol another in acetone; placing 5 ml of each soln in a test tube and adding to each tube 2 drops of alcoholic ammonia(See Reagent A). In other two tubes the 0.05% solns are mixed with 2 drops of alcoholic KOH soln(See Reagent C). Results of tests are recorded in the table] 5)Marshall **3**(1932), 235-36(Abbreviated version of Desvergnes' table) 6)E.W.

Scott & J.F. Treon, IEC, AnalEd **12**, 189-90 (1940) [Colorimetric detn of primary nitroparaffins(except NMe) consists of adding an excess of dil HCl to an alkaline soln of a nitroparaffin in a small volumetric flask, followed immediately by the addn of 10% ferric chloride soln. When the final pH is betw 1.25 & 1.30, a reddish-brown color develops which rapidly changes to deep red. After standing for 15mins the soln is diluted with w to the mark and compared in a colorimeter with a standard soln of nitroparaffin prepd in the same manner as above] 7)J. Ingham, Lancet **2**, 552(1941) (Modification of Webster's test for colorimetric detn of TNT in urea. Description of this procedure is also given in Ref 9) 8)R.L. Shriner, OSRD **2054**(1943)(Color tests for RDX, HMX & TNT) 9)Clift & Fedoroff, Vol **1**(1942) Chap I,p 14(Prepn of ferrous, sulfate reagent); Volume **3**(1944), Chap IX,p 5(Brown ring test for nitric acid and nitrates); Chap XX,p 1 (Colorimetric detn of TNT in air); pp 1-3 (Analysis of atmospheric samples contg expls, such as MNT, DNT, TNT, Tetryl, NG & PETN or materials used for their manuf, such as DPhA or dimethylamine); p 3(Improved Webster's test); pp 3-4(Snelling's colorimetric test for TNT on the skin); p 4(Neal's test for TNT on the skin); p 5(Pinto & Fahy's colorimetric test for TNT in the air) 10)G. Halfter & H. Winkler, Die Chemie **57**, 124-25(1944)(Colorimetric detn of TNT) 10a)I.Eisdorfer, PicArsn ChemLabRept **107708**(1944)(Identification of HE's) 11)M. Schechter & H.L. Haller, IEC, AnalEd **16**, 325-26(1944)(Colorimetric detn of 2,4-DNAns by shaking its acetonic soln with concd aq NaOH soln. A beautiful violet coloration develops in acetone layer on standing) 12)Ibid, **16**, 326-27(1944)(Detn of 1-Chloro-2,4-dinitrobenzene, as an impurity in 2,4-DNAns, by means of pyridine. It gives violet coloration with ClDNB and yel with DNAns) 13)Snell & Biffen(1944), 720 & 726(Color reagents & Tests) 14)L.R. Shriner, OSRD **5397**(1945) & PBL **30769**(1945)(Eleven color tests are described which permit the identification of 16 HE's. Discussion is also given to the application of these tests in the identification of any of the 8 mixts of HE's. The rept contains a list of reagents and apparatus necessary for these tests) 15)OSRD, "Summary Technical Report of Division 8, NDRC", Vol **1**(1946), 48-9(Summary of work done during WWII on development of color tests for identification of expls. More detailed description is given in OSRD Rept 5397, listed here as Ref 14) 16)T.C.J. Ovenston & C.A. Parker, JSCA **66**, 394-95(1947) (Detn of nitrosamine content in proplnts stabilized with Et Center)17)R. Dalbert, MP **30**, 327-34(1948) [Some colorimetric detns of ingredients of proplnts using Muraour's reagent(See item L) 18)Jabobs(1949), Chap XIII(Colorimetric tests for various org compds which include many expls & their primary materials) 18a)T.C.J. Ovenston, JSCI **68**, 54-9(1949) & Analyst, **74**, 344-51(1949) (Chromatographic analysis of proplnts using various streak reagents) 19)I. Nimeroff & S.W. Wilson, JRNBS **52**, 195-99(1954) & CA **48**, 11061(1954)(A photoelectric colorimeter for measuring the chromaticities of pyrotechnic smokes) 20)Snell & Snell **4**(1954) [Colorimetric tests of various org compds including many expls and their primary materials, such as: Carbazole(pp 314-15); Chloro-2,4-dinitrobenzene (p 17); 2,4-Dinitroanisole(p 19); Dinitrobenzene (p 16); Dinitrocresol(p 19); Dinitronaphthalene (p 19); Dinitrophenol(p 13); 2,4-Dinitroresorcinol (p 13); 2,4-Dinitrotoluene(p 18); Dinitroxylene (p 18); Diphenylamine (p 224); Dipicrylamine (p 25); Erythritol Tetranitrate (p 10); Ethyleneglycoldinitrate(p 7); Glyceryl Nitrates(p 8); Guanidine(p 324); Hexamethylenetetramine (p 56); Hexanitrodiphonylamine(p 25); Hydrazine(p 25); Nitroaminoguanidine(p 10); Nitroaniline(p 201); Nitrobenzenes(pp 12-13); Nitrofluorene(p 16); Nitroguanidine(p 10); Nitronaphthalenes(p 15); Nitronaphthols(p 13); Nitroparaffins(p 1); Nitrophenols(pp 12-13); Nitropropane(p 4); Nitrotoluene(p 14); Nitroxylene(p 9); Nitrosonitropropane(p 13); Nitrosophenol(p 13); Pentaerythritol Tetranitrate(p 10); Picric Acid(p 23); Tetryl(p 24); Trinitrobenzene (p 20); Trinitrotoluene(p 20) and Urea (p 317) 21)C.C. Porter, AnalChem **27**, 805-07(1955) Some mononitrocompds, such as o- & p-Nitroaniline, p-Nitrotoluene and some of their derivs produce orange or red color in dimethylformamide upon the addn of 0.1ml of 10% aqueous soln of tetramethylammonium hydroxide. Janovsky's reagent which is 10% aq NaOH soln may also be used) 22)G.H. Wetter, "Development of Instrumentation for Determining the Total Chromaticity and Chromaticity vs Time of

Pyrotechnic Items," PATR **2164**(1955) 22a) Anon, "Military Explosives", Dept of the Army TM **9-1910**(1955), 267-73(Identification of expls & proplnts) 23)M. Leclercq, MP **37**, 507-11 (1955) [Colorimetric detn of Dinitrochlorobenzene in Dinitromethylaniline(an intermediary in prepn of Tetryl) by means of pyridine. With this substance DNCB gives a red coloration, while DNMeA produces a yel color] 24)P. Tavernier & M. Lamouroux, MP **38**, 65-88(1956) [Colorimetric detns of 26org compds which are considered to be suitable as components of proplnts were made by, using Muraour's Reagent (qv)] 25) "The BDH Book of Organic Reagents", The British Drug Houses Ltd, Poole, England(1958) 26)F. Feigl, "Spot Tests in Organic Analysis", Elsevier, Amsterdam(1960)

In Chapter 4 of Feigl's book are described tests for functional groups, of which the following are of interest in the investigation of explosives, propellants & pyrotechnic compositions:

Alcohols (pp 185-92)
Aldehydes (pp 223-33)
Aliphatic and Aromatic Amines (pp 270-86)
Anilides (pp 301-02)
Azocompounds (pp 179-80)
Azoxycompounds (pp 180-81)
Ethers (pp 186 & 607-08)
Guanidino Compounds (pp 302-04)
Hydrazines (pp 304-10)
Hydroxyaldehydes and Ketones(pp 234-38)
Nitrates, Nitrites and Nitramines are tested by placing ca 0.5ml of diphenylamine reagent(ca 1% soln in concd sulfuric acid) on a spot plate and a grain of sample or 1 drop of its soln(or suspension) is added. A blue ring indicates the presence of one of the above compds. If reagent consists of 0.2% soln of DPhA in 85% phosphoric acid, the nitramines do not react, while nitrates and nitrites give a blue ring(pp 178-79)
Nitrocompounds. The following tests are included a)Test by fusion with tetrabase(tetramethyl -pp diaminodiphenylmethane) $(H_3C)_2N.C_6H_4.CH_2.C_6H_4.N(CH_3)_2$, mp 91° or with diphenylamine, mp 53(p 172) b)Test by reaction with Na pentacyanoammine ferroate, after reducing nitro group to nitroso; purple, blue or green colors are produced(p 173) c)Test for m-dinitrocompdsby reaction with K cyanide. For this a small amt of sample is heated gently in a micro crucible with a drop of 10% KCN soln. A violet or red color is produced with m-dinitrocompds, while with picryl chloride or with PA a red-brown color is obtd(p 174-75) d)Test for aromatic polynitrocompds by saponification to nitrates. For this a small amt of sample is heated to dryness with 1 drop of normal aqueous NaOH or KOH. The cooled residue is treated with 1 drop of 0.5% soln of sulfanilic acid in 1:1 AcOH, followed by 1 drop of 0.3% soln of alpha-naphthylamine in 1:1 AcOH. After saponification the following, among others, give intense red colors: DNB's, DNCB, DNT's, DNPh, DNA, DNBA, DNX, DNCrs, TNB, TNCrs, TNPh, TNT and HNDPhA(pp 176-77) e)Test for primary aliphatic nitrocompds by coupling with the dyestuff, Fast Blue salt B(pp 177-78)
Nitrosocompounds. The tests include: a)Test with phenol & sulfuric acid(Liebermann's Test)(p 165) b)Test with Na pentacyanoammine ferroate, $Na_3[Fe(CN)_5NH_3]$(pp 166-67) c)Test for N-nitroso derivs by hydrolytic splitting to nitrous acid, followed by testing for the acid with Griess' reagent(qv) (pp 167-69) d)Test for aliphatic nitrosamines by dinitrosation(p 169) and e)Test with N,N'-diphenylbenzidine (p 599)

Feigl also describes tests for individual organic compds, of which the following are of interest in the expls industry(Chapter 5):

Acetaldehyde(pp 352-53)
Acetic Acid(pp 369-71)
Acetone(p 421)
Benzoyl Peroxide (pp 485-86 & 606)
Carbohydrates(pp 424-26)
Carbon Tetrachloride(pp 606-07)
Chloroform(pp 353-54 & 603)
Dinitrobenzenes(pp 364-65)
Ethanol(pp 358-59)
Formaldehyde(pp 349-52)
Furfural(p 474)
Glycerol(pp 422-24)
Glyoxal(Aminophenol)(p 468)
Guanidine(p 454)
Hexamethyleneteramine(pp 356-57 & 604)
Hexoses(pp 427-28)
Hydroquinone(pp 418-19)
Inositol(pp 428-29)
Isopropanol(pp 421-22)
Methanol(pp 357-58)
Naphthalene(pp 342-43)

Nitranilines(pp 362-64)
o-or p-Nitro(Nitroso) *aniline* (pp 484-85)
Nitroethane(p 362)
Nitromethane(pp 360-61)
o- and p-Nitrophenol (p 412)
Nitrosodiphenylamines(pp 481-82)
Phenylhydrazine(pp 438-39)
Phloroglucinol(p 420)
Pyrocatechol(pp 416-17)
Resorcinol(pp 417-18)
Urea(pp 450-52)

The following expl compds or mixts are also among the color tests listed by Feigl:
Tetryl or Picrylnitromethylamine(pp 488-89) can be detected by treating(in a micro test tube) a drop of benzenic soln of the sample with powdered manganous sulfate filling the tube to 2/3 of its capacity. After evapg benzene, the tube is immersed (after covering its mouth with a disk of filter paper moistened with Nessler soln) into a bath preheated to 140° and the temp is raised to 150°. A gray or black stain on the paper appearing within 5 mins indicates the presence of a nitramine, such as tetryl. The filter paper should be ammonia free, such as qualitative papers
Cyclonite or RDX is called *Hexahydro-1,3,5-trinitro-s-triazine* by Feigl(p 489). The test is described in this volume under Cyclotrimethylenetrimitrosamine, Analtyical Procedures
Black Powder and Some Other Explosives. A brief outline of tests is given on pp 549-50 27)StdMethodsChemAnalysis, Vol **2B**(1963), pp 1347-49 (Same colorimetric procedures as given in Ref 22a)

Colt, Samuel(1814-1862). An American gunsmith, who invented in 1835 a revolver which served as a prototype of modern revolvers. It was adopted by the US Army after the Mexican War (1846-1848)
Refs: 1)Chamber's Biographical Dictionary", Macmillan, NY(1957), 235 2)W.H.B. & J.E. Smith, "Small Arms of the World", Stackpole, Harrisburg, Pa(1960), 173 3)EncyclBritannica **6**(1963), 107

Colt's Pistol Powder. A propInt prepd by mixing Guncotton 95& DNB 5%, passing the blend between heated rollers, then cutting the sheets into squares, 0.04"x0.04", which are coated with graphite
Ref: Colver(1918), 143-44

Colt's Revolvers. A series of successful revolvers invented by Samuel Colt, beginning from 1835. A brief description and illustrations of these revolvers, as well as system Smith & Wesson, are given in Ref
Ref: W.H.B. & J.E. Smith "Small Arms of the World", Stackpole, Harrisburg, Pa(1960), 174-88

Columbia Powder. An explosive patented in 1891 in France. It was prepd by intimately mixing K chlorate with sulfur and coating the resulting blend with paraffin. Eg: $KClO_3$ 82.6, S 8.7 & paraffin 8.7%. In some mixts part of K chlorate was replaced with a nitrate
Refs: 1)Daniel(1902), 151 2)Giua, Trattato, **6**(1)(1959), 395

Columbium Compounds for Smoke Producing Pyrotechnic Items were proposed by J. DeMent, USP 2995526(1961), pp 8-9

Combat Masterpiece. A Smith & Wesson's Cal .38, Special Revolver, used by the US Marine Corps in Korea
Ref: W.H.B. & J.E. Smith, "Small Arms of the World", Stackpole, Harrisburg, Pa (1960), p 185

Combination Fuze. See under FUZES

Combined Guidance Systems for Missiles are described by A.S. Locke et al in "Guidance", VanNostrand, NY(1955), 605-07

Comblain Rifle. A caliber 11mm, single shot, rifle designed by Belgian gunsmith, Comblain. It was used in various parts of South America, notably in Chile. In Belgium it was used by the Civil Guards as late as WWII
Ref: W.H.B.Smith, "Rifles", Vol 2 of the "NRA Book of Small Arms", Military Service PubgCo., Harrisburg, Pa(1949), p 123

Comburable or Combustible. Fr for Combustible

Comburant. Fr for a substance supporting combustion; an oxidizing agent

Combustible and Consumable Cartridge Cases and Other Items of Ammunition. Accdg to US

Ordnance practices, the term *combustible cartridge case* is referred to cases fabricated from fibrous NC and a binder. Such cases produce no residue when fired in a conventional artillery weapon system. The term *consumable cartridge case* has been reserved for cases prepd from combustible materials other than NC, such as woodpulp or paper impregnated with a phenolformaldehyde resin. In each type of case, fungicides, flash reducing agents and gun erosion inhibitors can be incorporated

The work on development of combustible cartridge cases began during WWII in Germany by Drs H. Walter & H. Leunig of Dynamit AG and by the firm of Dietsch & Co at Geyer & Hronov(Ref 2)

Accdg to Leunig, as quoted in Ref 2, p 2, the first Ger combustible cartridge was made of double-base DEGDN proplnt. As this material proved to be unstable at high temps, it was decided to use sheets of NC obtained by treating sulfate pulp sheets, ca 1mm thick, with 98% nitric acid for 30mins. The resulting material was hard and compact and was strong enough to be used without gelatinization. After washing the sheets with water and drying, three sheets were bonded together by means of cold hardening Araldite lacquer(made by CIBA). After drying the bonded sheets, discs of the same diameter as the base of the cartridges were punched

Other NC sheets were cut into strips corresponding to the height of cartridges and wrapped around a pin, which had a diameter 2 to 2.5mm smaller that the inside diam of the std metal cartridge case. Then the surface of the resulting tube was covered with Araldite lacquer and the 2nd layer of NC sheet was wrapped. This was followed by the 3rd layer, all layers being thoroughly bonded together. After the adhesive had hardened, the above disc was attached to one end of the tube by means of the same adhesive. After complete hardening, the finished cartridge case was pulledoff the pin and a primer was installed in the base of the cartridge case.

Accdg to Dr Walter, the above method of manuf is not very safe because handling of dry NC is rather dangerous. He proposed a safer process which consisted of preparing the cartridge case from a sheet of sulfite pulp and then nitrating it in 80/20 mixt (by volume) of concd nitric acid & glacial AcOH. None of the joints was bonded together but simply compressed tightly over each other before nitration. Bonding took place by itself during the process of nitration which was followed by washing in water. After drying, the case was made waterproof by dipping it into a 7% soln of benzyl cellulose mixed with 9 parts of methylene chloride and 1 part of ethanol. After removing the solvent by evaporation, the case was ready for use(Ref 2,pp 3-4)

In the process of Dietzsch & Co, a continuous roll of pulp, in the form of a sheet 45cm wide, was passed thru a chamber, countercurrently to the nitric acid fumes and then thru a bath contg concd nitric acid. After washing the resulting NC with water and air-drying, the sheet was coated on one side with an adhesive(such as Vinarol) and made into a cartridge case. It was waterproofed by coating it with a bituminous lacquer mixed with an acetonic soln of NC(Ref 2,pp 7-11)

It seems that no work on combustible and consumable cartridge cases was done in US until after the Korean War. Gen Ghormley states in his paper published in 1962(Ref 15,p 233), that about 10 years ago (i.e. 1952), the Army Ordnance Corps began to support a research program on combustible cartridge cases, which was carried out at Picatinny Arsenal, Dover, N.J.; Ballistics Research Laboratories at Aberdeen Proving Ground, Maryland; and Armour Research Foundation, Chicago, Illinois

Most of the work done in US on combustible and consumable cartridge cases has been published in classified reports(See Refs, 1,3,4, 6,7,8,9,10,11,14,16,17,18,21,22 & 26) and cannot be used here as sources of information. There are, however, some unclassified papers and reports(See Refs 5,12,13,15,19,20,23,24 & 25), which give some important info on the present status of research and development of combustible and consumable cartridge cases. Especially valuable info is contained in Refs 20,23 & 24

Axelrod & Mirko (Ref 20) describe a method of prepg cast combustible cartridge cases for artillery weapons. The method consists of thoroughly blending small grains of a single-base artillery proplnt (such as M10, which is described in Vol 2, p C35, Table VII of this

Encycl) with a binder (such as epoxy-polyamide, expoxy-anhydride, and polyurethane resins) and loading the resulting mixture into the annulus between the inner and outer parts of a cylindrical mold. After curing for 24hr, this cylindrical part of cartridge was attached to a noncombustible base(metallic or plastic), which served as an obturator

Testing of these cases, in the laboratory and in gun firings, showed that these cartridges were fairly stable in storage and their ballistic properties were not seriously affected by elevated temps. Compressive strength results at ambient temp were inconclusive, but at extreme temps (160°F & -65°F) and after temp and humidity cycling the epoxy--anhydride binder was least affected

The work on cast combustible cases was discontinued(probably in 1962), because the Combustible Cartridge Case Steering Committee decided to concentrate all efforts on the felted NC type of cases(Ref 20, p4)

The following description of the fabrication of cases by felting, which was developed at Armour Research Foundation is given in Ref 23,pp 6-15

Because closer tolerances were reqd the case walls were much smaller than those produced previously, the conventional female bag molding technique could not be applied to the fabrication of felt body cases. It became necessary to design new molds and to modify the fabrication technique

Thus a new felting die assembly was required. The successful procedure involved a die formed from brass 0.0625" thick, perforated with 1/16" holes in a hexagonal pattern. This die was covered with a small "sock" made of 44 x 44 mesh Saran screen, 0.010" thick. The sock has been sewn in the desired form with very fine cotton thread. The preform was felted onto this sock, which was then slipped from the die, collapsed & withdrawn from the interior of the preform

After the preform had been felted from a water slurry, it was compacted at atmospheric pressure, removed from the die & dried at 140°F in a steam-heated, circulating air oven. The dried preform was then soaked in the impregnating resin soln, drained, and finally flash-dried in a vacuum oven to a point where it could be post formed

Post forming consisted of placing the still plastic, impregnated preform over a close -fitting, highly polished metal plug and forcing the case into a female mold having the desired final dimensions. Because some difficulty was experienced in removing the case both from the mold & the metal plug, the polished metal plug was replaced by an elastometric male plug which had been cast from a plastisol of tricresylphosphate & Bakelite Corp QYNV vinyl chloride resin. Also the polished metal cavity mold was replaced by a split-cavity mold made from an Al-filled epoxy resin which had been cast on a polished metal mandrel. Finally, the molded case wall was trimmed to the proper length on a lathe and bonded to the obturating base with epoxy -polyamide cement(Ref 23,pp 6-15)

The formulations for felted combustible cartridge cases, developed by Armor Research Foundation and described in Ref 23,p 20, contained some fungicides, as for example copper pentachlorophenate. One of the compns contg this fungicide consisted of Kraft fiber 40.8, NC 40.8, resin + DPhA 17.4 & Cu pentachlorophenate, 2%

Axelrod(Ref 24) described two formulations of combustible cartridge cases prepd from felted NC combined with other ingredients. These cases were developed by the Armor Research Foundation, Chicago, Illinois. The 1st type, designated as XM-156 and intended for use in M68 105 mm Gun consisted of NC (12.3%N) 38, Northern Kraft fiber (bleached) 38 & Polyvinyl Formal, Grade 7/955(of Shawinigan Resins Corp) 24%, with 1% DPhA added. The 2nd type, designated as XM-157 for use in XM81 152mm Gun consisted of NC (12.3%N)65, Northern Kraft fiber(bleached) 15 & Polyvinyl Formal(same as above) 19%, with 1% DPhA added

It has been known for sometime(See Ref 1a) that NG will migrate from proplnts (double- or triple-base) into the combustible case and be absorbed by the NC, thus gelatinizing it. As this migration affects the physical props of the cartridge case, it should be prevented as much as possible. One of the methods to stop such migration is to coat the inside of cartridges with a barrier material. Axelrod found that a mixture of Methylcellulose (MC Std 15cps) 5.0,

water 94.8,Uranine (dye 0.1 & Aerosol OT 0.1% with 0.01% of Silicone Anti-Foam B added, proved to be an effective barrier material (Ref 24,pp 4 & 7)

The advantages of combustible cartridge cases over metal cases were discussed by Gen Ghormley (Ref 15). When made entirely (cylindrical part and base) from a combustible material, they are consumed completely in a gun chamber during firing. Such cartridges,however,do not perform the function of sealing off the rear end of the gun to prevent leakage of the high-pressure gas, as do all metal cases. This means that a completely combustible cartridge should be used only in conjunction with a breechblock which is designed to seal the rear of the gun chamber against the back pressure of the propellant gases

As the breech mechanisms of present guns are not designed to achieve complete sealing off the gases of combustion(complete obturation), the base of combustible cartridges should be made of a non-combustible material. This material can be either metal(brass or steel) or a noncombustible plastic. Obturators prepd from polyethylene are described in Ref 13. Cartridges prepd by these methods are known as *partially combustible*(Ref 15)

Because the manuf of combustible cartridges requires the construction of a NC plant and because handling of dry NC is rather dangerous, the Army Ordnance Corps contracted Armour Research Foundation and some other organizations to develop an entirely combustible cartridge prepd from materials not contg NC. Such cartridges are known as *consumable*. The work on their development at NOL was reported in Refs 5 & 13, but the most important work was done by the Armour Research Foundation(Ref 23). Evaluation of consumable cartridges for use in 105mm T254E2 Gun was reported in Ref 16

The consumable cartridge cases developed by Armour Research Foundation consist of Kraft fiber & resin binder with a small amt of a fungicide and a substance such as NGu or dicyanamidazide, which proved to be effective in increasing the consumability of cartridge cases at lower pressures

Mixing of components and shaping of cartridge by the felting technique were conducted in a manner similar to that of combustible cartridge case. As a binder, the Formvar resin, dissolved in alcohol, was selected, and as a fungicide Cu pentachlorophenate was used. When NGu was used as one of the components of consumable cartridges, it was recrystallized from a satd aqueous soln into the pores of Kraft fiber prior to resin impregnation. When dicyanamidazide was employed, it was added as a component of the impregnating resin-solvent(Ref 23,pp III-V, 23, 25 & 27)

The same type of obturation as used for combustible cartridges is suitable for consumable cartridges

Analytical procedures for combustible cartridge cases were developed by Armour Research Foundation(Ref 23,pp 34-6)

The procedure used until recently at PicArsn for the manuf of combustible cartridge cases was conducted, essentially, in three operations: a)Felting of NC & its derivatives on a special die to conform to the final article and drying it b)Dipping the felted article into toluene-alcohol soln of a suitable adhesive and c)Squeezing the excess lacquer & drying

In the new technique developed at PicArsn by R.P.Kiley, C. Stephens & J.H. Veltman under the direction of Dr. J.P. Picard, the entire process is conducted in a water slurry which renders the operation less costly and dangerous than the older method. The manuf of combustible cartridges by this process is practically a one-step operation(Ref 27). Detailed description of new method is given in Refs 28 & 29

Besides the combustible & consumable cartridge cases, other items of ammunition made of combustible or consumable materials have been developed. For example, a combustible igniter tube, developed at PicArsn, is discussed in Ref 19 and development of combustible primers is described in Refs 7 & 9. The last two refs are conf and were not used as sources of info (See also Cartridge, Combustible, in Vol 2,p C80-L)

This section on combustible and consumable cartridges was reviewed by R.G. Wetton, W.O. Seals & J. Veltman of PicArsn

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Combustibles. Substances which are capable of combining with oxygen, easily kindled, and burn with the evolution of large vols of gas and much heat are called combustibles. Some examples include carbon, starch, flour, sawdust, cellulose, woodmeal & finely divided metals. Many of these materials are combined with oxidizing agents & other additives to form expl mixts

The relative combustibility of materials in

storage is defined as follows(Ref 8):
Hazardous. Materials which, either by themselves or in combination with their packaging, are highly susceptible to ignition and will contribute to the intensity & rapid spread of fire
Moderate. Materials & their packaging, both of which will contribute fuel to fire
Low. Materials which, in themselves, will not normally ignite, but which in combination with their packaging will contribute fuel to fire
Noncombustible. Materials & their packaging which will neither ignite nor support combustion

All combustible materials are dangerous in a fire and/or expln, both because heat will cause them to catch fire and add to the smoke & fume hazard, and because small explosions are known to scatter clouds of flammable dusts which in turn cause still greater explns than the original ones(Ref 3)

See the Refs for additional info

Refs: 1)Kirk & Othmer **6**(1951), 903 2)A.S. Radford, Paint Technol **20**, 317-23(1956) & CA **50**, 17455(1956)(Spontaneous combustion of linseed-oil soaked materials & its early detection) 3)Sax(1957), Sect 6, 129-45 (Industrial fire protection) & Sect 7, 147-70 (Storage & handling of hazardous materials) 4)Anon, "Combustible Solids, Dusts, Chemicals and Explosives", National Fire Codes, Vol 2, 624pp, Publications Dept, National Fire Protection Assoc, Boston, Mass(1957) 5)L. Andrussov, Explosivst **1958**, 285-89 (Properties, uses & dangers of absolute nitric acid systems) 6)H.F. Sloan & G.K. Greminger, USP 2838384(1958)(Combustible gel) 7)Anon, Revue Militaire Générale (General Military Review), Éditions Berger -Levrault, Paris(1962), Chapter on "Des Combustibles Nouveaux", (New Combustibles), pp 639-47 8)Anon, "Dictionary of United States Army Terms", Headquarters Dept of the Army, Washington 25, DC, Army Regulations AR 320-5(1963), 97

Combustion. See Burning and Combustion in Vol 2,p B343-L of this Encycl and the following refs on combustion:

Refs: 1)W.Jost, "Explosion and Combustion Processes in Gases", McGraw-Hill, N Y(1946) 2)B.Lewis & G.von Elbe, IEC **40**, 1590-96 (1948)(Combustion) and in September issues of succeeding years 3)Third Symposium on Combustion, Flame and Explosion Phenomena, held at Univ of Wisconsin in 1948, published by Williams & Wilkins, Baltimore(1949), pp 536-94 4)H.C. Hottel, E.C. Williams & C.N. Satterfeld, "Thermodynamic Charts for Combustion Processes", Wiley, N.Y.(1949) 5)B.Lewis & G.von Elbe, "Combustion, Flame and Explosion of Gases", Academic Press, NY(1951) 6)Kirk & Othmer **6**(1951), 913-35(Combustion of fuels) 7)Fourth Symposium(International) on Combustion, held at Massachusetts Institute of Technology in 1952 and published by Williams & Wilkins, Baltimore(1953) 8)B.Lewis, "Remarks on the Combustion Waves", Selected Combustion Problems, NATO, AGARD," Combustion Colloquium", Cambridge Univ, Dec 1953, Butterworths, London(1954) 9)S.Haffner, "A Survey of Available Literature on the Rapid Combustion of Metals in Air", PATR **2061**(1954) 10)J.F. Roth, Nobelhefte **1954**, pp 29ff Abstracted in Explosivst **1956**(Combustion, initiation, detonation and explosion; definition of terms) 11)Fifth Symposium(International) on Combustion, held at Univ of Pittsburgh in 1954; published by Reinhold, NY(1955) 12)R. Morrison, "Shock-Tube Investigation of Detonative Combustion", Univ of Michigan Press, Oxford (1955) 13)The Combustion Institute, "Combustion in Engines and Combustion Kinetics", Reinhold, NY(1955) 14)D. Spalding, "Some Fundamentals of Combustion", Academic Press NY(1955) 15)B.P. Mullins, "Combustion Researches and Reviews", Interscience, NY (1955) 16)W.R. Hawthorne, D.P. Spalding & J. Fabri, "Selected Combustion Problems", Interscience, NY(1955) 17)B.Lewis, R.N. Pease & H.S. Taylor, Editors, "Combustion Researches", in Vol 2 of "High Speed Aerodynamics and Jet Propulsion", Princeton Univ Press, Princeton, N.J.(1956) 18)The Institution of Mechanical Engineers(London) & The American Society of Mechanical Engineers (New York) "Proceedings of the Joint Conference on Combustion", held in Boston & Londonin 1955; published by the IME, London (1956) 19)Sixth Symposium(International) on Combustion, held at Yale Univ in 1956, published by Reinhold, NY(1957) 20)NATO, AGARD, "Combustion Processes and Reviews," Pergamon Press,NY(1957) 21)NATO, AGARD

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Combustion of Pyrotechnic Compositions. See Burning and Burning Characteristics of Pyrotechnic Compositions in Vol 2, p B355-R

Combustion Rate(or Burning Rate) Catalysts. Accdg to Mr. R. Baumann of PicArsn, these compds are not catalysts, but rather "modifiers", or "additives". They are incorporated into solid proplnts in order to modify or affect their combustion characteristics. The compds which affect burning rate include Prussian blues, Amm dichromate, trichromate or tetrachromate, etc). Some compds may be employed to reduce the variability of burning rate with combustion pressure and temperature (Eg: Pb stearate or salicylate). Other compds may be used to reduce the tendency toward oscillatory combustion(Eg: Al powder in small quantity), or to affect flash characteristics of exhaust gases [Eg: K_2SO_4, $Ba(NO_3)_2$, KNO_3, etc)(Refs 1-12)

In some solid rocket proplnts, the effectiveness of a catalyst is reinforced by employing so-called synergist catalysts. These may include phenothiazine and other substances listed under Refs 9 & 12

Refs: 1)J. Linsk & R.W. Todd, USP 2936225 (1960) & CA **54**, 16834(1960)(Combustion catalysts: Prussian blue, Amm dichromate, fine carbon, MgO or sulfurized substituted diphenylene blue dye with color index of 956, for use in gas-producting materials suitable for use as rocket fuels) 2)W. Proell et al, USP 2938780(1960) & CA **54**, 17851(1960) (Na barbiturate and C black as combustion catalysts for gas-producing compns suitable for auxiliary turbojet starters) 3)W.M. St. John, Jr, USP 2941352(1960) & CA **54**, 21764(1960) (Comb catalysts for composite proplnts include Fe cyanide complexes like Prussian blue, Amm or K dichromates, Fe or Pb oxides, etc) 4)E.F. Morello, USP 2942962(1960) & CA **54**, 25830-31(1960)(Composite AN rocket proplnts contg Prussian blue as combustion catalyst & some DPhA which is incorporated in order to render the proplnt less susceptible to gassing at elevated tepms) 5)W.A. Proell & W.G. Stanley, BritP 834660(1960) & CA **55**, 2107 (1961); USP 2955033(1960) & CA **55**, 3982 (1961)(Cyanide complexes, such as Prussian blue as combustion catalysts for composite rocket proplnts) 6)E.D. Guth, USP 2963356 (1960)CA **55**,5958(1961)(Combustion catalysts for AN composite proplnts are materials which do not combine with AN at below 60° and combine with NO_2 at initial temps below 406°. They include $NaBH_4$, Ti, Ce, Zr, ZrC & ZrH and may be added in amts of 0.25-5 parts) 7)G.D. Sammons, USP 2969638(1961) & CA **55**, 11850(1961) [Combustion catalyst for AN comp proplnts comprises a mixt of 3-10 parts (based on the total catalyst) of Amm dichromate (preferably below 15 micron), 0.5-7 ps of a Cu powder or its compds (CuO, Cu_2O or Cu chromate)(preferably below 15 micron), and 2-20 ps of any org or inorg salt of picric or styphnic acid] 8)J. Linsk, USP 2973256 (1961) & CA **55**, 12856(1961)(Catalysts for AN comp proplnts can be Prussian blues, Amm or Na chromate or dichromate & mono-Na -barbiturate, added in amt 2-6% of total proplnt) 9)W.A. Proell & W.G. Stanley, USP 2987389 (1961) & CA **55**, 24016(1961) [Cyanide complexes of Fe''' - Fe'', alkali metal-Fe''' -Fe'' or NH_4-Fe'''-Fe'' as combustion catalysts in comp AN proplnts such as: AN 75, catalyst 6 &cellulose acetate -glycoldiglycolate mixt 19%. The proplnt becomes even more effective when 0.5-5.0% of a synergist catalyst is added. The synergists include phenothiazine,linseed oil & sucrose or Mg stearate(or ricinoleate, or oleate) in the presence of hydrocarbons] 10)R.W. Lawrence, USP 2978305(1961) & CA **55**, 25260(1961) (Combustion catalysts for comp proplnts include Amm trichromate or tetrachromate or their mixts, added in quantity 0.1-4% of total proplnt) 11)W.G. Proell, USP 2987388(1961) & CA **55**, 25259(1961)(Combustion catalysts for AN comp proplnts include Prussian blues, chromates & Na barbiturate in quantities of 2-6% of total proplnt) 12)W.G. Stanley & W.G. Proell, USP 2988437(1961) & CA **55**, 25258 (1961)(Combustion catalysts for AN comp proplnts include stable metallic cyanamides of Ba, Cu, Pb, Hg or Ag or their mixts in amts 2-4% of total proplnt)

Combustion, Spontaneous or Self-Ignition. When piles of materials, such as oily rags, soft coal in small size, rotten wood, etc are stored improperly, a gradual slow oxidation

with evolution of heat might take place. Because of the poor thermal conductivity of these materials, the heat does not dissipate but gradually accumulates. During this process some air seeps thru the crevices and when the temp inside the pile reaches the ignition point, the mass spontaneously ignites (Ref 3)

The same phenomenon happens to expls or proplnts if they are not properly stabilized. For example, the disastrous explns with loss of many lives, which took place in 1907 aboard the French battleship Jena and in 1911 aboard battleship Liberté, were due to self-ignition of smokeless proplnts stored inside the hold. These proplnts were stabilized with amyl alcohol. Investigation conducted by M. Marqueyrol has shown that all Fr proplnts stabilized with amyl alcohol have the tendency to decompose on prolonged storage with evolution of nitrous fumes, accompanied by a gradual rise in temp. As soon as the temp reached the ignition point of proplnt, it spontaneously ignited and then exploded. As a result of this investigation, amyl alcohol was replaced by DPhA, which proved to be a much better stabilizer(Ref 1)

Spontaneous combustion of gases is discussed in Ref 2

Refs: 1)Davis(1943), 308 2)A.S. Sokolik, "Samovosplameneniye, Plamia i Detonatsiya v Gazakh", IzdatAkadNauk, Moskva(1960); Engl translation by A.Kaner under the title: "Self-Ignition, Flame and Detonation in Gases", published for NASA and NSF in Jerusalem (1963); available thru OTS, Washington 25, DC 3)EncyclBritannica 6(1963), 127

Combustion Theories as Applied to Solid Propellants. Solid proplnts can be divided into two major categories: *homogeneous* NC proplnts & *heterogeneous* composite proplnts. The former class is used primarily in gun & artillery propulsion, and in some rocket applications; the latter is used in rocketry

Although it was possible, at one time, to categorize most proplnts into the above classes, the demarcation line between them is becoming more obscure; rocket & gun proplnt manufacturers use the same ingredients in both classes

Several excellent review articles on the combustion of solid proplnts have appeared in the literature such as by Geckler(Ref 8), Huggett(Ref 9), and the Symposium on Kinetics of Combustion(Ref 7). In addn to these reviews, one can keep abreast of the newer developments by referring to the journals of "Combustion and Flame" and the Amer Rocket SocJ & Journal of the Aerospace Sciences combined into "AIAA Journal". Another source of current info is the published symposia sponsored by the Combustion Institute, Pittsburgh, Pa

Combustion mechanism as described here is limited to proposed theories relating to burning rates. No effort is made to describe the complete kinetics in the combustion process, the detailed structure of the flame, or the flame species

Before attempting to sketch some of the burning rate theories, it should be noted that this complicated process is not only dependent on numerous intrinsic props of the system but also on the method used to determine burning rate. The burning rate value detd in a "strand burner" is seldom equiv to that obtd in a "vented vessel". It is also diffc to compare burning rates detd from "closed bomb" firings with those obtd by other methods. This difficulty is attributed not only to such factors as erosion & radiation but also to environmental conditions. It has been found recently by Lenchitz(Ref 12) that there are differences in burning rates of proplnts even in the same system(strand burner). At constant press & temp (where the temp of proplnt was monitored during burning, it was found that a given sample of ½" diam burned more than 13% quicker than one of ¼" diam(at 1200 psi & ambient temp). Any burning rate theory will, therefore, be limited by exptl unknowns and can be only approx

Although it is diffc to derive a perfect burning rate equation, the effect of individual parameters has been related successfully to burning rate. The best example of this, and perhaps the most important is the relationship between burning rate & pressure. The linear burning rate of a proplnt is in general strongly pressure dependent. This statement must be qualified, however, because proplnts can be made which have burning rates independent of pressure, over a limited

range. Most of the literature on such proplnts is classified, and a conf review of this subject has been made by McEvan(Ref 11)

Vieille has found that the relationship between burning rate & pressure for a given propellant can be expressed by:

$r=bp^n$ (Vieille's Law)

where r is linear burning rate, P is pressure and b & n are constants; n is often called the pressure exponent. Similar individual relationships for temp, flame radiation, and erosion can also be derived for any given system. It is evident, therefore, that burning rate is contingent on the afore-mentioned factors as well as the chem compn of the proplnt

Theories relating the combustion of NC based proplnts to pressure are fairly well developed, the most successful being those proposed by Daniels(Ref 1), Rice (Ref 2), Crawford(Ref 3), Boys & Corner(Ref 5), and others(Ref 4). The classification of these theories is contingent on the proposed position of the rate-determining step in the surface-flame structure

Surface Theories. A review of the earlier theories on combustion is presented by Corner(Ref 6). Theories in which the rate -determining step occurs at the surface of a burning proplnt were proposed by Muraour (cited in Ref 6) and Daniels(Ref 1 and cited in Refs 7 & 9). The theory proposed by Muraour is based on the burning rate law:

$r= a+ bP$

which equation is derived from observed rate measurements. The theory of Daniels is based on the first-order decompn of NC where the energy of activation(E_a) is 46.7 kcal/mole and the frequency factor(A) is 3×10^{13} sec^{-1}. Thus the burning rate (r) is expressed by:

$$r=\frac{nAe^{-E_a/RT_s}}{m}$$

where n is the number of nitrate groups /sq cm of reacting surface, m is the number of nitrate groups/cubic cm, T_s is the temp at the surface, and R is the gas constant. Using this equation a surface temp of 1000° is reqd. However, this value is much too high when one considers that the ignition temp of a double-base proplnt is 200°. Observations of Crawford et al(Ref 1a) also confirm a lower surface temp for NC proplnts

It should be noted that the equation proposed by Daniels reasonably approximates the burning rate of NC proplnts. But any theory based entirely on surface decompn is,however, incomplete because it fails to consider the overall surface structure with the accompanying heat flow relationships

Law of Steady Heat Flow. A law which is basic to any proposed theory on proplnt burning is the "Law of Steady Heat Flow" proposed by Boys & Corner(Ref 5). This law states that the amt of heat crossing any plane of the combustion zone per unit of time is constant:

$$MH - \frac{\lambda dT}{dx} = MH_o = MH'_s = MH_1' = MH_3$$

where M = mass burning rate, H = heat content, λ = thermal conductivity, T= temperature, x = distance from flame, and the subscripts refer to different sections of the propellant-surface-flame structure. This law is written to conform with the surface to flame structure(shown later), proposed by Rice & Ginell(Ref 7,pp 885-917), and based on exptl work of Crawford et al (Ref 1a). The equation shows that the energy flow is composed of the intrinsic energy per unit mass transported by mass flow(MH) and the flow of heat by thermal conduction ($\lambda dT/dx$). At large distances from the reaction zone $-\infty$ and $+\infty$(See sketch later), the flow of heat due to conduction is zero

Gas Theory. Boys & Corner(Ref 5) propose a theory where the flame reaction determines the rate of burning of a proplnt. Their model is based on the propagation of a plane reaction zone thru the combustible gases, but does not include the surface of the burning proplnt, the rate-determining step being in the flame. The order of the reaction determines how the rate of burning depends on the pressure. For a first-order reaction, $r \propto p^{1/2}$; and for a second-order reaction $r \propto P$

The theory of Boys & Corner gives an adequate picture of the burning rate of a propellant at high pressures(in guns) but

is not applicable to burning at low pressures (in rockets). These investigators combine the energy flux equation:

$$\frac{\lambda}{M}\frac{dT}{dx} = H-H_3 = (1-\epsilon)\,Q + (T-T_3)\,Cp$$

with the rate equation:

$$\frac{d\epsilon}{dt} = f(\epsilon, V, T)$$

where ϵ = fraction that the reaction has progressed toward completion, Q = heat liberated, Cp = av specific heat of the gases, and V = volume of gas. The rate equation used is contingent on the assumed order of reaction. The final burning rate equation, assuming a second-order rate from a bimolecular reaction is:

$$M^2 = \frac{\lambda A (PW)^2 \psi e^{-E_a/RT_3}}{Cp(RT_3)^2(1+n)^2}$$

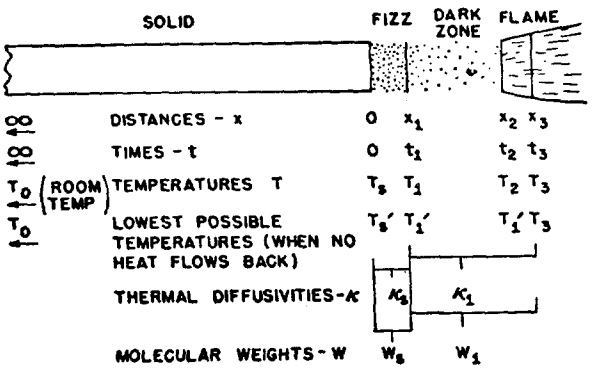
where M = burning rate, λ = thermal conductivity, A = reaction rate constant, P = pressure, W = molecular wt of gases, ψ = a constant depending on the compn of the proplnt, e = natural logarithm, E_a = activation energy, R = gas constant, T_3 = flame temp, Cp = av specific heat of the gas, and n = moles of gas. The solution of this equation is obtd by successive approximations. Addnl details are given in Refs 5 & 6

Combination Theory of Rice & Ginell and Parr & Crawford. This theory combines the surface theory of Daniels and the gas phase theory of Boys & Corner. The essential feature of the theory is the determination of the surface temp (T_s) of a burning proplnt and its dependency on the gas phase reactions. A model which shows the proplnt surface -flame-structure relationship and summarizes the burning process is sketched in the following Fig

Starting with the law of steady heat flow and following a small portion of the gas moving outward into higher temp regions, Rice & Ginell(Ref 7,pp 885-917) obtain an equation for the rate of increase in temp with time:

$$\frac{dT}{dt} = \frac{dT}{dx}\cdot\frac{dx}{dt} = \frac{M^2(T-T_s)}{\rho^2\,\eta}$$

where $\eta = \lambda / Cp\,\rho$(thermal diffusivity)



Schematic Sketch of a Stick of Burning Propellant

and ρ = density of gas. Setting $\rho = PW/RT$ and integrating the above equation from t=o and $T = T_s$ at x=o to $t=t_1$ and $T = T_1$ at $x=x_1$, the authors obtain an expression for the relationship between the pressure, burning rate & temp at different planes of the flame at time (t). For M, the Arrhenius relationship

$$M = Ae^{-E_a/RT_s}$$

is substitured, and for time (t) is substituted

$$t = 1/kP^{n-1}$$

where n = the order of the reaction and k = the reaction rate constant. A detailed analysis of the solution is given in Refs 4,6,7,8 & 9. The final equation includes the effect of diffusion, and relates the effect of pressure & temp on the burning rate

Composite Propellents. The theory of Rice (Ref 2a), of Rice & Ginell(Ref 7,pp 885-917) and of Parr & Crawford(Ref 7,pp 929-54) can also be applied to rocket proplnts. The review by Geckler(Ref 8) includes an appraisal of composite proplnts. A significant development of the burning mechanism of rocket proplnts is reported in the work at Princeton Univ by Professor Summerfield et al(Ref 10) Using an Amm perchlorate proplnt, these investigators set up a model of the phys & chem structure of the flame zone, based on exptl determinations. They also combine the equations of steady heat flow & the Arrhenius relationship to obtain a final equation which relates burning rate (r) to pressure:

$$\frac{1}{r} = \frac{a}{P} + \frac{b}{P^{1/3}}$$

where a & b are constants, and P = pressure. The agreement with the exptl measurements is excellent. Summerfield concludes that the reaction zone is almost entirely in the gas phase and that diffustional mixing occurs simultaneously with chem reaction

This article cannot do justice to the great quantity of work being done in the field on combustion theories. Much work is being done, for example, in the field of resonance burning. If a serious investigation of the field is intended, the reader is referred to the Refs listed and to the current literature

See also Burning and Burning Characteristics of Propellants, Vol 2,pp B 346-L to B 355-R

(This section was written by Mr. C. Lenchitz, Chief of the Ballistics & Combustion Res Branch, Picatinny Arsenal, Dover, N.J.)

Refs: 1)F. Daniels, "The Mechanism of Powder Burning", OSRD Rept **3206**(Jan 1944) 1a)B.L. Crawford et al, "Observations on the Burning of Double-Base Propellants", OSRD **3544**(April 1944) 2)O.K.Rice, "The Theory of Burning of Double-Base Rocket Powders", OSRD Rept **5224**(June 1945) 2a)O.K. Rice, "The Theory of the Burning of Rocket Powders", OSRD Rept **5574**(Nov 1945) 3)B.L. Crawford et al, "Studies on Propellants", OSRD Rept **6374**(1945) 4)Summary Tech Rept of Div 8, NDRC, Vol **1**(1946), 90-95 5)S.F. Boys & J. Corner, PrRoySoc **197A**, 90-106(1949) 6)Corner, Ballistics(1950), 42ff 7)Symposium on Kinetics of Propellants, JPhysChem **54**, 847-954(1950) 8)R.D. Geckler, "The Mechanism of Combustion of Solid Propellants", in "Selected Combustion Problems-Fundamentals and Aeronautical Applications", AGARD Publications, Butterworths, London (1954), 289-339 9)C. Huggett, "Combustion of Solid Propellants" in Vol 2 of B. Lewis et al, Editors, "High-Speed Aeorodynamics and Jet Propulsion Combustion Process", Princeton Univ Press, Princeton, N.J.(1956), 514-74 10)M. Summerfield et al, Jet Propulsion **28**, 478-81(1958); "Burning Mechanism of Ammonium Perchlorate Propellants", Paper presented at the 13th Annual ARS Meeting, NY(1958)(ARS preprint 737-58); and ARS **29**, 791-92(1958) 11)W.S. McEvan, "State of the Art of Combustion of Solid Propellants", US Naval Ord Test Station, China Lake, Calf **AC/193**(1961)(Conf)(Not used as a source of info) 12)C. Lenchitz, *private communication*, PicArsn(1964)

Combustion Wave Propagation. This subject has been discussed in the following refs.

Refs: 1)J. Manton et al, JChemPhys **20**, 153-7(1952) & CA **46**, 6385(1952)(Nonisotropic propagation of combustion waves in explosive gas mixts and development of cellular flames) 2)G.H. Markstein, JChemPhys **20**, 1051-52 (1952) & CA **46**, 11688(1952)(Nonisotropic propagation of combustion waves)

Comet Powder. An older Amer commercial expl: K chlorate 75 & pine resin 25%

Refs: 1)Daniel(1902), 151 2)Pérez Ara (1945), 211

Command Guidance. A form of missile guidance, developed by the Germans during WWII and used to control the flight of HS-293 & Fritz 1400 air-to-surface missiles. In this method, the controlling radio-frequency signals are sent to one or several missiles which are in the air either from the ground (or from one of the missiles), in order to direct their flight or to influence their performance

For more detailed description see Refs

Refs: 1)A.S. Locke et al, "Guidance", VanNostrand, NY(1955), 562-74 2)G. Merrill, Edit, "Dictionary of Guided Missiles and Space Flight", VanNostrand, NY(1959), 138-39

Commercial Blasting Devices. This term includes various devices which are used to break coal in gaseous and/or dusty coal mines without resorting to the use of expls. In these devices, the pressure necessary for breaking up coal is usually produced in a closed container by one or more substances capable of developing high gas pressure, and then suddenly venting the compressed gases into borehole

Historical. The earliest of there devices was a cast-iron vessel invented in 1799. It contained highly compressed air which on heating expanded and cracked the cast-iron body, thus releasing air under high pressure. In its modified version, the vessel did not break on

heating, but the pressure of air was released thru a vent (Ref 3,p198). More successful were the devices invented more than a century later. Of these **Cardox** invented in 1920, achieved some popularity and it is still used in both Europe and America.

Cardox, briefly described in Vol 2,pC67-R of this Encycl and more fully in Ref 5,pp123-27) uses liquefied carbon dioxide as a pressure-producing medium. A similar device using water as a charge (in lieu of liq CO_2), was developed in Russia, and it presented certain advantages over Cardox (Ref 3,p198). There are also Cardox-type devices operated by compressed air. Two of them *Airdox* and *Armstrong Air-breaker* were developed in the USA in the early 1930's. They are listed in Vol 1,pp A117-R & A485-L of this Encycl and described in Ref 5, pp 137-42)

In view of the inconveniences of using liquid carbon dioxide, water or compressed air, a search was undertaken, beginning 1930's for solid mixts which would produce, on heating, flameless gases at high pressure. One of the most successful of these mixts consisted of dry Na nitrite and Amm chloride, in stoichiometric proportions. On heating a small region of this mixt by means of a BkPdr igniter, the decompn commencing in the vicinity of igniter would spread immediately thru the rest of the charge, exothermically, evolving gases and leaving a residue of NaCl. The reaction is assumed to proceed as: $NaNO_2 + NH_4Cl = NaCl + N_2 + H_2O$ (gas). This mixt has been used in a device known as **Hydrox**, which was developed in 1955 (Ref 5,pp 127-33). The pressure of gases evolved from Hydrox is sufficient to break the coal into desired size lumps. Another successful device utilizing a mixt of solid components for generating compressed gas, is the **Chemecol**. It was developed by the DuPont Co and is described in Ref 1,pp 79-82 & Ref 5,p133. It is listed in Vol 2,pC165-L of this Encyl; not described in the 1958 edition of Blasters' Hdbk (Ref 6)

The active list of US permissible "blasting devices", which was published in 1958 as InfoCirc **7832**, (Ref 4), included nine **Cardox** models (manufd by Cardox Corp of Chicago, Ill) and two **Hydrox** models, (manuf by Hercules Powder Co of Wilmington, Del). The active list published in 1962 as InfoCirc **8087** (Ref 7) includes also one model of **Cobra**. This device, manufd by Hercules Powder Co, contains a special AN compn which is converted to gas by a pressurized chem reaction initiated by an electrically ignited chem heater embedded in a starter mixture. The shell consists of a steel tubing 2-5/8" diam & 66" long and the cartridge is 1-7/8"in diam, 13" long and weighs 555g (Ref 7, p7)

General requirements and tests for blasting devices used in USA are given in Ref 2

In conclusion it may be mentioned that several devices utilizing water under high pressure have been developed. They are known as **Hydraulic Coal Bursters**. For their description, see Ref 5,pp 134-37

Refs: 1)Blasters' Hdbk(1952),79-82 2)Bureau of Mines Schedule **26A**, "Blasting Devices", Federal Register, March 22,1955, Vol 20,No56 3)Yaremenko & Svetlov(1957), 198 4)N.E.Hanna & G.H.Damon,"Active List of Permissible Explosives and Blasting Devices Approved Before December 31, 1957", USBurMinesInfoCirc **7832** (1958) 5)Taylor & Gay(1958), 123-44 6)Blasters'Hdbk(1958)- no description of blasting devices 7)N.E. Hanna, "Active List of Permissible Explosives and Blasting Devices Approved Before February 26, 1961," USBur MinesInfoCirc **8087**(1962)

Commercial Blasting Explosives, Cartridging of. See Anon, Explosivst **1964**, 67-71

Commercial Boosters. Under this title are listed in Ref 1 the following three items manufd by the DuPontCo:

a)DuPont Primacord Primers
b)DuPont "Nitramon" WW(Water Work) Primers and
c)DuPont "Seismograph Boosters"

In Ref 2 is listed only "Nitramon" WW Booster

Refs: 1)Blasters'Hdb(1952), 103-04 2)Blasters' Hdb(1958), 116

Commercial Detonators. See under BLASTING CAP in Vol 2,p B185 in this Encycl

COMMERCIAL OR INDUSTRIAL EXPLOSIVES

Civil or Civilian Explosives). They are expls used for other than military purposes, such as coal mining, blasting of rocks, etc. It is desir-

able that these expls combine their efficiency in executing certain work with economy and safety. Many of the older types expls are based on NG(such as Dynamites), but the trend in modern expls is to reduce the amt of NG by replacing it with other, less expensive components(such as DNT, TNT, etc). The majority of modern commercial expls contain AN and some combustible(such as charcoal, coal, woodmeal, coal tar, or petroleum products)

Typical example of modern inexpensive commercial explosive is *AN-Fuel* or *Do-It-Yourself Explosive* descriped by Cook(Ref 54,p 14) (See also Ref 54,pp 4,8 & 10)

Many commercial expls designed for certain kind of work may also be used for other purposes

According to Blasters'Hdb(Ref 52, pp 429ff, the term commercial explosives includes expls used for the following purposes:

Agricultural blasting which may be subdivided into ditching(Ref 52, pp 429-30) and field clearing(pp 439-46). The latter includes: stump, blasting(pp 439-46); boulder blasting(by "snakeholing" "mudcapping", or "bulldozing" method, called in GtBrit "plaster shooting") (p445) and rock ledge blasting(p 446) (See also Argiculture and Forestry Uses of Explosives in Vol 1,p A112-R of this Encycl)

Blasting ice jams(p 464)

Blasting in steel mills(pp 468-70)

Blasting log jams(pp 463-64)

Blasting old foundations(p 464)

Blasting underwater or submarine blasting, which includes harbor & channel work(pp 447-52) and blasting wrecked ships(pp 452-54)

Breaking & cutting steel (pp 465-68)

Coal Mining(pp 327-46 & 397-412)

Construction work, which includes: grade construction(Ref 35a,pp 413-23) and ditch blasting for pipeline construction(pp 423-28)

Coyote tunneling(pp 215-20 & 390-92)

Cutting timbers(p 463)

Demolishing bridge piers(pp 458-59)

Digging pole holes(pp 462-63)

Excavating in hard ground(pp 455-56)

Metallic mines. See Ore mines

Nonmetallic mines, such as limestone(pp 359-65), clay(pp 365-67), gypsum(pp 367-78), salt (pp 368-70), potash(pp 370-71), talc(pp 371-72) & phosphate(p 372). To these may be added expls used in Sicilian sulfur mines, such as *Tionite* listed in Ref 68,p 17

Open Pits(pp 392-93)

Ore Mines(p 347ff). The methods used in ore mines include "stoping" (pp 349-52), "caving" (pp 352-54), "long hole blasting"(pp 354-57) and "chute & grizzly blasting" (p 358)

Quarries (pp 373-90)

Seismic prospecting, called oil prospecting in Gt Brit (pp 471-86)

Shaft sinking (pp321-26)

Trenching in hard material (pp459-60)

Tunneling (pp 313-21) and

Well shooting (pp 460-63)

Explosives used in coal mines are the most important and numerous of all commercial expls. They are employed for breaking coal or rock in mines Expls for breaking coal should be of low brisance, but possess "heaving properties", so that the coal will be broken in rather large lumps without pulverizing it. Expls for breaking rock in mines are of higher brisance than those used for breaking coal"

Many expls described under BLASTING EXPLOSIVES Vol 1, pp B202ff of this Encycl, can be used in coal mining [See also: AMMONIUM NITRATE BLASTING EXPLOSIVES in Vol 1, pp A341ff; AMMONIUM NITRATE DYNAMITE in Vol 1, pp A355 & A356; Antigrisou (Explosifs) in Vol 1,pp A466 & A467; BLACK POWDER in Vol 2, pp B166ff: CHEDDITES in Vol 2, p C155 and CHLORATE EXPLOSIVES in Vol 2,pp C202ff]

Coal mining expls used in the USA are subdivided into *nonpermissible (nonpermitted* in Gt Brit) and *permissible (permitted* in Gt Brit) explosives. The former are employed in *strip-mining (opencast mining* in Gt Brit) or in *underground* mining, provided the atmosphere in mines does not contain expl mixts of firedamp/air or coal dust/air. The permissible expls are allowed to be used in any kind of coal mine, provided strictly prescribed conditions are fulfilled. In some countries, such as France, the expls used in gaseous and/or dusty coal mines are subdivided into those permitted for blasting only rock in such mines ("grisounites-roche" or "grisou-dynamites roche") and those allowed to

blast coal ("grisounites-couche" or "grisou-dynamites couche"). Some expls used in gaseous and/or dusty coal mines are *sheathed* (See further at the end of this section) *Historical on Coal Mining - and on Other Commercial Explosives.* As was stated under COAL, no explosive was used for breaking coal in mines until the beginning of the 19th century, although blasting of rock in sinking pits in some coal mines was practiced about one century earlier. Previous to this, however, ca 1630, BkPdr was used in Germany & Hungary for blasting rocks and ores in metal producing mines, such as of Cu or Pb

Until 1870, BkPdr was the only expl used in coal mining. As it was not safe against firedamp, many disastrous explns took place in coal mines. BkPdr was permitted, however, for surface blasting of any coal or for underground blasting of coals either contg no volatiles (such as anthracites) or contg a little of them (such as semibituminous coals) (Ref 49,p18). Because expl mixts known up to 1870 (such as those contg chlorates) were even less suitable than BkPdr, attempts were made to modify BkPdr by mixing it with "cooling agents", such as Amm sulfate, starch, paraffin, etc. One of the modified BkPdr's of that period is *Bobbinite* (see Vol 2,p 218-L of this Encycl). Other methods included: insertion of a cylinder filled with water in front of the charge in the borehole; use of wet moss or sawdust satd with an aq soln of alum for tamping; or dipping BkPdr pellets in paraffin. None of these methods proved to be successful (Ref 49,p 21)

When, in the middle of the 19th century, Schönbein invented NC and Sobrero invented NG, attempts were made to use NC and/or NG as ingredients of coal mining expls. One such expl mixts was *Tonite*, which consisted of NC pressed together with Ba nitrate powder. This expl, as well as *Ammoniakkrut*, invented in 1867 by Norrbin & Ohlsson (See Vol 1, p 306-R) and *Guhrdynamite* (NG 75 & kieselguhr 25%), invented the same year by Nobel, were not suitable for use in gaseous or fiery coal mines. Guhrdynamite, the first of the series of dynamites invented by Nobel, belongs to the class of "Powdery Dynamites with Inactive Base" (Ref 14,p 264). The later developed "Powdery Dynamites with Active Base" consisted of NG and an active adsorbent consisting of a fuel (such as woodmeal) and an oxygen contg salt (such as K or Na nitrate). These expls are known in US as *Straight Dynamites* (Ref 14, p283) (See also Atlas Powders in Vol 1,p A498-R)

In about 1875, Nobel invented a plastic expl which consisted of NG-NC jelly without other ingredients This expl, named in Ger *Sprenggelatine* and in Engl *Blasting Gelatin* (See Vol 2,p B211-R) was too brisant for use in coal mines. Less brisant were plastic expls: *Gelatin-Dynamites,* or simply *Gelatins,* also invented by Nobel. They contain NG-NC jelly (less than in Blasting Gelatin), plus a mixt of fuel and oxygen - contg salt. A typical compn consists of NG62.5, CollodCotton 2.5, woodmeal 8 & Na nitrate 27% (Ref 14,p10). There are also semi-plastic expls, such as *Gelignites* or *Semi-Gelatins.* They contain less NG-NC jelly than Gelatins, but more of other ingredients. A typical example is: NG 47.5, CollodCotton 2.5, woodmeal 3.5, rye meal 9 & K nitrate 37.5% (Ref 14,p 330)

As none of the expls developed up to about 1876 was safe against firedamp and/or coal dust and the number of coal mine explns increased, some European governments were considering the possibility of prohibiting the use of expls in fiery mines and resorting to the use of hydraulic devices or compressed air. Before resorting to such drastic measures, some governments decided to appoint scientists, or commissions headed by them, to investigate this problem and the first in this matter was France who appointed in 1876 the famous chemist M. Berthelot. As result of his recommendations the so-called "Commission Française de Grisou" (French Commission of Firedamp) was created in 1877 and then in 1878 the "Commission des Substances Explosives", with Berthelot its director. These commissions issued several reports but they did not solve completely the problem of making expls safe in gaseous and/or dusty coal mines. Other European countries followed the example of France and created commissions: Great Britain & Belgium in 1879 and Germany in 1880 (Ref 14,p 13)

Meanwhile Nobel started to incorporate AN in his dynamites. The first of these was patented

in 1879 under the name (in Ger.) of *Extra Dynamit* or *Ammon-gelatine-dynamit (Ammonium Nitrate Gelatin Dynamite).* It contained NG 71, NC 4, AN 23 & charcoal 2%. As this expl was not safe in fiery mines, Nobel reduced the amt of NG-NC and obtd a crumbly-plastic dynamite of compn: NG 25, NC 1, charcoal 12 & AN62%, which may be considered as a prototype AN permissible expl.. Another AN developed by Nobel was the so-called *Nobel Extra Dynamite Type II.* It contained NG-NC jelly 20-30 & AN 80-70%. A similar expl was developed in France under the name of *Grisoutine.* In Belgium a similar expl became known as *SGP* which stands for Secûrité, Grisou, Poussière (Ref 14,pp 11 & 14)

In order to test the newly developed expls,a testing gallery was constructed at Neunkirchen, Germany and rules for testing of expls for use in coal mines were established. Here was tested the "Wetter-Dynamit" of Müller & Aufschläger, prepd by mixing AN dynamites with considerable amts of salts contg water of crystalization (such as Na carbonate, Mg sulfate, alum, etc) in order tocool the gases of expln by evaporating the w of crystn. It passed the test. (Ref 14,p 14). Other "cooling agents", which were proposed later, included Amm oxalate, KCl & NaCl (Ref 49,p 30). Another expl tested and approved at Neunkirchen was the *Carbonit.* This mixt did not contain AN (See Vol 2,p C61-R of this Encycl)

Further development of expls safe against firedamp and/or coal dust will be discussed in section II of this item, entitled Coal Mining Explosives, Permissible. In section I, which follows, are described commercial expls which are not permitted in gaseous and/or dusty coal mines, but can be used for many other purposes

Section I

Coal Mining Explosives, Nonpermissible (Called Nonpermitted in Gt Britian). This term applies to expls suitable for use in blasting coal in mines contg no firedamp or other combustible gases (such as in anthracite or semibituminous mines). These expls are also suitable for blasting rock in coal mines. There are no exactly equivalent terms for "nonpermissible" in other languages, but the French term *grisounites-roche* or *grisou-dynamites-roche* and the Ger *Gesteinssprengstoff* refer to expls used for blasting rock in coal mines even if the mines contain firedamp and/or coal dust. In Russian these expls are called "nepredokhranitel'-nyiye" (Ref 48,p 25)

One of the requirements of any expl used in underground mines is to possess a positive oxygen balance to CO_2, usually ca 4%. This is in order to avoid the formation, on expln or deflagration, of CO which is extremely toxic and can form with air expl mixts. In this respect neither a K or Na oxidizer is an ideal oxygen supplier for mining expls, because the K or Na redical fixes oxygen, forming K_2O or Na_2O (Ref 49,pp 25 & 28-9). Amm nitrate, on the contrary, is very suitable, but its price in the 19th century was too high for use in mining expls. Only after 1920, when the synthetic nitrogen industry had been established and the price of AN dropped, it became possible to use it in many coal mining expls, especially in "permissible" NG expls (Ref 49,p 25)

One of the drawbacks of expls contg NG (such as dynamites) was their ease of hardening due to freezing of NG (fr p ca 13°). As NG expls in such a state are hard to initiate, it was necessary to thaw the frozen cartridges before inserting them in boreholes. In order to avoid the tedious and dangerous operation of thawing, the so-called "low-freezing" dynamites were developed. They are usually based on mixts of NG with Nitroglycol (fr p - 22.3°) (Ref 49, p 27)

As examples of expls suitable for use in nongaseous coal mines, may be cited several types of dynamites developed by Nobel, such as Gelatin-Dynamites, Semigelatin-Dynamites (such as Gelignites) & Straight Dynamites (See this section, under Historical). Many expls listed in Refs & Addnl Refs under Blasdng Blasting Explosives in Vol 2,pp B202 to B211 of this Encycl are suitable for coal mining

Belgian Commercial Explosives of Nonpermissible Type are listed in Vol 2,p B29 of this Encycl, under Belgian Industrial Explosives, items A, B, D & E

British Commercial Explosives of Nonpermitted Type are listed in Ref 28a, p14 and Ref 49, p 26). They include: *Ammonal* [AN 83, TNT 12

& Al 5%; powdery, d 1.1 & strength 88% of BG (Blasting Gelatin)]
Ammon-Gelatine Dynamite (AN 42.5, NG 50.0, NC 2.5 & carbonaceous material 5.0%; rubbery plastic, d 1.5 & strength 92% of BG)
Ammon-Gelignite - AN 44.0, NG 35.0, NC 1.0, carbonaceous material 5.0 & Na nitrate 15.0%; plastic, d 1.5 & strength 75% of BG)
Ammonium Nitrate Powder (AN 92 & carbonaceous material 8%) (powdery, d 1.15 & strength 81% of BG)
Ammon-Semigelatine (AN 78.7, NG 15.0, NC 0.3 & carbonaceous material 6.0%; cohesive, d 1.2 & strength 82% of BG)
Blasting Gelatine (NG 92 & NC 8%; rubberlike, d 1.5 & strength 100% of BG)
Nitroglycerine/Ammonium Nitrate Powder (AN 80, NG 10 & carbonaceous material 10%; powder, d 1.0 & strength 79% of BG)
TNT/Ammonium Nitrate Powder AN 82 & TNT 18%; powder, d 1.1 & strength 83% of BG)

French Commercial Explosives of Nonpermissible Type. Commercial expls, other than *Explosifs antigrisouteux,* are subdivided by Médard (Ref 31a,pp 209-19) into the following types:

Explosifs chloratés ou Explosifs du type OC. These are actually *Cheddites* and include the pre-WWII expls On°5, On°5A, On°6B, On°8, On°14,& On°14A. These compns, as well as expls 55-CSE-1948 & 58-CSE-1948, which were developed after WWII are listed in Vol 2, pp C157 & C159 of this Encycl. (See also Ref 31a, pp 210-12)

Explosifs nitratés ou Explosifs du type N. Among the expls developed before WWII, but still used in some cases may be cited: a) Nn°O [AN 78.7 & TNT 21.3%; power as CUP 120 (PA = 100)] b)Nn°1b(AN 91.5 & DNN8.5%; CUP 103) c)Nn°1c(AN 87.4 & DNN 12.6% (CUP (CUP 111). These three expls belong to the class of "explosifs de sûreté" (safe to handle & transport), although they are not safe against firedamp (Ref 31a,p 213) d)Nn°1d(AN 89, DNN 9 & woodflour 9%)(developed in 1947)(Ref 31a, p 223)

Explosifs nitratés à la pentolite. These expls were developed after WWII and two examples are given by Médard: a)Nn°20(AN 78.2, Pentolite 80/20 18.8 & woodflour 3.0%; CUP 119.5) b)Nn°21(AN 75.4 & Pentolite 80/20 24.6%; CUP 123.5)(Ref 31a,p 214)

Explosifs nitratés à l'aluminium. The first three expls listed below were developed before WWII, while the others after WWII: a)Nn°30 (AN 80.2, TNT 10.6 & Al 9.2%; CUP 132) b)Nn°31 (AN 78.5, Pentolite 80/20 12.3 & Al 9.2%; CUP 138) c)Nn°33 (AN 69, TNT 10 & Al 21%; CUP 146) d)63-CSE-1949 (AN 67, Pentolite 80/20 12 & Al 21%; CUP 147) (Ref 31a, p215) e)Nn°32 (AN 78, DNClBz 12 & Al 10% (Ref 31a, p223)

Explosifs nitratés résistant à l'eau. In these expls, crysts of AN were coated with 0.5 to 1% of Ca stearate. Good results were obtained with expls Nn°O & Nn°1c, but not with aluminized expls Nn°30 & Nn°31 (Ref 31a, p217)

Explosifs plastiques. These include: a)Explosif du type n°18, developed in 1933. It is listed as French Gelatin Cheddite n°18 in Vol 2, p C159, Table 4 of this Encycl b)*Nobélite* (NG 20, NC 1.6, Na nitrate 60, liq DNT 14 & woodflour 4.4%; CUP 78) (developed before WWII) c)*Tolamite* (NG 27, NC 1.0, AN 61, liq DNT 9.5 & woodflour 1.5%; CUP 131) (developed before WWII) d)*Sofranex A* (NG 40, NC 2, AN 48, liq DNT 2 & Al 8%) (developed after WWII) e)*Sevranite n°1* [Amm perchlorate 31, PETN 48, plasticizer (polyvinylacetate in liq DNT) 18 & Al 3%; CUP 142] (developed after WWII) - f)*Sevranite n°2* [Amm perchlorate 42, PETN 42 & plasticizer (as in n°1) 16%; CUP 138]

German Commercial Explosives of Nonpermissible Type (Gesteinsprengstoffe in Ger), used before and during WWII, are listed in Ref 53, pp Ger 29ff & Ger 69 and under individual compositions

No info at our disposal about current German commercial expls

Italian Commercial Explosives of Nonpermissible Type, are subdivided into: *esplosivi per uso a cielo aperto* (explosives for use in open work), *explosivi per galleria* (explosives for tunnel work) and *explosivi per cava* (explosives for quarry works) (Ref 73)

Following are current Ital nonpermissible explosives:

Montecatini's catalog (Ref 68) lists under the title *esplosivi da mina* (mining expl),the expls manufd by the firm, without giving their compns.

For some of them, however, compns are given either by Belgrano (Ref 35, pp280-81) or by Vettori (Ref 73)

Esplosivi gelatinati (Gelatinous expls): *Gomma A* - NG 92 & collodion cotton 8% (Ref 35, p281 & Ref 68). Its props as given in Ref 68, p 12 are: d ca 1.60, strength 100 & detonation rate 7600-7800m/sec; used in open works, tunneling & seismic research; *Gomma B* - NG 82.5, collod cotton 5.5, woodflour 3, Na nitrate 8.5 & Na carbonate 0.5% (Ref 35, p 281); not listed in Montecatini's catalog; *Gomma BM* - NG 66, collod cotton 4, AN 20, woodflour 2.7 & Na nitrate 7.3% (Ref 35, p 281 & Ref 73). Its props are given in Ref 68, p 12; *GEOM* - NG 57, collod cotton 3.5, AN 29, woodflour 3.5 & Na nitrate 7% (Ref 35, p281). Its props are given in Ref 68, p13. Dr. Vettori (Ref 73) gives for GEOM: NG or NGc 57, NC 3 and Amm & K nitrate 40%; *GDI°MT* - NG 38, collod cotton 2.3, AN 50.9, Na nitrate 6, red ochre 0.5, oil 2 & Ca carbonate 0.3% (Ref 35, p 281). Its props are given in Ref 68, P13. Vettori (Ref 73) gives for GDIMT: NG or NGc 38, NC 2, TNT 10, AN 48 & woodmeal 2%

GDI°MB Properties but no compns are given
GDS in Ref 68, p 14

Gelignite SA - NG 25%, the rest of components are not given (Ref 73); its props are in Ref 68, p 14; *Semigel.* Props, but no compn, are given in Ref 68, p 15

Explosivi pulverulenti. (Powdered expls):

Dinamon 1°. Props, but no compn, are given in Ref 68, p 15. Vettori (Ref 73) gives its compn as TNT + AN

Dinamon S. Same remarks as above

Cava M. Props, but no compn, are given in Ref 68, p 16

Explosivi per prospezioni geosismiche (Explosives for seismic prospecting):

GEO-DIN A Props are in Ref 68, pp 17-19,
GEO-DIN B but no compns
GEO-MON A

Esplosivi per usi speciali (Special uses expls):

Tutamite, used for blasting slag in Siemens-Martin furnances; its props, but no compn, are in Ref 68, pp 20-21

Mangiarotti's catalog (Ref 69), lists the following nonpermissible expls:

BM1 per galleria - TNT, AN & thermite; lt gray pdr
BM as per uso a cielo aperto - AN, nitrocompds & metallic powders; reddish pdr
BM ac per uso a ceilo aperto - AN & TNT; brn - yel pdr
BM 57 per uso a cielo aperto - based on AN; gray - blk pdr
Super BM per galleria - based on org nitrocompds, org nitrates and plasticizers; lt green, sl plastic pdr
BM a2 - AN, TNT & thermite; brown pdr
Super BM cava - compn not given
BM 2 per galleria - based on inorg nitrates, org nitrocompds & powdered Al; shiny gray pdr

Sorlini's catalog (Ref 70) lists the following expls:

Martia Alpha, Martia Beta, Martia Gamma, Martia Delta 45 and *Martia Eta S.* All these expls are for use in open works ("per impiego a cielo aperto"). Their props, but no compns, are given in Sorlini's catalog

Sorlini also manufactures *shaped charge* expls (Cariche cave) for breaking large insolated rocks

Vulcania's catalog (Ref 71) of the firm located at Brescia and now belonging to the Consorzio Fabricanti Dinamiti (which includes also the firms of Montecatini - SIPE and Bombrini Parodi Delfino) lists the following nonpermissible explosives:

Ager C (AN & aromatic nitrocompds); for quarries & nongaseous mines
Ager D (AN & aromatic nitrocompds); for open works
Ager E (AN & aromatic nitrocompds); for open works
Antonite per cava (AN & TNT); for quarries & nongaseous mines
Antonite per galleria (AN & TNT); for tunneling
Cariche cave. Shaped chge expls of 200, 350, 500 & 1000g for civilian uses
Sismite: AN 48, TNT 32 & Tetryl 20%; used for seismic prospecting (Ref 73)
VE_4 (AN & aromatic nitrocompds); for quarries & nongaseous mines
Vulcania DB (inorg nitrates & TNT); for seismic prospecting
Vulcania DBS (AN, TNT & RDX); for seismic prospecting
Vulcanite P (inorg nitrates, aromatic nitrocompds & org nitrates); for tunneling in hard rock

Vulcanite PR (inorg nitrates, arom nitrocompds & other ingredients); for tunneling in hard rock

In addn to the above listed Italian nonpermissible expls, the following may be included:

Albita italiana: AN 58, NGu 19 & GuN 23% (Ref 29, p 95)

Amatolo (for mining): AN 90 & TNT 10% (Ref 35, p 289, table)

Ammondinamite: NG 63, collod cotton 2, AN 30 & woodflour 5% (Ref 14a, p 339)

Ammonite No 1: AN 88, DNT 3, vegetable flour 5 & DPhA 1% (Ref 14a, p 339)

ANS (Esplosivo) or Antisanzionate (also designated as ASN). See Vol 1, p A457-R of Encycl & Ref 29a, p 116

Astralite. See vol 1, pp A497-98 of this Encycl

Avigliana 3 or Nitramite. See Vol 1, p A516-R of Encycl

BM(Esplosivi). See above under expls listed in Mangiarotti's catalog (Ref 69)

Cadinite or Kadinite: NG 26, Na nitrate 56, sulfur 10, carbon 4 & lignosic substance 4% (Ref 35, p 173 & Ref 58, p 388)

Chedditex. See Vol 2, p C159, Table 4 of this Encycl

Cloramite. See in this Vol

Cremonita. See in this Vo.

Dinamiti. Various compns are given in Ref 35, pp 280-81 & Ref 58, pp 338-45

Dinamon: AN 69, K chlorate 8, TNT 20 & Al 3% (Ref 35, p 163)

Dinamon 1° & *Dinamon S* are listed in Ref 39, p 32 w/o giving their compns. They are manufd by Consorzio Fabricant Dinamiti

Echos or Escho: AN75-80, ferrosilicon or silicon 14-16, ipposino (horse dung) 5-7 & Al 2-3% (Ref 25a, p 225)

Esplosivo P: Amm perchlorate 61.5, Na nitrate 30 & paraffin 8.5% (Ref 14a, p 205)

Esplosivo S: Na chlorate 90, paraffin 7 & vaselin 3% (Ref 14a, p 205)

Esplosivo S20: *AN 79, TNT 20* & woodflour 1%

Ref: L.Médard & A.LeRoux, MP **34**, 201 (1952)

Gelatina 808: NG 62, NC 18 & DNT 20% (Ref 29a, p 106)

Gelatina-dinamita NO: NG 74, collod cotton 5, $NaNO_3$ 15.5, woodflour 5 & Na_2CO_3 0.5% (Ref 58, p 344)

Gelignite: NG 59, collod cotton 4, K nitrate 29 & woodflour 8% (Ref 14a, p 291)

Gelignite all'ammonio: AN 70.0, NG 29.3, & collod cotton 0.3% (Ref 14a, p 291)

Gomma (Esplosivi). See above under item A

Gomma incongelabile: NG 65-72, collod cotton 5-6, liquid TNT 7-8 & AN 15-22% (Ref 14a, p 303)

Idrolita: AN 70, RDX 20, paraffin 3 & water 7% (Ref 21a, p 246)

Imperialita: AN 85-90 & Al scales 15-10%. It was invented ca 1913 and intended for use as a blasting expl. This idea was, however, abandoned after the inventor was killed in a violent expln during manuf of this compn (Ref 58, p375)

Kadinite. Same as Cadinite

Macarite: Pb nitrate 72 & TNT 28% (Ref 35, p 177)

Mangiarotti's Mining Explosives. Mixts of C powder (particle size < 0.4mm), AN and expl nitrocompds. Example: C 5-13, TNT 0-15, RDX 0-5%, the rest being AN. Can be prepd either by cold mixing of ingredients or by hot mixing with molten TNT

Ref: G.Mangiarotti, ItalP 577811 (1958) & CA **55**, 3982 (1961)

Manlianite: Amm perchlorate 72, carbon 14.7 & sulfur 13.3%. It is one of the expls proposed by U.Ulvisi (Ref 58, p 401)

Martia(Explosivi). See above under Sorlini's catalog

Nitramite. Same as Avigliana 3

Piombite: Pb nitrate 76, TNN 16, Ca silicide & vaseline 3% (Ref 35, p 177)

Plastigel. See Vol 2, p C159, Table 4 of this Encycl

Polvere "Cannel". See Cannel Explosives in Vol 2, p C25-R of this Encycl

Romite galleria & Romite cava, for use in open work have been manufd by Polverificio Stacchini. They are listed in Ref 39, p 32 w/o giving their compn

Sabulite: AN 78.2, Ca silicide 13.8 & TNT (or TNN or DNB) 8.0% (Ref 58, p 376)

Solfite: AN 83-88 & sulfur 17-12%; it was proposed in 1930 by Dr. Rannoncini (Ref 58, p 377)

Umbrite A: NGu 48.4, AN 37.3 & ferrosilicon 14.3% (Ref 35, p 133)

Umbrite B: NGu 45.1, AN 41.4 & ferrosilicon 13.5% (Ref 35, p 133)

Vulcan: NG 30.0, Na nitrate 52.5, carbon 10.5 & sulfur 7.0% (Ref 58, p 388)

Japanese Commercial Explosives of Nonpermissible type some of which have been used for military purposes:
Ammonyaku: AN & charcoal (Ref 24a, p29)
Dainamaito: Japan for Dynamite
Ennayaku: K chlorate 80, MNT 15 & castor oil 5% (Ref 24a, p 28)
Entoyaku: K chlorate 80, DNT 16 & castor oil 4% (Ref 24a, p 28)
Haensosanbakuyaku or Type 88 Explosive: Amm picrate 75, ferrosilicon 16, woodmeal 6 & petroleum 3% (Ref 25b, pp 152-53)
Haishokuyaku No 1: Amm perchlorate 77, RDX 17, Si carbide 1.5 & paraffin 4.5% (Ref 24a, p 28)
Haishokuyaku No 2: Amm perchlorate 48, GuN 20, RDX 25, graphite 2 & paraffin 5% [PB Rept 50394 (1946)]
Matsu. Japan for Blasting Gelatin
Sakuma-dainamaito. Japan for Gelignite
Shoanyaku. See under Japanese permissible expls
Shotoyaku. Japan for Amatol

This list does not include the numerous military explosives given in Ref 24a, many of them can also be used as commercial expls

Russian Nonpermissible Explosives (Nepredokhranitel'nyiye Vzryvchatyiye Veshchestva, in Russ):
Almatrity. See Ref 45a, p Rus 1 and Vol 1 p A140-L of this Encycl
Ammoksil or Ammonxyl: See Ref 45a, p Rus 1 & Vol 1, p A286-R
Alumit No 1: AN 80, TNT 12 & Al 8% (Ref 46, p 104)
Ammonaly. Several types contg AN, TNT & Al powder are listed in Ref 45a, p Rus 1 & in Vol 1, p A292-L. Newer types, such as Skal'nyi Ammonal No 1 and Skal'nyi Ammonal No 1-JB (Rock Blasting Ammonals) are composed of RDX, powdered Al and coated AN (Ref 47, p 160)
Ammonity, such as Ammonit No 6 (AN 79 & TNT 21%); Ammonit No 6, JB (Coated AN 79 & TNT 21%); Ammonit No 7 (AN 81-2, TNT 14 & woodflour 5-4%); Ammonit E-3 (AN 82, TNT 16.5, asphaltite 1.0 & paraffin 0.5%) (Ref 46, p 104 & Ref 47, p 158) (See also Ref 45a, p Rus 1 & Vol 1, p A308-R of this Encycl)
Ammonit-Goudronit. See Ref 45a, p Rus 2 & Vol 1, pA310-L
Ammonpek. See Ref 45a, p Rus 2 & Vol 1, p A382-L
Ammontol. See Vol 1, p A383-L
Belity. See Ref 45a, p Rus 3 and Vol 2, p B-33-L under Bellites
Dinaftalit, Zernënnyi K(Granular Dinaphthalite) AN 88 & DNN 12% (Ref 46, p 104)
Dinamity, such as Gremuchii Studen' (Blasting Gelatin) - NG 87-93 & Collodion cotton 13-7% and Studenistyi (Zhelatin) Dinamit, also called Plasticheskii Dinamit (Plastic Dynamite): NG 62-83, collodion cotton 3-6, K or Na nitrate 27-29 & woodmeal 8-2% (Ref 45a, p Rus 5)
Dinamony, such as Dinamon D (AN 85 & woodflour 15%); Dinamon K (AN 85 & pine bark flour 15%); Dinamon SSh (AN 85 & pine cone flour 15%); Dinamon T(AN 88 & lignite 12%) (Ref 47, p 149) (See also Ref 45a, p Rus 3 under Dynammons)
Igdanit. An expl proposed in 1961 by Khablov: AN (granulated) 95-7 & Diesel fuel oil 5-3%; the ingredients are combined just before use (Ref 61)
Mansit: AN 72, Amm picrate 23 & petroleum asphalt 5% (Ref 45a, p Rus 11)
Pobedity: These are Ammonity (See item E) to which 8-10% of NG is added. Vaskovskii (Ref 48, p 25) lists Pobedit No 6 and Pobedit VP-2 as nonpermissible expls, but he does not give their compns
Skal'nyiye Ammonaly. See under item D

Spanish Commercial Explosives of Nonpermissible Type (Explosivos industriales de no seguridad, in Span):
Amonal (AN 78.5, K nitrate 17.5, Ba nitrate 0.5, Al 1.0 & vegetable oil 2.5% (Ref 25, p 372) b)AN 84.5, K nitrate 1.5, Ba nitrate 0.5, Al 5.5 & charcoal 8.0% (Ref 25, p 372) (See also Vol 1, p A289 of this Encycl)
Amonita número 5: AN 80, TNT 14, NG 2, sawdust 2 & ferrosilicon 2% (Ref 25, p 377)
Dinamitas manufd at the Fábrica de Galdácano (Vizcaya): a)Dinamita goma num 1, especial - NG 71.78, NC 5.00, AN 20.22 & flour 3.00% b)Ditto núm 2, especial - NG 37.50, NC 2.25, AN 18.00, Na nitrate 29.25 & flour 13.00% (Ref 25, p 397) c)Dinamita goma núm 2 - NG 49.00, NC 2.50, Na nitrate 35.50 & flour 13.00% d) Ditto núm 3 - NG 25.00, AN 22.00, Na nitrate

45.00 & flour 8.00% e)Ditto núm 3 - NG 22.50, Na nitrate 65.00 & carbon 12.50% (Ref 25, p 398)

Donarita - AN 80, NG 4, TNT 12 & linseed flour 4% (Ref 25, p 383)

Explosivos F.E, developed during WWII by Capt A. Otaolauruchi: a)Explosivo F.E-1 - AN 68, TNT 17 & $Na_2HPO_4..ZH_2O$ 15% b) Explosivo F.E2 - AN 70, TNT 20 & Na_2HPO_4-$.7H_2O$ 10% and c)AN 65, TNT 25 & $Na_2HPO_4.7H_2O$ 10% (Ref 25, p 384)

Nitramita española de la Fábrica de Granada: AN 80 & TNT 20% (Ref 25, pp 378 & 383) (Compare with item under Spanish Permissible Explosives)

Plomoplastrita, also called *Ploplastrita.* A mixt of finely pulverized TNT, soln of collodion cotton & Pb nitrate (Ref 25, p 381)

Sabulita, listed in Ref 25, p 382 consists of TNT, AN & Ca silicide

Sabulitas españoles, núm 0 & núm 1, listed in Ref 36, pp 117 & 119, contain TNT, AN, Ca silicide (14 & 12%, respectively) and K perchlorate (8 & 9.5%, respectively)

Tonita: Ba nitrate 68, DNB 13 & NC 19% (Ref 33a, p 31)

Triplastita. A gelatinous expl prepd by mixing DNT with NC (Ref 25, p 380 & Ref 33a, p 31)

Swedish Commercial Explosives of Nonpermissible Type. With the exception of the book on Nitroglycerin and Dynamite (See Ref 55b), we do not have knowledge of any recent Swedish books, and therefore, our info about current Swed expls is rather limited

The following list includes some of the older nonpermissible types & some military compns:

Ammongelatine Dynamite of Nobel or Extra Dynamit. See Vol 1, p A295-R of this Encycl

Ammoniakkrut. See Vol 1, p A306-R

Blastin: Amm perchlorate 63, Na nitrate 23, DNT 8 & paraffin wax 6%. See also Vol 2, p B184-L this Encycl

Blasting Gelatin. See Vol 2, p B219-R of this Encycl

Bofors Plastic Explosive. See Vol 2, p B219-R of this Encycl

Bonit. See Vol 2, p B248-R

Dynamit. The first dynamit was prepd in Sweden by A.Nobel in 1863 by pouring NG into zinc cartridges contg pulverized black powder. As BkP dr was not a good adsorbent for NG, Nobel replaced it in 1866-67 with kieselguhr (See Guhrdynamit). The next invention of Nobel (1867) was dynamite contg NG adsorbed by charcoal. In the same year Trauzl proposed a dynamite consisting of NG 73, guncotton 25 & charcoal 2% and in 1868 Schultze patented under the name of *Dualin* a mixt of NG 50 & woodnitrocellulose 50%. The first dynamite which corresponds to present formulations was invented by Nobel in 1869. It was prepd by coating with NG the particles of a mixt of an oxidizer (such as Amm, Ba, K or Na nitrates) and solid combustible materials (such as woodmeal, charcoal, rosin, starch or sugar). Example: NG 20, Ba nitrate 70 & rosin or charcoal 10%. These dynamites with an "active base" are also known as Straight Dynamites (Ref 14, pp 282-86)

Dynamite No 1. Same as Guhrdynamit

Dynamite with Inactive Base. See Guhrdynamit

Extra Dynamit. See Ammongelatine Dynamit.

Gelatindynamit (Gelatinerad-dynamit in Swedish), was invented in 1875 by A. Nobel and prepd by dissolving 2.5 parts collodion cotton in 62.5 ps of slightly warmed NG and incorporating into the resulting soft jelly 27 ps of K nitrate & 8ps of woodmeal (Ref 14, p324)

Guhrdynamit or Nobel's Safety Powder (Dynamite with an Inactive Base or Powdery Dynamite) was prepd in 1866-67 by A.Nobel by mixing NG 75 with diatomaceous earth, known as guhr or kieselguhr 25% (Ref 14, p 264)

Hexotol. Same as Bonit (See also Ref 59a, p 201)

Hexotonal is a mixt of RDX, TNT & a finely divided Al. Small portions of desensitizers (wax) are added, and in some formulations carbon black. Examples: a)RDX/TNT/Al/Wax - 40/44/15/1 b)RDX/TNT/Al - 30/50/20 plus 1% wax & 1.5% C black (Ref 59a, 203)

Nauckhoff's Explosives. Low-freezing, blasting plastic perchlorate expls patented in 1909 & 1910 by S.A.G.Nauckhoff of Sweden. It was claimed that these expls were equal in strength to dynamites. One of these expls, known as *Territ,* contained Amm perchlorate 43, Na nitrate 28, liquid mixt of DNT & TNT 27.8 & collod cotton 1.2%, As the above compn was difficult to detonate, later formulations of

Territ contained some NG (Ref 8a, pp 247 & 681; Ref 18, p 316)
Nobelit. See under Swedish permissible expls
Nobel's Safety Powder. Same as Guhrdynamit
Novit: a)TNT 60 & HNDPhA 40% and b)TNT 55.7, HNDPhA 27.9 & Al powder 16.4%; used for underwater explns [Ref 25b, p 107 and A. Stettbacher, Protar **9**, 38 & 41 (1943)]
Seranin. A blasting expl patented in 1867 by Björkmann of Sweden: AN 72.5, NG 18.1, woodmeal or charcoal 8.7 & benzene or creosote 0.7% (Ref 1, p 713).
Sprängdeg. Swedish for plastic explosives, such as *Bofors Plastic Explosive* described in Vol 2, p B219-R of this Encycl
Spränggelatin. Swed for Blasting Gelatin
Svartkrut. Swed for Black Powder
Territ. See under Nauckhoff's Explosives

Swiss Commercial Explosives of Nonpermissible Type (Some of them "Gesteinssprengstoffe", others "Sicherheitssprengstoffe". The latter are "safe to handle", which does not mean that they are safe for use in gaseous or dusty coal mines):
Aldorfits. See Vol 1, p A123-R of this Encycl
Ammonpenthrinits. See Vol 1, p A382
Cheddite, Suiss 60N. See Vol 2, p C157, Table 3
Dynamit F. Same as German Dynamit 1 (NG + NGc 65, collod cotton 2.5, Na nitrate 27.0 & woodmeal or ryemeal 8%; with or w/o pptd chalk 0.5%); except that Na nitrate is replaced with AN, and woodmeal (or ryemeal) is replaced with other C-contg substances (Ref 27, p 85 and private communication from Dr A.Stettbacher, Zürich, 8 July, 1954)
Gamsits. A class of AN dynamites manufd at Gamsen bei Brigg and successfully used in blasting some tunnels thru the Alps. One of these called Telsit-Gamsit or Gelatine-Gamsit is described here under Telsits. Another compn known as Gamsit A or Gelatine-Telsit J consists of NG + NGc 22, collod cotton 1.5, liq TNT 13 & AN 63.5% (Ref 27, p 86). This expl seems to be identical with Gelatine-Aldorfit A, manufd at Dottikon and with Gelatine-Telsit J, manufd at Isleten-Liestal. Trauzl test value of Gamsit A is 433cc (vs 311 for TNT), but there are also weaker Gamsits B & C, but their compns are not given by Stettbacher (Ref 27, p 114)
Gelatine-Aldorfit A. Same as Gamsit A. There are also Gelatine Aldorfits B & C, but their compns are not given by Stettbacher (Ref 27, p 114)
Gelatine-Cheddite. See under Cheddites in Vol 2, p C159, Table 4
Gelatine-Dynamit 1 - NG 45, NGc 11.5, collod cotton 3, woodmeal 8.5 & Na nitrate 32% (Ref 18, p 233)
Gelatine Gamsit. Same as Telsit-Gamsit
Gelatine-Penthrinit. A series of expls developed by A.Stettbacher consisting of Penthrinit (PETN + NG) in which collod cotton is incorporated in order to make the blend more plastic and less exudable. Eg: a)PETN 50, NG 35, NGc 11.7 & collod cotton 3.3% (Ref 14b, p 9) b)PETN 40.9, NG 40.9, collod cotton 1.6 & AN 16.6% (Ref 14b, p 39) c) PETN 15.5, NG 77.5 & collod cotton 7% (Ref 14a, p 847)
Gelatine-Penthrinit-Perchlorat - PETN 59.0, NG 24.7, collod cotton 0.6 & Amm perchlorate 15.7% (Ref 14a, p 847)
Gelatine-Telsit J. Same as Gamsit A described here under Gamsits. There are also listed by Stettbacher (Ref 27, p 114) Gelatine - Telsit C and Gelatine-Telsit, spezial, but their compns are not given
Pentastits. Blends of PETN with 5-8% of PE-tri- and tetrastearate developed by the Schweizerische Sprengstoff-Fabrik AG at Dottikon. They are intended for use as military expls but can also be used as blasting expls [Swiss P 220756 (1942) & CA **42**, 6538(1948) A.Stettbacher, private communication, 8 July 1954]. Some props are given in Ref 27, pp 122-23
Penthrinits. Plastic, non-exuding expls, consisting of PETN & NG, developed by Dr. A.Stettbacher beginning 1928-29. They are actually military expls, but can be used for blasting hard materials, such as rocks. See also *Ammonpenthrinits* and Gelatine-Penthrinits (Refs 14a, 14b, 18 & 27)
Pentro or Pentryls. Blends of PETN & TNT, similar to Amer Pentolites. Some props are given in Ref 127, p123
Pierrit. A blasting Cheddite-type expl consisting of K chlorate 80, MNT 12, PA 2 & castor oil 6%. Large quantities were used during construction of Simplon Tunnel [E.

Bravetta, SS 1, 122 (1906)]

Plastolit. A plastic expl, safe to handle: Na nitrate 57, NG 15, NGc 4, collod cotton 1.3, liq DNT 13, woodmeal 9.6 & Na bicarbonate 0.1% (Ref 18, p 233)

Simplonits. Blasting expls manufd by the Société Suisse des Explosifs, Gamsen-Brigue. Accdg to Dr. Stettbachor (private communication, Dec 14, 1953) compns of some of them are similar to those of Gamsits. Some props of Simplonit A, pulvrig (mit Aluminum) and of Simplonit B, pulvrig are given in Ref 27, p 114, without listing their compns

Spezialsprenggelatine. A name given by Stettbacher to a low-freezing powerful expl which was manufd at Isleten plant and used in the construction of Jungfrau tunnels. Essentially this expl was based on blasting gelatin, in which part of the NG was replaced by an aromatic nitrocompd (Ref 13, p 113)

Sprenggelatine 93/7 or Dynamit Gomme J. The usual compn is NG 93 & NC(ca 11.5%N) 7%, d ca 1.6, OB to CO_2 ca +0.75%, powder by Trauzl test 520cc (TNT 311cc) brisance by copper cylinder compression test 131% TNT. It has been manufd at the Isleten-Liestal plant of the Schweizerische Sprengstoff Fabrik AG (Ref 27, pp 82, 114 & 149). Another expl, Sprenggelatine 92/8, listed in Ref 27, p 104, has the following properties: d 1.60, loading d 1.50-1.55, gas volume 711 liters/kg, heat of explosion (H_2O liq), Q_v=1630kcal/kg, temperature of explosion 4970°, detonation rate 7800m/sec and impact sensitivity, 2kg wt 15cm. Both types of Sprenggelatine might contain nitroglycol in order to make them low-freezing.

Telsits. A series of dynamites manufd by the Schweizerische Sprengstoff AG Cheddit & Dynamit at Isleten and used in the blasting of tunnels. Compns of some of them are similar to those of Gamsits. The so-called Telsit-Gamsit consists of AN 60, NG 21, NGc 5, collod cotton 1.6, woodmeal 0.4 & liq DNT 12% (Ref 18, p233) and Telsit (Sicherheitsdynamit) consists of AN 55.5, NG 22, collod cotton 1.5 & liq DNT + TNT 21% (Ref 18, p 242)

Totalit. A blend of AN 95.5 & paraffin (of mp 54-6°) 4.5%, suitable for blasting and for military purposes. Its props are described by A. Stettbacher, Nitrocellulose **10**, 109-10 & 128-30 (1939)

Unknown Name Explosives. a) Ammonium Nitrate Explosive of Société Suisse des Explosifs: AN 70, PETN 10, TNT 10 & $NaNH_4HPO_4.4H_wO$ 10% [SwissP 228940 (1943) & CA **42**, 6538 (1938)] b)Plastic Explosives of O. Matter: 1)Mixt of coal tar distillates with inorganic oxidizers, such as nitrates, chlorates or perchlorates [SwissP 220755 (1942) & CA **42**, 6538 (1948)] and 2)Mixt of a water-insol hydrocarbon (such as chloronaphthalene) and resinous thickening agent (such as rosin or tar-distillation residue) with inorganic oxidizers (such as nitrates, chlorates or perchlorates) [SwissP 228653 (1943) & CA **43**, 2437 (1949)]

Vergé Explosives. Low-freezing dynamites patented by A.E.Vergé in 1913. Essentially they consist of NG, NC, liq DNT + TNT and inorganic oxidizer such as nitrate or perchlorate. Eg's: a)NG 40.0, liq DNT + TNT 17.0, NC 5.0 & Amm perchlorate 38.0% b)NG 37.0, liq DNT + TNT 15.0, NC 4.5, Amm perchlorate 25.2 & Na nitrate 18.3% c)NG 41.0, liq DNT + TNT 18.0, NC 3.0 & K perchlorate 38.0% (Ref 13, p 112 & Ref 14, pp 364-5)

United States Commercial Explosives of Nonpermissible type. Many expls of this type are known and used in mining, quarries, etc and they are too numerous to describe in this section. It would be more appropriate to describe them in alphabetical order throughout the Encyclopedia, such as Blasting Explosives, (See Vol 2, pp B 202ff), Blasting Gelatin (See Vol 2, p B 211-R), etc.

The most important group of commercial expls, Dynamites, will be described in forthcoming Vol 4 of this Encyclopedia

Section II

Coal Mining Explosives, Permissible (Permitted in GtBritain). This term applies to explosives which, because they have passed special prescribed tests, can be used in coal mines contg firedamp and/or coal dust. These tests are described under COAL MINING EXPLOSIVES, TESTING FOR PERMISSIBILITY. Approved expls will not ignite expl mixts of firedamp, coal dust & air, when used under prescribed conditions.

Permissible expls are known in Belgium

as *Explosifs SGP* (Explosifs Sécurité-Grisou -Poussière); in France as *Grisounites-couche, Explosifs antigrisouteux, Grisoutines* or *Grisou -dynamites;* in Germany as *Wetter-* or *Schlagwettersichere Sprengstoffe;* in Italy as *Esplosivi ammissibli, Esplosivi antigrisoutosi, Explosivi grisou or Esplosivi di sicurezza;* in Spain as *Explosivos aprobados, Explosivos antigrisú or Explosivos de seguridad contra el grisú;* In Russia as *Predokhranitel'nyiye-* or *Antigrisutnyiye- Vzryvchatyiye Veshchestva*

The term "Safety Explosives" means expls safe to handle in storage or in transportation, but does not mean that they are "safe against firedamp". Expls which do not contain NG are usually safe to handle. The French term "Explosif de sûreté" is defined in MP **9**, 157 (1897). The Ger term "Sicherheitssprengstoff" has sometimes been used to mean "safe against firedamp" (See Ref 18, p 242, bottom line), but it is preferrable to use the term only in the sense of "safe to handle and transport", although the Germans have for these expls a special name, "Handhabungssichere Sprengstoffe"

The Brit term "Authorized Explosives" refers to expls which conform to certain conditions with respect to "safety in handling or transport". The Ital term for safety expls, "Explosivi di sicurezza" and Spanish "Explosivos de seguridad" are also used to mean expls safe against firedamp. The Rus term "Bezopasnyiye VV", means safety expls

There were formerly Brit expls on a "Special List" which meant that they passed special, very vigorous tests and could be used in coal mines known as most dangerous from the point of view of expln of firedamp.

In US practice, an expl in order to be placed on a A "Permissible List", should undergo a series of tests designed to prove that it will not cause the ignition of the following expl mixts: firedamp & air; coal dust & air; and firedamp & coal dust & air (See also under COAL MINING EXPLOSIVES, TESTING FOR PERMISSIBILITY)

As was mentioned in this section under "Historical", several European countries appointed beginning in 1877, various "commissions" to develop expls which would be safe to use in coal mines contg firedamp and/or coal dust. Many well known scientists took part in these investigations

As the 1st French commission on firedamp did not come to any conclusive decision in regard to expls, safe against firedamp, the 2nd commission was created in 1887, under direction of Berthelot. This was followed shortly by appointment of Austrian commission and construction of a gallery at Mährisch Ostrau (now Moravska-Ostrava, Czechoslovakia). This gallery, as well as other European galleries, was similar to that at Neunkirchen (See also under Coal Mining Explosives, Testing)

It seems that the most important research of the 1880's was done in France by Mallard & Le Chatelier. One of their principal findings was that although the minimum ignition temp of firedamp is ca 650°, it does not mean that any expl developing this temp on blasting coal would necessarily ignite the gas. This is because there is always a certain delay period of induction before the firedamp actually ignites and the higher the temp developed on blasting the shorter is the delay period. For example, at a temp developed on expln (or deflagration) of a charge equal to 650° (as calcd by the method approved by the Commission des Substances Explosives), the delay period is ca 10 sec and if "duration of flame" for this charge is less than 10 sec, the firedamp would not ignite. The delay at 1000° is ca 1 sec and at temp of 2200°, there is practically no delay, and the firedamp would ignite immediately following the expln of the charge. This means that charges with temps of expln below 2200° would not ignite the gas unless the duration of flame is longer than the delay period and those with temps of 2200° or higher would usually ignite the firedamp. Sometimes brisant expls do not ignite the firedamp even if their temps of expln is higher than 2200°. This is because their flame is of extremely short duration

The "Commission du Grisou" confirmed the findings of Mallard & Le Chatelier but ruled that the max temp, 2200°, established by them is too high and that it would be safer to lower it to 1500°C for expls intended for use in a coal seam ("couche" in Fr) and to 1900° for those intended for use in the accompanying rock ("roche" in Fr) (Ref 1, pp 747-50; Ref 14, pp

384-87; Ref 17, pp 230-51; Ref 47, pp 113-17 and Ref 59, pp 497-98)

Duration of flame is usually proportional to the *length of flame* and the latter is in many cases proportional to the temperature of flame. In general, the shorter the flame, the safer the explosive. There are, however, some exceptions to this rule, when an unusually long flame is produced at comparatively low temp of flame. This is particularly noticeable when the expl mixt is compounded to have an oxygen balance negative to CO_2. In this case, the combustible gases CO, H_2 & CH_4, evolved together with CO_2, H_2O & N_2, would be ignited with some delay, thus forming an additional flame some distance from a borehole (Ref 14, pp 384-87). Duration and length of flame may be detd as described in US BurMines Bull **346** (1931), pp 67-71)

Another phase of investigations conducted in France concerned the so-called "Charge Limite". This problem is discussed in Vol 2, p C153-R of this Encycl (See also Refs in Vol 2, p C153-L)

Audibert, who conducted investigations after Mallard & Le Chatelier, worked out the following explanation of ignition of firedamp during blasting operations. When gases, evolved on expln of a charge in a borehole, reach firedamp atm, they start to mix with it and at the same time to raise its temp. The resulting mixt, in the neighborhood of borehole, consisting of methane, air & products of expln, would explode when the temp of gas reaches at least 650° and the CH_4 content in air would be within the ignition range (Ref 47, pp 114-15 & Ref 59, p 498).

Considerable work on safety of expls against firedamp and/or coal dust was done in Germany since the Govt appointed in 1881 a special commission (Ref 23, p 347). The work was also done by private concerns employing chemists like Müller, Aufschläger, Heise, Kast, Bichel, Beyling, Naoúm, etc. As results of their work, various Wetterdynamite were developed and strict mining regulations were introduced (Ref 14, pp 387-94) (See also Ref 47, p 117 and Ref 59, pp 500-02)

Some important work was done in Russia before & after WWI, both by private concerns and the govnt. Among the recent investigators may be mentioned the names of Andreev, Beliayev, Dubnov, Gol'binder, Semenov, etc (Ref 47, p 115 and Ref 59, pp 502-03) (See also Refs 26, 28, 41, 45a, 46, 47, 48, 57 & 59)

The work conducted in Belgium up to 1914 was mostly under the direction of Wetteyne (Ref 21, p 554). His, and later investigators' research led to the development of successful permissible expls, known now as "explosifs SGP" (explosifs secûrite-grisou-poussière) (See Vol 2, p B29-R of this Encycl). Many of these expls are exported

The work on development of British expls, safe against firedamp and/or dust (known now as "permitted") began sometime in 1870's and several Royal Commissions (the first in 1879) were appointed since then. Many formulations were suggested, but only the expls which underwent and passed the test in the galleries at Hebburn-on-Tyne (and later at Woolwich were placed on the "permitted list". In 1921, the "Safety in Research Board" was created and is still in existence (Ref 21, pp 552-57; Ref 23, p 347 and Ref 49, pp 10-17)

Recently the design of new "permitted" expls has been considerably influenced by the application of the hydrodynamic theory of detonation to mixts of HE with inert solids (See Ref 34a)

Although many disastorous coal-mine explns took place in USA in the 19th century when mines went deeper underground, nothing of importance was done by private concerns for preventing such explns and the Govt did not interfere. This condition lasted until Dec 3, 1908 when the Govt station for testing "permissible" expls was established at Pittsburgh, Pennsylvania. This station was at first under the jurisdiction of the US Geological Survey, but was later taken over by the Department of Commerce when it established the Bureau of Mines (See Ref 23, p 347 and Vol 2, p B342-R of this Encycl). When the Bur of Mines Explosives Experiment Station was established at Bruceton, Pa, the tests for permissible expls started to be conducted there. Right from the beginning many expls were submitted for testing but only a few passed the tests. This was because most of the submitted mixts were compounded without any consideration to oxygen balance, heat of expln (or deflagration) or max temp developed on expln. In

later years, the practice of calcg the thermochemical data of expls, before submitting them to permissibility tests, was well established and the number of rejections considerably dropped

Because the method of thermochemical calcns given in Ref 10a, p 106-14 became obsolete, the US Bureau of Mines published in 1941 & 1942 Technical Papers **632** & **642** compiled by F. W. Brown. The first paper deals with temperatures of explosion & gaseous products and the effects of changes in carbonaceous material, while the 2nd paper deals with pressures of explosion.

More recent data on the thermochemistry of expls is found in Ref 34a, pp 40-64 and Refs 54, 57 & 59

It seems that one of the simplest methods of calcg "permissibility" of expls is given in Ref 57, pp 108-15

It is known that ignition and expln of dangerous coal mine atmosphere depends on many factors, such as compn of mine atm, wt of charge of expl, induction period (time elapsed betw expln of charge and ignition of mine atm), temp of products of expln, presence of hot solid particles in products of expln, duration of flame, heating of mine atm due to adiabatic compression created by incident and reflected shock waves, etc. It may be assumed that most of these factors contribute to the ignition of coal mine atmosphere. Although formulas for calcg these factors exist, it is sufficient in most cases to calc only the factors which are considered as the most responsible for ignition and expln of mine atm

The method of calcn described in Ref 57, pp 111-14 determines the permissibility of several explosives, of which the following are typical examples: *Example 1.* To det if *Ammosol of Rudakovskii* (TNT 12.0, AN 56 & NaCl 32%) is safe for use in gaseous and/or dusty coal mine:

One kg of this mixt contains in moles: 120:227=0.5286 of TNT; 560:80=7.0 of AN and 320:58.5=5.47 of NaCl. This gives the equation of expln as:

$0.5286\ H_3C.C_6H_2(NO_2)_3 + 7.0NH_4NO_3 + 5.47\ NaCl = 3.7CO_2 + 15.32H_2O + 5.47\ NaCl + 7.79N_2 + 0.72\ O_2$

Oxygen balance to CO_2, H_2O & N_2 = $\frac{0.72\times32\times100}{1000}$ = + 2.3%

Number of moles (n) of gaseous products of expln = 3.7+15.32+7.79+0.72 = 27.53

Heat of expln of *Ammosol* would be equal to sum of heats of formation of products of explosion, plus compensation for gas formed on expln (which is equal to 27.53 × 0.57 kcal/kg), minus sum of heats of formation of TNT & AN. Sodium chloride is not included in the calcn since it does not decomp during expln

Q_V = 3.7×94.5+15.32×57.7+17.53×0.57-0.5286×13-7.0×88.1=349.65+883.964+15.7-6.87-616.7 = =1249.314-623.57=625.47kcal/kg

Molar heat capacities at const vol (C_V), of individual products of expln are assumed to be equal to a+bt, where a is the molar heat capacity at 0°C, b- the increment of the mean molar heat capacity of gas for each 1°C and t - calcd temp of expln

This gives for products of expln:

CO_2 (9.0+0.000585t)×3.7 = 33.3 + 0.002146t

H_2O (4.0+0.00215t)× 15.32 = 61.28 + 0.032938t

N_2+O_2 (4.8 +0.00045t)× 8.51 = 40.848 + 0.003829t

NaCl (12.0 + 0.0t)× 5.47 = 66.64 + 0.0t

ΣC_V = 202.07 + 0.0389t, where 202.07 is the sum of a's for individual gases and 0.0389, the sum of b's

The temp of expln, t, can be calcd from the formula:

$$t = \frac{-a + \sqrt{a^2 + 4bQ_V} \times 1000}{2b} =$$

$$\frac{-202.07 + \sqrt{(202.07)^2 + 4\times0.038913\times625740}}{2\times0.038193} =$$

= 2180°C

As this temp lies below 2200°, the expl is considered to be "permissible" from this point of view. But there are other factors to be considered

The next step is to calc the amt of heat absorbed by NaCl, using the formula:

$Q_{NaCl} = [13.33\times 800 + 5670 + 15.92\ (t-800)]-$

×m kcal/kg, where 13.33 is molar heat capacity of solid NaCl (up to 800°), 5670 its molar latent heat of fusion, 15.92 molar heat capacity of molten NaCl and m moles

of NaCl per 1kg of expl. On substituting 2180 for t and 5.47 for m, we obtain 209.523 kcal/kg. By subtracting this value from Q_v we obtain: 625.74-209.532=416.217 kcal/kg

By dividing the last value of n=27.53, which represents number of moles of permanent gaseous products of expln, we obtain 15.12kcal/mole, the quantity of heat contained in 1 mole of gases. This value is designated as q. The next step is to det the value h which is the smallest amt of heat required to obtain the temp of 650°(ignition temp of firedamp) on mixing 1 gram-mole of gaseous products of expln with mine atm surrounding the charge. This value can be calcd from the exptl equation: h=21.5-94v, which is applicable for chges giving on expln in coal layers: CO_2, H_2O, N_2 & O_2. Here v is equal to the ratio moles of free oxygen (0.72) to total moles of gases of expln. This gives:

$h = 21.5 - \frac{94 \times 0.72}{27.53} = 19.04$kcal/mole. If the same expl would be used for blasting rock in gaseous and/or dusty coal mine, then $h = 21.5 - 75v = 21.5 - \frac{75 \times 0.72}{27.53} = 19.55$

If the value (h-q), known in Russia as "pokazatel' antigrisutnosti VV" (index of antigrisoutness of explosives) is positive, the expl is considered "permissible" and if it is negative the expl is "nonpermissible"

In our case h-q=19.04-15.12=+3.92 kcal/mole or 19.55-15.12=+4.43kcal/mole and the expl might be considered as permissible

Maximum charge (charge limite) of this or other expls is usually detd experimentally either in a gallery or an experimental coal mine, as described under COAL MINING EXPLOSIVES, TESTING FOR PERMISSIBILITY. These tests verify also the theoretical finding of permissibility of the expl to test

Example 2. Using the above described method Bandurin & Rukin (Ref 57, p 115), calcd for the French expl consisting of AN 49.5, NG 13.5, woodmeal 8.5 & NaCl 28.5%, the "index" h-q as equal to +1.4kcal/mole and the temp of expln equal to 2374°. This expl passes, accdg to Audibert, the gallery tests for "antigrisoutness", as required by the Commission des Substances Explosives, although its calcd temp of explosion is much higher than allowed for permissible expls in France

Example 3. Let us suppose, that an expl contg 20% NaCl (other ingredients being AN, TNT & pine bark flour) meets the requirement of q being smaller than h, but has the calcd temp of expln equal to 2286°, which is considered too high by Russian standards

The simplest method to lower the temp of expln is to incorporate in the formulation an addnl amt of an inert salt, such as NaCl, KCl, etc. In Russia they usually add NaCl and the number of moles (M) of this salt which are necessary to add to the explosive is calcd from the equation:

$$M = \frac{Qv - bt^2 - at}{12t}, \text{ where}$$

Qv= the heat of expln of the mixt before adding extra amt of NaCl; t=the desired temp of expln; a=the sum of molar heat capacities of individual gases of expln at 0°C and b= the sum of increments of the mean molar heat capacities of gases for each 1°C

If it is desired to lower the temp of expln to 2000°C, the value of M can be calcd by inserting the calcd numerical values (given in Ref 57, p 112) for Qv, a and b in the above equation:

$$M = \frac{658920 - 0.043471 \times (2000)^2 - 188.783 \times 2000}{12 \times 2000}$$

$$= \frac{263966}{24000} = 10.99 \text{ moles of NaCl}$$

It was already mentioned that firedamp can be ignited not only by flames evolved on expln of blasting charges, but also by other factors

Let us examine some of the problems created by these factors:

Problem 1: Will the firedamp of initial temp 27°C and ignition point of 650°C be ignited by adiabatic compression to 20 atm, created by an incident shock wave?

The temp T_2(in °K) to which the gas will be heated, by compression, to p_2 = 20atm can be calcd from the equation:

$T_2 = T_1 \times \left(\frac{p_2}{p_1}\right)^{(k-1)/k}$, where T_1 - the initial temp in °K = 27 + 273 = 300°; p_1 - the initial pressure = 1atm; p_2 - the final pressure = 20 atm; and k - the ratio of the heat capacity of firedamp at const pressure to that at const volume

On inserting the numerical values in the above equation we obtain:

$$T_2 = 300 \times \left(\frac{20}{1}\right)^{(1.4-1)/1.4}$$

$$= 300 \times 20^{0.3} = 737°K = 464°C$$

As this temp is lower than ignition temp for firedamp, no ignition would take place (Ref 57, p 110)

Problem 2: Will the reflected shock wave travelling with velocity 2000m/sec and creating pressure of 40atm ignite the firedamp previously heated to 737°K by incident shock wave?

The temp, T_3, to which the gas would be heated by compression to p_3 = 40atm, can be calcd from the equation:

$$T_3 = T_2\left(\frac{p_3}{p_1}\right)^{(k-1)/k} = 737 \times \left(\frac{40}{1}\right)^{(1.4-4)/1.4}$$

$$= 2226\ °K = 1953°C$$

where T_2 = 737°K; p_1 = 1 atm; and k is the ratio of the heat capacity of firedamp at const pressure to that at constant volume

Note: The above calcn is the same as given in Ref 57, p 110. Mr. J. Alster of PicArsn pointed out, however, that the pressure of firedamp before it was hit by reflected shock wave of pressure p_3 = 40 atm was 20 atm (p_2) and not one atm (p_1). In this case the equation for calcn of T_3 should be written as:

$$T_3 = T_2\left(\frac{p_3}{p_2}\right)^{(k-1)/k} = 737 \times \left(\frac{40}{20}\right)^{1.4-1/1.4}$$

$$= 737 \times 2^{0.3} = 907°K = 634°C$$

As this temp is lower than the ignition point of firedamp (650°C), no expln of gas would take place.

If we assume, however, that the temp T_3 is 2226°K, as calcd in Ref 57, p 110, then the gas would ignite if the period of contact, τ_{cont}, of firedamp with the compression zone is longer than the period of induction, τ_{ind} (the time elapsing betw expln of charge and ignition of firedamp)

The period of induction can be calcd from the equation:

$\tau_{ind} = C \times e^{\gamma/T_3} \times p_3^n$,

where C is the constant equal to 10^{-12}; e is the base of natural logarithms, equal to 2.718; γ = R/E, where R is the gas constant and E is energy of activation; γ for firedamp given in Ref 57, p 110 is 30000 cals; n is the constant equal to -1.8

On substituting these values in the above equation we obtain:

$\tau_{ind} = 10^{-12} \times 2.718^{(30000/2226)} \times 40^{-1.8} \cong 10^{-9}$ sec

Period of contact of firedamp with compression zone may be cald from the equation:

$$\tau_{cont} = \frac{\text{Width of compression zone}}{\text{Velocity of reflected shock wave}}$$

If the width of zone is 4×10^{-3}m and velocity of wave 2×10^{-3}m, then

$\tau_{cont} = 4 \times 10^{-3} / 2 \times 10^{-3} = 0.5 \times 10^{-6}$

As τ_{cont} is longer than τ_{ind}, the firedamp would ignite (Ref 57, pp 110-11)

Problem 3: Will the hot solid particles, ca 1×10^{-3} m long, travelling with velocity of 2500m/sec, ignite the firedamp, if their temp is 2000°K?

For solving this problem, it is necessary to calc τ_{ind} as in previous problem and then the period of contact, τ_{cont}:

$\tau_{ind} = 10^{-12} \times 2.718^{(3000/2000)} \times 1^{-1.8}$

$= 0.55 \times 10^{-6}$ sec and

$$\tau_{cont} = \frac{\text{Length of particles}}{\text{Velocity of their travel}} = \frac{1 \times 10^{-3}}{2.5 \times 10^{3}}$$

$= 0.4 \times 10^{-6}$ sec

As both values are of the same order, there is a probability of ignition of firedamp by the hot solid particles (The portion dealing with calculation for "permissibility" of explosives was review by Mr. Jack Alster of PicArsn)

A very important improvement in the field

of permissible expls was done in 1914 by Lemaire of Belgium, who enclosed cartridges of explosives, already accepted as safe from the basis of gallery tests, by a mantle of inert safety material, such as Ca fluoride. These expls, named **sheathed explosives or sheathed charges** proved to be so safe that many countries started to adopt them (Ref 34a, p 19; Ref 49, pp 30-1)

The action of safety sheaths has been widely investigated and it seems probable that the finely dispersed material of the sheath functions as a flame trap in the firedamp mixt, and prevents propagation of any flame or ignition (Ref 34a, p 22)

There are also **core explosives or core charges** patented by Lawrence (Ref 33b). They are prepd by placing inside a cartridge of AN dynamite, a "core", either hollow or filled with an inert material. As the presence of core causes the reduction of brisance and of detonation rate, it renders the chge safe for use in firey mines **Belgian Permissible Explosives**, known as *SGP* (Sécurité-Grisou-Poussière), sheathed or non-sheathed (gainés ou non gainés in Fr) are listed in Vol 2, p B29 of this Encycl **British Permitted Explosives.** Accdg to Taylor (Ref 34a, p 19), the current expls are subdivided into two main types:
a)Explosives based on NG with AN & woodmeal as energy - and gas-producing ingredients and NaCl as safety ingredient
b)Explosives based on TNT & AN as energy producers and NaCl or KCl as safety ingredients

A few special low density expls in which there is some Na nitrate, have also been developed for producing lump coal

The previously mentioned "sheathed explosives" were introduced in GtBritain in 1934, after many sheathing materials were tried and rejected. Finally it was decided to use a sodium bicarbonate (and in some cases NaCl) sheath (Ref 49, pp 30-1)

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 the borehole which is too tight). If a sheath is damaged, the cartridge cannot be used in fiery mines. In order to have expls which are safe under any circumstances it was decided to incorporate uniformly the material used for sheath in the explosive, itself. These expls are designated as **EqS**, because they are "equivalent" in safety to sheathed explosives. These EqS expls proved to be such a success that they represent now about 50% of all permitted expls used in GtBritain (Ref 30) (See also Ref 49, pp 31 & 86)

Typical British permitted explosives which are "sheathed" with Na bicarbonate or Na chloride, as well as some corresponding EqS explosives are listed in Ref 34a, pp 20-1; Ref 49, pp 103-05 and in the Table given in this section

French Permissible Explosives. Considerable work on development of permissible expls was done in France before WWII, but it was interrupted during WWII by Ger occupation. Accdg to Médard (Ref 31a, p 219), about 25% of expls used in France (at the time of publication of his paper) were permissible (*explosifs antigrisouteux).* Following are compns of some of these expls developed before and after WWII:
Grisou-dynamite chlorurée n° 1 (1932) - AN 55.5, NG 20.0, NC 0.5, woodflour 2.5 & NaCl 21.5%; "coefficient d'utilisation pratique" (CUP) 77 (PA 100) (See Vol 1, p IX of this Encycl); "charge limite" 300g (See Vol 2, p C151-R of this Encycl) (Ref 31a, p 220)
Explosif du type N, n° 7 (of Burlot & Schwob) (1933) - AN 76, DNN 7, woodflour 2 & NaCl 15%; CUP 87, charge limite 300g (Ref p 219)
Explosif du type N n° 9 (of Audibert) (1938) - AN 48.2, TNT 9.15, woodflour 1.65 & NaCl 41.0% (Ref 31a, p 220)
Since the above expls were not considered entirely safe against firedamp, a search for new formulations for "explosifs antigrisouteux" was undertaken before WWII, but was not completed for the reason explained above

Following are examples of Fr expls developed since WWII:
Grisou-dynamite chlorurée n° 14 (1948) - AN 10.0, NG 19.5, NC 0.5, woodflour 1.5 & NaCl 68.5%; CUP 32 (Ref 31a, p 220)

Grisou-dynamite chlorurée n° 15 (1949) - AN 20.0, NG 19.5, NC 0.5, woodflour 2.0 & NaCl 58%; CUP 41 (Ref 31a, p 220)
Explosif du type N n° 62 (of Médard) (1949) - AN 12, PETN 23 & NaCl 65%; CUP 38. This expl gave in tests most favorable results - charges as high as 1500g did not ignite firedamp and/or coal dust (Ref 31a, p 221 & Ref 62b, p 272)

Among the permissible expls developed after WWII in the labs of the CSE (Commission des Substances Explosives), the following are listed by Médard:
Explosif 123-CSE-1948 - AN 17, PETN 23 & NaCl 60%; CUP 43 (Ref 31a, p 222)
Explosif 136 - CSE - 1948 - AN 22, PETN 20, TNT 3 & NaCl 55%; CUP 46 (Ref 31a, p 222)
Explosif 68 - CSE - 1949 - AN 20, PETN 20, DNN 1.5 & NaCl 58.5%; CUP 43 (Ref 31a, p 222)

German Permissible Explosives. Many successful expls were developed in Germany before and after WWII. Some of the expls used before and during WWII are listed in Ref 53, pp Ger 260-261 under *Wettersprengstoffe.* They are also known as *Wettersicheresprengstoffe.* An interesting type of Ger permissible expl was developed by the Westfälisch-Anhaltische Sprengstoff Aktiengesellschaft (WASAG) under the the name of *Bikarbit.* It contains a large amt of Na bicarbonate (with/or without Na chloride) and a small amt of NG. Compns and props of these Bikarbits are given in Ref 53, pp Ger 11-12 (See also Ref 20, pp 145-46 and Vol 1, p B111-R of this Encycl)

Italian Permissible Explosives (Esplosivi antigrisoutosi; esplosivi di sicurezza or grisoutini in Ital). The following are described in the literature and catalogs:
Ammongelatina: NG (with collod cotton) 20, AN 75 & woodflour 5%
Dinamite a base attiva: NG 20-25, AN 20-25, Na nitrate 30-35 & charcoal 20%
Dinamite a base inerte: NG 35-40, kieselguhr 10-14 & $MgSO_4.7H_2O$ 32-50%
Esplosivi da mina polverulenti con nitroglicerina (Powdery mining explosives with NG): a)NG 14, collod cotton 0.3, AN 65.7 & NaCl 20%; Trauzl 2700cc, gap test 2cm & deton rate 2120m/sec b)NG 11, woodflour 6, AN 51, Na nitrate 10 & NaCl 22% c)NG 4, DNT 1.5, collod cotton 1.5, AN 82 & NaCl 11% d)NG 10, collod cotton 0.3, DNT 4, AN 65.7 & NaCl 20% e)NG 12.7, collod cotton 0.3, AN 79 & DNN 8% f)NG 12.76, collod cotton 0.24, AN 83 & DNN 4% (Ref 35, pp 282-83)
Esplosivi da mina polverulenti senza nitroglicerina (Powdery mining explosives without NG): a)AN 85.5, TNT 4.5 & woodflour 10.0%; Trauzl 330cc, gap 1cm & detonation rate 1900m/sec b)AN 80, Na nitrate 10, DNN 5 & Ca silicide 5% c)AN 84.5, DNN 10, K dichromate 4.5 & powdered carbon 1.0% (Ref 35, p 284)
Esplosivi gelatinosi al binitrotoluolo (Gelatinized explosives with DNT): a)DNT 16, Na nitrate 15, collod cotton 0.5, AN 45, TNT 7 & K perchlorate 16.5% b)DNT 20, Na nitrate 14.5, collod cotton 1, AN 50.5, TNT 7 & PETN 7% (Ref 35, p 286)
Grisoutina C, manufd by the Nobel di Avigliana contained: NG 13.20, collod cotton 0.25, AN 86.35, fossil flour 0.1 & Ca carbonate 0.1% (Ref 58, p 375)
Grisoutina al 13.20%. A similar expl manufd by Montecatini; its strength is 25% of Blasting Gelatin; detonation rate 3500 -3700m/sec and density 0.1 (Ref 68, p 16). Vettori (Ref 73) gives the following props: d 1.05, Trauzl 330cc, deton rate 3800m/sec, gap test 2.9cm, gas volume at 20° & 946mm l/kg, temp of expln 1450°C, and specific pressure 615.3 atm/kg
Grisoutina al 10%. Vettori (Ref 73) gives its compn as NG 10, collod cotton 0.25 & AN 89.75% and properties: d 1.05, Trauzl 305cc, deton rate 2038m/sec, gap test 2.0cm, gas vol at 20° & 760mm pressure 1021 l/kg, temp of expln 1450°C, and specific pressure 6378 atm/kg
(See also Ref 35, p 282). Montecatini's catalog (Ref 68, p 16) gives strength 22% of Blasting Gelatin, deton velocity 3100-3300m/sec and d 1.05
Sheathed Explosives (Esplosive con guaina in Ital). Accdg to Dr. Camillo Belgrano, as communicated in July 1964 to Dr. Omero Vettori (Ref 72), Ital sheaths consist of mixts of Ca fluoride & sulfate with Na bicarbonate. Sheathed expls

British Permitted Explosives (Sheathed and EqS)

Explosive	Physical Form	NG+ NGc	NC	MNT	TNT	NH_4NO_3	$NaNO_3$
Douglas Powder (Sheathed	Powdery	-	-	-	14.0 -16.0	67.5 -70.5	-
Unirend (EqS)	Powder	-	-	-	10.7 - 12.7	49.1 - 52.1	-
Polar Ajax (Sheathed)	Plastic	25.5 -27.5	0.25 - 1.25	0.8 - 2.8	-	39.0 - 42.0	-
Unigex (Initial)(EqS)	?	15.7 -17.7	0.1 -1.1	-	-	-	43.5 -46.5
Unigex (Modified (EqS)	?	16.0 -18.0	-	-	-	42.4 -75.4	-
Unigel (EqS)	?	26.3 - 28.3	0.4 - 1.4	0.2 - 2.2	-	29.5 - 32.5	-
Polar Dynobel Nc 2 (Sheathed)	Cohesive powder	14.0 - 16.0	0.1 -1.1	0.2 -2.2	-	61.0 -64.0	-
Unibel (EqS)	?	10.4 -12.4	0.1 -1.1	0.3 -1.3	-	47.0 - 50.0	-
Polar Viking (Sheathed)	Granular	9.5 -11.5	-	-	-	69.2 - 72.2	-
Unifrax (EqS)	?	11.2 - 13.2	-	-	-	48.4 - 51.4	-
A1 Rounkol (Sheathed)	Granular	9.0 -11.0	-	-	-	54.0 -57.0	9.0 -11.0
Unikol (EqS)	?	9.0 -11.0	-	-	-	35.1 -37.1	12.5 -14.5
Polar Thames (Sheathed)	Granular	9.0 - 11.0	-	-	-	59.5 - 62.5	-
Denaby Powder No 2 (Sheathed)	Powder	-	-	-	11.0 - 13.0	57.5 - 60.5	5.0 -7.0
Gathurst Powder (Sheathed)	Powder	-	-	-	16.5 -18.5	49.0 -52.0	5.0 - 7.0

Abbreviations and notes: EqS - Equivalent in safety to sheathed expls; NG - Nitroglycerine; NGc - such as woodmeal, wheat flour, oat husk meal or plant fibers; Other ingredients, such as AM (Acid Barytes, Resin or AS No 2(?); d - density of unsheathed cartridges; Strength as % of BG (Blasting cartridge suspended in 9% methane/air

British Permitted Explosives (Sheathed and EqS)

NaCL	NH_4CL	China Clay	Carbo-naceous Material	Other Ingredients	d, g/cm^3	Strength % BG	Charge Limit oz
15.0 - 17.0	-	-	-	0.01 - 0.05 (AM) 0.001 -0.01 (ASNo2)	1.15	66 (43)	5
36.2 -39.2	-	-	-	0.001 - 0.1 (AM) 0.001 - 0.01 (ASNo2)	1.2	42	5
23.5 - 25.5	-	1.5 - 3.5	2.5 - 4.5	0.001 - 0.05 (AM) 0.001 - 0.01 (ASNo2)	1.55	56 (43)	10
-	28.0 -30.0	3.0 - 5.0	1.5 - 4.5	0.5 - 1.5 (NaCMC) 0.1 - 1.1 (Ca stearate)	1.3	42	6
25.7 -27.7	-	2.7 -4.7	2.2 -5.2	0.005 - 0.5 (AM) 0.001 - 0.01 (ASNo2) 2.7 - 4.7 (Ca sulfate) 0.1 - 1.1 (NaCMC) 0.1 - 1.1 (CA stearate)	1.3	44.5	6
28.3 - 30.5	-	3.3 - 5.3	0.4 - 2.4	0.1 - 1.1 (DAmmPh) 0.001 - 0.1 (AM) 0.001 - 0.01 (ASNo2) 3.3 - 5.3 (Barytes)	1.7	44	?
14.2 - 16.2	-	0.25 - 1.25	4.0 - 6.0	0.01 - 0.05 (AM) 0.001 - 0.01 (ASNo2)	1.12	62 (43)	8-12
33.9 - 36.9	-	-	2.8 - 4.8	0.01 - 0.05 (AM) 0.001 - 0.01 (ASNo2)	1.15	40.5	24
9.0 - 11.0	0.1 - 1.1	-	7.8 -9.8	0.01 - 0.05 (AM)	0.98	66 (47)	8
29.0 - 32.0	-	-	6.2 - 8.2 (Plant fiber)	0.1 - 1.1 (DAmmPh) 0.1 - 1.1 (Resin) 0.05 - 0.05 (AM) 0.001 - 0.01 (ASNo2)	0.8	46	8
11.0 -13.0	-	-	10.5 - 12.5 (Plant fiber)	0.0 - 0.5 (DAmmPh) 0.0 - 0.5 (Resin) 0.005 - 0.05 (AM) 0.001 - 0.01 (ASNo2)	0.8	46	8
28.9 - 31.9	-	-	9.0 - 11.0 (Plant fiber)	0.0 - 0.5 (DAmmPh) 0.0 - 0.5 (Resin) 0.05 - 0.1 (AM)	0.8	39	8
18.0 - 20.0	-	0.1 - 1.1	7.5 - 9.5	0.1 - 1.1 (?)	0.98	56	?
19.0 - 21.0	-	-	1.5 - 3.5	-	0.95	58 (?)	?
24.0 - 26.0	-	-	-	-	1.10	53 (?)	?

Nitroglycol; NC - Nitrocellulose; MNT - Mononitrotoluene; TNT - Trinitrotoluene; Carbonaceous Materials, magenta), DAmmPh(Diammonium phosphate), NaCMC (Sodium carboxymethyl cellulose), Ca sulfate, Ca stearate, Gelatin) for unsheathed cartridges and in brackets for sheathed; Charge limit in ounces - no ignition of gas by a

have been used in Sicilian sulfer mines
Tionite. This expl is manufd by Montecatinni SGIMC for use in Sicilian sulfur mines. Its props are similar to those of permissible coal mining expls. It would not ignite sulfur dust or other combustible dust (Ref 68, p 17)

Japanese Permissible Explosives: The following expls are described in the literature and in reports:

Gelatin-dynamite: NG 28, collod cotton 1.2, AN 43.3, starch 1.0, urea 20 & soft wax 0.5% (Ref 58, p 373)

Shoanbakuyaka: AN 79, DNN 10, sawdust 1 & NaCl 10% (Ref 24a, p 29)

Shoanyaku. A series of coal mining expls manufd at the Uji Factory, Kyoto Prefecture and described in PB Rept **50394** (1946) pp 36-8:

No 1: AN 70, DNN 9, woodmeal 1 & NaCl 20%
No 2: - same as Shoanbakuyaku
No 5: AN 64, TNT 12, woodmeal 3, wheat starch 1 & NaCl 20%
No 6: AN 71, TNT 12, woodmeal 3, wheat starch 1 & NaCl 13%
No 7: AN 75, DNN 9, TNT 1.5, woodmeal 1.5 & NaCl 13%
"Special": AN 64, DNN 3, TNT 7, woodmeal 2, GuN 2, Na nitrate 2 & NaCl 20%

Russian Permissible Explosives (Predokhranitel'nyiye ili Antigrisutnyiye Vzryvchatyiye Veshchestva, in Rus): *Ammonity.* Gorst (Ref 46, p 105) lists Predokhranitel'nyi Ammonit contg AN 68, TNT 10, pine bark (or oil cake) flour 2 & NaCl 20%; Yaremenko & Svetlov (Ref 47, p 162) give props, but no compns of Ammonit No 8 (ugol'nyi), Ammonit PJV-20 (ugol'nyi), Ammonit AP-1 (porodnyi), Ammonit P-2 (porodnyi) Ammonit AP-4JV (porodnyi) and Ammonit AP-5JB (porodnyi); of these, the ones marked "ugol'nyi" are permitted for blasting coal and the ones marked "porodnyi" are permitted for blasting rock in any kind of coal mines. Vaskovskii (Ref 48, p 25) lists, w/o giving compns, Ammonits No 8 & PU-2 as permitted for blasting coal. Andreev & Belyaev (Ref 59, p 511) give compns of Ammonit III/1 (AN 56, TNT 9, woodmeal 3 & NaCl 32%); Ammonit III/2 (AN 70, NG 6, DNT 2, woodmeal 3 & NaCl 19%) and Ammonit PJV-20 (AN 64, TNT 16 & NaCl 20%) (See also Ammonits in Ref 45a, p Rus 1 and Vol 1 of Encycl pp A308-R & A309-A310)

Ammosol'. A permissible expl proposed by C.I.Rudakovskii (listed in Ref 57, p113): AN 56, TNT 12, & NaCl 37%; OB to CO_2 + 2.3 heat of expln 625kcal/kg & temp of expln 2180°C (Calcn of its "index of permissibility" is given in this section)

Dinamity predokhranitel'nyiye, such as Grisutin plastovoy (or ugol'nyi) - AN 88 & NG (slightly gelatinized with NC) 12% and Grisutin porodnyi- AN 70-71 & NG (gelatinized with NC) 30-29% (Ref 48, p 510)

Pobedity are, accdg to Vaskovskii (Ref 48, p 30), Ammonity, to which 8-10% NG is added. He also lists on p 25, w/o giving compns, the following: "Pobedit PU-2" and "Pobedit No 8 v predokharanitel'noy obolochke" (Sheathed Pobedit) as permissible

Porodnyiye predokhranitel'nyiye Ammonity. See under item A

Sostavy Favié (Compositions Favier) - AN 80-95 & DNN (or DNB or TNN) 20-5% (Ref 59, p 510)

Ugol'nyiye Ammonity. See under item A

Russian "sheathed" expls, known as *VV v predokhranitel'noy obolochke* using "inert sheaths" ("inertnyiye obolochki"), such those consisting of Na bicarbonate with or w/o NaCl and "active sheaths" (aktivnyiye obolochki) which consist of Na bicarbonate with ca 10% of NG. The active sheaths have advantages because they are pulverized completely on expln of enclosed cartridge, thus filling the mine atmosphere uniformly with non-combustible dust (Ref 59, p 514)

Spanish Permissible Explosives (Explosivos antigrisú or Explosivos de seguridad contra el grisú, in Span:)

Antigrisú (Explosivos): a)Número 1 - AN 80.6, DNN 6.4 & Amm chloride 13.0% b)Numero 2 - AN 80.9, DNN 11.7 & Amm chloride 7.4% and Número 3 - AN 82.0, TNN 5.0 & Amm chloride 13.0% (Ref 25, p 379) [Comp with Antigrisou (Explosifs) in Vol 1, p A466 of this Encycl]

Astralita antigrisutosa AN 57, NG 12, sawdust 2, coal dust 2 & NaCl 27% (Ref 25, p 380) (Comp with Astralits in Vol 1, p A497-R of this Encycl)
Explosivos de seguridad de la Fábrica de Dinamita de Galdácano: a)Número 2 AN 70.0, NG 29.1 & NC 0.9% b)Número 5 Na nitrate 34.0, NG 25.0, sawdust & flour 39.5, Ba nitrate 1.0 & Na carbonate 0.5% c)Número 7 AN 88.00, NG 11.76 & NC 0.24% (Ref 25, p 378)
Explosivo núm 3 Na nitrate 64.0, AN 17.5 & MNN 13.5% (Ref 25, p 379)
Grisúnaftalita AN 90, MNN 5 & K nitrate 5% (Ref 36, p 119)
Grisutitas capa: a)AN 95.5 & TNN 4.5% and b)AN 83.00, NG 6.00, NGc 1.50, collodion cotton 0.25, Tetryl 4.25, K nitrate 4.50 & Na carbonate 0.50% (Ref 25, p 379)
Grisutitas roca: a)AN 91.5 & TNN 8.5% and b)AN 69.00, NG 12.00, NGc 3.00, Collodion cotton 0.50, Tetryl 10.00, K nitrate 5.00 & Na carbonate 0.50% (Ref 25, p 379)
Nitramita de la Fábrica de Galdácano AN 88.0 & DNN 12.0% (Ref 25, p 378)
Nobelita antigrisú gelatinizada AN 26.5, NG (gelatinized) 30.0, sawdust 0.5, NaCl 40.0 & Ca nitrate (50% soln) 3.0% (Ref 36, p 119)
Sheathed Cartridges, introduced in 1934, called "cartuchos con corteza", consist of a cartridge of a permissible expl, 25mm in diam, completely surrounded by a 3.5mm thick "sheath" (corteza de seguridad), consisting of Na bicarbonate mixed with 15 to 18% NG (Ref 36, p 119)

Swedish Permissible Explosives. Our information is limited to one explosive only: *Nobelit:* NG 28,7, collod cotton 0.7, AN 39.7, dextrin 2.5, potato flour 10.0, woodmeal 1.0, vegetable oil 0.5 & NaCl 17.6% (Ref 14, p 407)

Swiss Permissible Explosives. It does not seem that these expls have ever been manufd in Switzerland. Accdg to Dr A. Stettbacher (private communication dated 9 July 1954), the so-called "Sicherheitssprengstoffe" manufd in Switzerland are safe to handle and transport, but not against firedamp and/or coal dust, and there are no Testing Galleries in Switzerland for permissible expls

United States Permissible Explosives. Accdg to Davis (Ref 23, p 348), permissible expls may be divided into two principal classes: a)Those based on an explosive which is cool in itself, such as AN, GuN or NGu and b)Those based on a HE which is not cool, such as NG, NS, TNT, DNB, DNN, TNN, TeNN, etc.

In order to lower the flame temp of these expls, salts, like NaCl, KCl, Amm chloride, alums, blue vitriol, hydrated Amm oxalate, Glauber's salt, gypsum, borax, Epsom salt, hydrated Al sulfate, etc, may be incorporated (Ref 23, pp 352-53)

Permissible expls include also *sheathed explosives* and *core explosives,* briefly described in this Section II before *Belgian Permissible Explosives*

Many US expls which might pass the tests required for permissibility are described in Vol 1 of this Encycl under Ammonium Nitrate Blasting Explosives, pp A342-A345; Ammonium Nitrate Dynamite, p A355 & Ammonium Nitrate Gelatin, p A367. Some expls not based on AN, but suitable as permissibles are listed, without separating them from nonpermissibles, in Vol 2, under BLASTING EXPLOSIVES, pp B202 to B211

No expls can be used in US gaseous and/or dusty coal mines unless they pass the required tests and are placed on the current "Active List of Permissible Explosives and Blasting Devices Approved Before February 28, 1961" (Ref 64). This list includes 124 different brands. Fifteen of these are gelatinous, and the rest are of the more commonly used granular AN type. Their detonation rates range from 1300 to 5600m/sec, and the weights of 1¼x8" cartridges range from 86 to 244g. The list of "permissible blasting devices" includes nine **Cardox**, one **Cobra** and two **Hydrox** models (See under Commercial Blasting Devices)

The expls placed on the US active permissible list are tested as prescribed by Schedule 1-H and blasting devices as prescribed by Schedule 26A (See Refs 43 & 35 under COAL MINING EXPLOSIVES, TESTING FOR PERMISSIBILITY)

US permissible *nongelatinous* expls are listed in Table 1 of Ref 64, without revealing their compns. These expls are suitable

for use in mines which are relatively dry. Their deton rates range from 1300 to 3400m/sec

US permissible *gelatinous* expls are listed in Table 2 of Ref 64, without giving their compns, They are designed especially for blasting rock in coal mines, although under certain conditions, they have proved suitable for blasting coal. They are better adaptable for use in wet mines. Their deton rates range from 3200 to 5600m/sec

The following US firms manufacture permissible expls and blasting devices:

American Cyanamid Co, New York, NY; produces fourteen brands of nongelatinous expl American (Brands: 2^2, 3-A^2, 4-A^2, 5^3, 11^2, 12^2, 12-A^2, 14-A^3, 21^2, 22^2, 23^3, A^2 & B) and two gelatinous expls Permigel A & Permigel B^2

Apache Powder Co, Benson, Arizona; produces three brands of nongelatinous expl called Apache Coal Powder (grades A, B & H); no permissible gelatinous expls are produced

Atlas Chemical Industries, Wilmington, Delaware; produces the following nongelatinous expls: twenty brands of Coalite (brands: B, C, C-1, G, G-1, LL, LL-1, LS-1, M, M-1, MS, S, S-1, T, T-1, 5-S, 7-R, 8-R, & 8-S) and Peerless No 2; also two gelatinous expls: Gel-Coalite Z & Gel-Coalite No 3

Austin Powder Co, Cleveland, Ohio; produces the following nongelatinous expls: nine brands of Austin Red Diamond (brands: No 1, No 2, No 3, No 4, No 5, No 9-B, No 9-C, No 10-A & No 11) and one gelatinous expl Austin Red-D-Gel

Cardox Corp, Chicago, Illinois; produces blasting devices called Cardox (See a brief description in Vol 2, p C67-R of this Encycl)

E.I. du Pont de Nemours & Co, Wilmington, Delaware; produces the following nongelatinous expls: six brands of Duobel (brands: A^2, B^2, C^2, D^2, E^2 & F); EP-38, Lump Coal C^2, Lump Coal CC^2; and six brands of Monobel (Brands: A^2, AA^2, B^2, C^2, D^2 & E^2); also produces three brands of gelatinous expl Gelobel (brands: A, AA & C^2)

Hercules Powder Co, Wilmington, Delaware; produces the following nongelatinous expls: Bituminite D, Collier C, HP-47, HP-48 & six brands of Red (brands: HA^2, HB, HC, HD^2, HF & HL); also produces gelatinous expls Hercogel 2 & Hercogel A. The firm also is manufg blasting devices Cobra & Hydrox

Independent Explosives Co, Scranton, Pennsylvania; produces nine brands of nongelatinous expl Independent (brands: A, B, C, C-1, D, E, F, G & H), and one gelatinous expl Independent Gel-A

National Powder Company, Eldred, Pennsylvania; produces ten brands of nongelatinous expl National (brands: A-1, B, C, D, E, F, F-1, G & H) and one gelatinous expl Napcogel No 1

Olin-Mathieson Chemical Corp, East Alton, Illinois; produces eleven brands of nongelatinous expl Super-X (brands: No 2, No 2-A, No 3, No 3-A, No 5, No 7, No 8, No 9, No 11, Big Coal D & Big Red No 7-C); also produces gelatinous expls Super-X Gel, Super-X Gel A & Super-X Gel B

Trojan Powder Co, Allentown, Pennsylvania; produces seven brands of nongelatinous expl Trocol (brands: A, AA, B, BB, B-1, C & C-1); no permissible gelatinous expls are produced

West Coast Powder Co, Everett, Washington; produces one nongelatinous expl Wesco Coal Powder No 1 (See also COAL MINING EXPLOSIVES, TESTING FOR PERMISSIBILITY)

Refs: 1)Daniel (1902), 747-61 [Sûreté (Explosifs de)] 2)C.Hall & W.O.Snelling, US Geological Survey Bull **333** (1907) (Coal mining accidents, their causes and prevention) 3)V.Watteyne et al, Ibid, **369** (1908) (Prevention of mine explns) 4)R.T.Chamberlin, Ibid **383** (1909) (Notes on explosive mine gases & dusts) 5)G.S.Rice et al, Ibid **425** (1910) (Explosibility of coal dust) 6)J.J.Rutledge & C.Hall, USBurMines Bull **10** (1912) (Use of permissible expls) 7)C. Hall et al, Ibid **15** (1912) (Investigation of expls used in coal mines) 8)Marshall **1** (1917), 44-47 & **2** (1917), 582-606 (Permitted expls) 8a)Colver (1918), 247 & 681 (Nauckhoff's expls) 9)Barnett (1919), 117-42 (Safety coal mine expls) 10)E.Lemaire, AnnMines-Belg **23**, 649(1922) (Sheathed expls) 10a)C. A.Taylor & W.H.Rinkenbach, USBurMines Bull **219** (1923), 107-14 (The thermochemistry of expln) 11)H.Henkel, SS **22**, 173 (1927)

(Theorie und Praxis der Wettersprengstoffe) 12)P.Naoúm, ZAngChem **40**, 1351-59 (1927) (Wettersprengstoffe) 13)Naoúm, Expls (1927) 137-57 (Wettersprengstoffe) 14)Naoúm, NG (1928), 382-420 (NG expls permissible in gaseous & dusty coal mines) 14a)Molina (1930), 339 14b)A.Stettbacher, Angew-Chem **43**, 844-47 (1930) (Penthrinits) 14c)A. Stettbacher, SS **26**, 8-10 & 38-40 (1931) (Penthrinits) 15)Marshall, **3** (1932), 115-23 (AN expls) 16)Sukharevsky & Pershakoff (1932), 224-51 (Mechanism of firedamp and/or coal dust explns and development of expls safe for use in gaseous or dusty coal mines) 16a)G.St.Perrott & N.A.Tolch, "Liquid Oxygen Explosives", USBurMines Bull **349** (1932), 1-4 (Not suitable for work in gaseous and/or dusty coal mines, but suitable for use in strip mining) 17)Vennin, Burlot & Lécorché (1932), 228-31 (Explosifs antigrisouteux) 18)Stettbacher (1933), 242-52 (Die Schlagwettersicheren Sprengstoffe); 316 (Territ) 19)Pepin Lehalleur (1935), 401-22 (Emploi des explosifs dans les mines et les carrières) 20)Beyling & Drekopf (1936), 105-50 (Theorie der Wettersprengstoffe) 21)Thorpe **4** (1940), 552-57 (Coal mining expls) 21a)E.Piantanida, "Chimica degli Explosivi"; Tipo-Litografia della R. Accademia Navale, Livorno (1940) 22)CondChemDict (1942) (Coal carbonites) (Not listed in later editions) 23Davis (1943) 346-53 (Permissible expls) 24)Pérez Ara (1945), 250-64 (Explosivos de seguridad contra el grisú) 24a)OpNav (1945), 25-34 (Japanese expls) 25)Vivas, Feigenspan & Ladreda **2** (1946), 371-85 (Explosivos a base de nitrato amónico) 25a)Mangini (1947), 226-32 25b)All & EnExpls (1947), 107 & 152-53 26)A.I.Gol'binder & K.K.Andreev "Antigrisutnyiye Vzryvchatyiye Veshchestva" (Antifiredamp Explosives), Ugletekhizdat, Moskva (1947) 26a)B.Lewis & G. von Elbe, USBurMinesReptInvest **4382** (1948) (Ignition of firedamp by expls) 27)Stettbacher (1948), 82-94, 104, 114-& 149 28)A.I.Gol'binder, DoklAkadN **59**, 261-63 (1948) & CA **43**, 3199-3200 (1949) (Role of inert salts in compns of firedamp-proof expls) 28a)J.Taylor, Research (London) **1**, 69-72 (1948) (Advances in Brit permitted expls) 29)J.Taylor & O.A. Gurton, USP 2481795 (1949) & CA **44**, 840 (1950) (Permitted expls) 29a)C.Caprio, "Corso di Explosivi", Vol 2 ScuolaSalesiana del Libro,, Roma (1949) 30)J.Taylor, Trans-MiningEngrs (London), **109**, 358 (1949-50); W. Shepherd & H.Grimshaw Ibid **109**, 889 (1949-50) (Recent Brit expls equivalent in safety to sheathed expls) 31)J.Taylor & A.H. Phillips, USP 2513531 (1950) & CA **44**, 9150 (1950) (Sheathed coal mining expls) 31a)L.Médard, MP **32**, 209-24 (1950) (Recent progress and actual tendencies in the field of mining expls in France) 32)Kirk & Othmer **5** (1950), 975-76 (Coal mining safety expls) 33)J.Taylor & G.P.Sillitto, TransInstMining Engrs (London) **110**, 355-60 & 616-21 (1951); CA **45**, 8771 (1951) (Permitted expls Unigel & Unikol which are equivalent in safety to sheathed expls) 33a)Jiménez, Explosivos (1951), 23-32) 33b)R.W.Lawrence, USP 2565380 (1951) & CA **46**, 1768 (1952) (Core chrges) 34)H.Elsner (to DAG), GerP 803645 (1951); CA **46**, 1260 (1952) [Firedamp-proof detonators are prepd by incorporating inert ingredients (such as Na carbonate or bicarbonate, K bromide and wax) in amts 3 to 20% in both primary (such as LA 65 & LST 35%) and secondary charges (such as Tetryl, PETN, etc)] 34a)Taylor (1952), 17-22 (Permitted expls); 40-64 (Thermochemistry of expls) 35)Belgrano (1952) 165-68 (Esplosivi antigrisutosi) 35a)Blasters'-Hdb (1952), 79-81 (Chemecol); 66-70 (DuPont's permissible dynamites) 36)Stettbacher (1952), 112-14 (Explosivos de seguridad); 116-18 (Explosivos de chlorato y de perchlorato); 119 (Explosivos antigrusú) 37)H.Ahrens, Sprengtechnik **1952**, 101-07, 131-34 & 159-65; CA **47**, 12817 (1953) (Factors influencing the firedamp safety of permissible expls) 38)P. Speeckaert, Explosifs (Liège) **5**, 65-72 (1952); Chim & Ind (Paris) **68**, 749 (1952) & CA **48**, 11059 (1954) (The role of inhibiting salts in firedamp-proof expls) 39)Izzo, Minatore (1953), 51-9 (Esplosivi di sicurezza) 40)Weichelt (1953), 39 & 375 (Schlagwettersichere Sprengstoffe) 40a)W.E.Tourney & G.H.Damon Mechanization **17**, No 12, 99-102 (1953) (High expls for strip mining) 41)L.V. Dubnov, "Predokhranitel'nyiye Vzryvchatyiye Veshchestva" (Permissible Explosives),

Ugletetkhizdat, Moskva (1953) 42)T.Murata & N.Tomoishi, Kôgyô Kagaki Kyôkaishi (JIndExplsSoc of Japan) **14**, 84-7 (1953)& **15**, 22-30 (1954) (in Engl); CA **49**, 11284 (1955) (Effects of salts,talc & water on safety of expls, such as AN 83.7, NG 8.0, NC 0.3 & woodmeal 8.0%,against firedamp) 42a)N.E.Hanna et al, USBurMinesReptInvest **5049** (1954) (The mechanism of ignition of firedamp by explosives) 43)N.E.Hanna, et al, USBurMinesReptInvest **5051** (1954) & CA **48**, 7302 (1954) (Factors affecting the incendivity of permissible expls to firedamp) 44)Dynamit AG, GerP 903186 (1954) & CA **52**, 10582 (1958) (Permissible expls contg 10% of expl oil & 90% $NaHCO_3$-NaCl are not powerful, while mixts with higher expl oil content are not safe against firedamp. The expl oil content can be increased to 17-18% without sacrifice of safety if part of the above salts is replaced by 4-5% $Al_2O_3.2H_2O$ or $Fe_2O_3.3H_2O$) 45)J.Prior (to Dynamit AG), GerP 919156 (1954) & CA **52**, 14172 (1958) (Firedamp-proof blasting caps and detonators in which primary and secondary expls are admixed with one of the following compds: polycarboxylic- & oxygenated polycarboxylic acids; halogen-substituted polybasic carboxylic- & oxygenated carboxylic acids; and, their salts) 45a)B.T.Fedoroff et al PATR **2145** (1955) (Russian industrial & military expls) 45b)A.Cachin, Industrie Chimique Belge **20**, Spec Number, pp 217-20 (1955) & CA **50**, 6796 (1956) (A brief summary of the work done by CERCHAR toward the perfection of permissible expls of the reduced density type) 45c)R.L.Grant et al, USBurMinesReptInvest **5224** (1956) (Influence of atmospheric moisture on ignition of firedamp by expls) [See also RevIndMinérale **39**, 477-78 (1957)] 46)Gorst (1957), 102-05 (AN expls including some permissibles, called in Rus "predokhranitel'nyiye") 47)Yaremenko & Svetlov (1957), 113-17 (Theory of permissible expls); 147-50 (Rus Dinamony); 150-70 (Ammonity & other Rus coal mining expls) 48)Vaskovskii (1957), 24-32 (Ammonity & other Rus coal mining expls) 49)Taylor & Gay (1958), 18-22 (Historical on coal mining expls); 23-32 & 86 (Development of modern Brit coal mining expls) 49a)R.L.Grant et al, "Influence of Borehole Freespace on Ignition of Firedamp by Explosives," Presented at restricted meeting of CERCHAR,Verneuil, France, Oct 6-9 (1958) 50)McAdam & Westwater (1958), 6-9 & 34-50 (Brit permitted expls) 51)Poudrerie Royale de Wetteren, Cooppal et Cie (by H.Verlinden), GerP 1028923 (1958) & CA **54**, 25829 (1960) (Permissible expls consisting of a mixt of salts with exchangeable ions; to prevent ion exchange before detonation, at least one of these salts is coated, preferably with paraffin) 52)Blasters' Hdb (1958), 71-4 (DuPont's permissible expls) 53)B.T.Fedoroff et al, PATR **2510** (1958), pp Ger 101-102 (Kohlensprengstoffe); pp Ger 260-262 (Wettersprengstoffe) 54)Cook (1958), 10-12 (AN in NG contg high expls and also in non-NG expls); 15-16 (Developments in permissibles); 37-8 (Measurement of temp in detonation) 54a)A.Berthmann & G.Kuhn, USP 2829036 (1958) & CA **52**, 10581 (1958) (Firedamp-proof expls prepd by mixing alkali metal or alk-earth nitrates, Cl- contg salts, expl oils & other compds. Eg: $NaNO_3$ 56.8, NH_4Cl 35.7, NG 4.2, NGc 2.8 & argillaceous earth 0.5%) 55)J.E.Dolan, JApplChem(London) **9**, 59-64 (1959) & CA **53**, 10763 (1959) (Ignition of firedamp by expls contg NG-NaCl can take place if the salt is not ground finely enough) 55a)N.E. Hanna, G.H.Damon & R.W.VanDolah, "Probability Studies on the Incendivity of Permissible Explosives", USBurMines ReptInvest **5463** (1959) (Addn of relatively small quantities of NaCl tends to reduce the incendivity of expls to firedamp, but its addn cannot insure reduced incendivity. Chemical compn alone is not sufficient to define the incendive character of an expl) 55b)S.Nauckhoff & O.Bergström, "Nitroglycerin och Dynamit. Bidrag till Fabrikationens Historia", Nitroglycerin AB , Gyttorp, Sweden (1959) 56)Poudreries Réunies de Belgique SA, BelgP 573923 (1959) & CA **54**, 3957 (1960)

[Permissible semigelatinous or powdery expls consisting of an expl oil, an oxidizer, an adsorbent, an inhibitor and a polyethylene (or polypropylene) glycol of mol wt

≧ 2000 (which serves as a fuel and prevents agglomeration of mixt) together with an inorg water-insol product which may be coated with a fatty acid. Eg: Expl oil (such as NG & NGc) 10, Na nitrate 54.5, kieselguhr 1.5, Na nitrate 54.5, Amm chloride 32.5, a 75:25-polyethyleneglycol/carboxymethylcellulose mixt 1.0 & stearinated chalk 0.5%)] 57)Bandurin & Rukin (1959), 105-15 (Thermochemical calcns for permissible expls) 58)Giua, Trattato **6** (1) (1959), 368-78 (Esplosivi con nitrato d'ammonico) 58a)N.E.Hanna et al, USBurMinesRept Invest **5683** (1960) & CA **55**, 3981 (1961) (Reducing the incendivity of permissible expls by Na chloride) 59)Andreev & Belyaev (1960), 497-507 (Mechanism of ignition of firedamp and/or coal dust on expln of blasting charges); 507-16 (Basic properties to consider in formulating compns of permissible expls) 59a)Anon, "Analytical Methods for Powders and Explosives", AB Bofors, Nobelkrut, Borfos, Sweden (1960), 201, 203 & 206 60)A.Berthmann, "Explosivstoffe" (Part of Winnaker-KüchlerChemische Technologie, Vol 4), Hanser Vlg, München (1960), 59-61 (Wettersprengstoffe) 60a)H.J.Stratton, Edit, "List of Publications Issued by the Bureau of Mines From July 1, 1910 to January 1, 1960"Special Publication, USGovt Prtg Off, Washington, DC (1960) 60b)M.W. Hardison & O.V.Weaver, "List of Journal Articles by Bureau of Mines Authors Published from July 1, 1910 to January 1, 1960", USGPO, Washington, DC (1961) 61)W.S. Khablov, PromStroitel' **39**, No 4, 55 (1961); CA **55**, 20433 (1961) & Explosivst **1964**, 47 (Nonpermissible expl "Igdanit") 62)R.W. Van Dolah et al, USBurMinesReptInvest **5863** (1961) (Relative efficacy of stemming materials in reducing incendivity of permissible expls) 62a)Z.G.Pozdnyakov, "Izsledovaniye Stabil'nosti Promyshlennykh Vzryvchatykh Veshchestv" (Testing the Stability of Industrial Explosives), Izdat-InstGornagoDela, Moskva (1961) 62b)L. Médard, MAF **35**, 272 (1961) (Explosif type N n° 62) 63)N.E.Hanna et al, Ibid **5867** (1961) (Factors influencing incendivity of permissible expls: AN & carbonaceous materials) 63a)Anon, "List of Bureau of Mines Publications and Articles, January 1 to December 31, 1961", USGPO, Washington, DC (1962) 64)N.E.Hanna et al, US BurMinesInfoCirc **8087** (1962) (Active List of Permissible Explosives and Blasting Devices Approved before Feb 28, 1961) 65)Dr.Ing.Meerbach, Nobelhefte **1962**, 221 & Explosivst **1964**, 104 (Versuche zur Zündung von Schlagwettern unter Grubenbedingungen) (Investigation of firedamp ignition by permissible expls under conditions encountered in coal mines) 66)Anon, "List of Bureau of Mines Publications and Journal Articles, January 1 to December 31, 1962", USGPO, Washington, DC (1963) 67)Anon, "Publications of the Explosives Research Center 1946 Through 1963", US BurMines, Pittsburgh, Pa (1963) 68)Catalog, "Esplosivi ed Accessori da Mina", Montecatini SGIMC (Società Generale per l'Industria Mineraria e Chimica), Milano 69)Catalog, "BM Esplosivi", Mangiarotti, Codroipo (Udine), Italy 70)Catalog, "Esplosivi", Sorlini, (Antonio),Ghedi-Brescia, N.Italy 71)Catalog, "Esplosivi" Vulcania-SpA, Brescia, Italy 72)Dr.Camillo Belgrano, formerly Director of Vulcania SpA, Brescia, Italy, now Director SIPE di Gallicano, Lucca, Italy; private communication to Dr. O Vettori (June 15, 1964) 73)Dr.Omero Vettori, Chieri -Torino, Italy; private communications, July 1 & August 17, 1964 74)Dr.Camillo Belgrano; private communication, August 24, 1964 75)G.Giorgio, "Tecnica degli Esplosivi", Del Bianco, Udine, Italy (1964), pp 156-62 (Expls contg AN, K nitrate, chlorates or perchlorates); 166-85 (Mining expls)

Commercial or Industrial Explosives Prepared from Surplus Military Propellants. After termination of WWI there was left in Europe great quantities of proplnts and smaller quantities of high explosives. Some of them were loaded into ammo, others stored in boxes, cans, bags, etc. Some unused NC was stored in the wet condition.

Pepin Lehalleur (Ref 1, pp 456-58) describes methods of prepn and lists some Fr & Ger commercial expls prepd from surplus materials. Following are some examples: a)*Pyrolit 1* - Ballistite 74-6 & Na nitrate with or w/o K perchlorate 26-24% b)*Pyro-*

lit 2 - Ballistite 40-42, K perchlorate with or w/o Na nitrate 43-45 & aromatic nitrocompds, such as TNT 13-15%

Mixing of ingredients in these compns is conducted in presence of water

Belgrano (Ref 2, pp 115-16 & 287), under the title *"Esplosivi da mina con risiduati di polveri di lancio"* lists twenty formulations, most of them using Ballistite, but some using Italian Polvere C-2 (See Ref 2, p 113 & Vol 2, p C1-L of this Encycl), French Poudre B or Amer NC or proplnts. Following are some examples of such expls: a)Ballistite 50, Amer NC 45 &·Mn dioxide 5% b)Polvere C-2 50, K nitrate 40 & TNT 10% c)Ballistite 50, K nitrate 40 & TNT 10% d)Poudre B 15, DuPont proplnt 10, AN 60, TNT 10 & Ca silicide 5%

Refs: 1)Pepin Lehalleur (1935), 456-58 2)Belgrano (1952), 116 & 287

Commercial Plastisols. Accdg to definition given in Ref 2, a plastisol is a liquid dispersion of finely divided resin in a plasticizer. It is usually 100% solid with no volatiles; when volatile content exceeds 5% of the total wt it is called *organosol.* When the plastisol is heated, the plasticizer solvates the resin particles, and the mass gels. With continued application of heat the mass fuses to become a conventional thermoplastic material

Nine commercial plastisols were investigated at PicArsn in order to determine their possible use in Ordnance items. Results of invistigation are given in Ref 1

Refs: 1)R.J.Samuels, "Evaluation of Commercial Plastisols: A Comparison of Their Physical Properties", PATR **2023**, (1954) 2)CondChemDict (1961), 902-03

Commission des Substances Explosives, abbreviated as **CSE.** French "Commission of Explosive Substances", established in 1878 as a part of Service des Poudres et Salpêtres (Department of Powders and Saltpeters). The purpose of this Commission is to study the questions relative to manufacture and employment of all kinds of expls and proplnts. In order to report the results of investigations by CSE, a special journal was established in 1881, under the title of "Mémorial des Poudres et Salpêtres" (Ref 1). This title was changed in 1921 to "Mémorial des Poudres"

Refs: 1)Anon, MP **1**, III-IV (1882-83) 2) "Avant-propos, MP **18** (1921)

Commission Française du Grisou (French Commission for Firedamp). In order to study the problems in connection with the use of expls in gaseous coal mines, a special Commission was established in 1877 as a branch of the Service des Poudres et Salpêtres. Its first president was E. Mallard. The work of this Commission is briefly discussed under COAL MINING EXPLOSIVES AND SOME OTHER COMMERCIAL EXPLOSIVES and in Refs 1 & 2

Refs: 1)CSE, "L'Emploi des Explosifs en Présence du Grisou", MP **2**, 355-41 (1884-89) 2)CSE, Supplementary Rept on the same subject, MP **2**, 466-572 (1884-89)

Common-banded Coals. See under COAL

Common Pointed Shell, abbr as **CP Shell.** A Brit designation for a shell provided with a point which is not designed to penetrate the armor. Such a point, usually ogive of 2 caliber radius,is used in the shell known as: **Common Shell.** This shell has a hole in the nose for a fuze, and a very large cavity which is completely filled with a high explosive. If this expl is PA, the shell is known as "Common Lyddite"; if TNT or CE (Tetryl) is bursting chge, the shell is called "Common HE Shell"; and if the chge is BkPdr, such as used for practice, the name is "Common Shell". There are some Brit shells, which although known as "Common", have no hole in the nose, and consequently take a base fuze

Note: The shells similar to Brit "Common" are called in USA - "High Explosive Fragmentation Shells"

Ref: Anon, "Treatise on Ammunition", War Office, HMSO, London (1915), 158-61

Compacting of Materials. This term is applied to the process of closely packing or pressing together loose solid particles in order to form a dense body. It should be noted that compacting, itself, does not hold particles together as strongly as molding or

casting, unless some binding agent is used. Before compacting any material, the particles should be of the desired size. This can usually be achieved by granulating & sieving the material

Compacting of materials may be divided into bricketting, compressing, pelleting & tableting. Some compacting methods are used in ordnance, for example in the prepn of Tetryl pellets & in the prepn of some pressed expl charges, such as Composition A & Composition C type expls

Ref: Perry's Chemical Engineers' Handbook, McGraw-Hill, NY (1963), pp 8-59ff (Size Enlargement)

Compatibility of Explosives with other Substances. The term compatibility refers almost exclusively to chemical reactivity between materials, that is, the ability of a given expl to exist unchanged under certain conditions of temp & moisture, when in the presence of some other material. If this condition exists, the two materials are said to be compatible. Incompatibility of an expl and another material in which it may be in contact may result in loss of effectiveness of the expl, or it may result in extreme hazard

Various aspects of this subject, with respect to specific expls in contact with particular materials, are discussed in detail in the following refs

Refs: 1)O.E.Burton, "A Study of the Action of Explosives on Metals Used in Ammunition", PATR **65** (May 1931) 2)D.W.Biklen, "Reaction of Tetryl with Phenolic Plastic Materials", PATR **1264** (March 1943) 3)W. H.Rinkenbach, "The Reactivity of Waxes with TNT and Amatol", PATR **1323** (Aug 1943) 4)L.H.Eriksen, "Action of Explosives on Metals Used in Ammunition", PATR No's **1325** (Aug 1943), **1388** (Feb 1944), **1451** (Oct 1944) & **1493** (Jan 1945) 5)L.Gilman, "Effects Produced upon Stability of Explosives by Contact with Plastics", PATR **1376** (Jan 1944) 6)L.H.Eriksen, "Surveillance of PETN and Pentolite Stored in Contact with Steel Strips", PATR **1598** (May 1946) 7)S.Axelrod, "Effects Produced upon Explosives by Contact with Plastics", PATR **1636** (Dec 1946) 8)H.J.Eppig, "Effects of Moisture on the Stability of Pyrotechnic Compositions", PATR **1661** (Aug 1947) 9)A.J.Clear & O.E.Sheffield, "The Reaction Between TNT, Iron, Water and Trichloroethylene", PATR **1689** (April 1948) 10)O.E.Sheffield, "Compatibility and Reaction of Explosives with Other Materials. Effect of Sea Water and Iron on Explosives", PATR **1693** (May 1948) 11)S.Axelrod, "Effects Produced upon Explosives by Contact with Plastics", PATR **1780** (Sept 1950) 12)O.E.Sheffield, "Effects of Materials on the Properties of Explosives", PATR **1783** (Nov 1950) 13)S.Axelrod, "Effect of Rubber Compositions upon the Stability of Explosives", PATR **1797** (Feb 1951) 14)S.Axelrod, "Effects of Fungicidal Treatments for Lumber upon the Stability of Explosives", PATR **1819** & **1828** (June 1951) 15) J.E. Abel, "Stability of Rocket Propellants. Effect of Metals on Double-Base Propellants", PATR **1822** (July 1951) 16)O.R. Abolafia, "Compilation of Data on the Compatibility of Explosives and Polymers", PATR **1838** (October 1951) 17)S.Axelrod & B.Zlotucha, "Effect of Rubber Compositions upon the Stability of Explosives", PATR **1885** (Jan 1952) 18)S.Axelrod, "Effect of Polymetric Materials upon the Stablilty of T9 Composite Propellant", PATR **1897** (April 1953) 19)S.Axelrod, "Suitability of Plastics for Use in Contact with RDX Composition C-4 (Harrisite)", PATR **1907** (April 1953) 20)S.Axelrod, "Effects Produced upon Explosives by Contact with Plastics", PATR **1951** (August 1953) 21)B.Z.Zlotucha & M.Baer, "The Reactivity of Explosives with Metals and Protective Finishes", PATR **2288** (May 1956) 22)M.J.Bodnar, "Bonding of Explosives to Metal with Adhesives," PATR **2412** (June 1957) 23)M.C.StCyr, "Compatibility of Explosives with Polymers", PATR **2595** (March 1959) 24)Anonymous, "Compatibility of Rocket Propellants with Materials of Construction", DMIC Memo **65**, Defense Metals Information Center, Battelle Memorial Institute (Sept 1960) (PB No 161215) 25)K.Schriever, " A New Apparatus for the Determination of the Chemical Stability of Powders and Explosives", Explosivstoffe

8, 5-7 (1960) 26)J.W.Frazer, "A New Explosive Compatibility Test", University of California Research Laboratory, UCRL Report **6244** (1960), Contract W-7405-eng-48

Compex. One of the Brit "permitted" mining expls which are designated as EqS (equivalent in safety to sheathed explosives). It is listed in Ref without giving the composition
Ref: Taylor & Gay (1958), 96

Complete Round of Ammunition. See "Ammunition, Complete Round of" in Vol 1, p A385-L

Complete Round Tests. These tests may be considered as a final check on the proper assembly of the round (such as fixed-, or semi-fixed ammunition) and detn of its ability to function satisfactorily as a unit under service conditions. Complete rounds may be tested while the ammunition is being developed, for acceptance or surveillance. Final acceptance for issue to service, or storage for future issue, is based upon the results of this test (Ref 5)

The acceptance tests, which include: determinations of velocity, of safety and of functioning, are described in Ref 1 and in various military specifications (Refs 2-4)
Refs: 1)Anon, "Complete Rounds", Ordnance Proof Manual OPM **6-10** (1943) 2)USMilitary Specification MIL-R-20375 (1951) (Round, Complete, Shell, HE, M58 for Gun 90mm, M1) 3)Ditto, MIL-R-20520(1951) (Rounds, Complete, APC Projectile Ammunition, Base Fuzed, for Guns) 4)Ditto, MIL-R-20506 (1951) (Rounds, Complete, for Gun, 105mm, M3 5)OrdTech Term (1962), 79

Complex Explosives. These are formed by a combination of two or more compds or ions. Several cobalt & chromium complexes were studied by Tomlinson (Ref) to determine their promise as initiators or ingredients of priming compositions. As a class, chromic salts appeared to be more sensitive to initiation & more brisant, and to possess greater initiating ability than cobaltic compds. The latter type of complexes, however, appeared to be more stable at elevated temps and less affected by hot water than the former. These and other "coordinated complexes" are reported in some detail in Vol 1 under **Ammine or Ammoniate and Coordination,** pages A275-A286
Ref: W.R.Tomlinson, Jr, "Explosive Properties of Complex Compounds", PATR **1364** (Nov 1943) & **1632** (Oct 1946)

Complexometric Titrations. Title of the book by G.Schwarzenbach, Interscience, NY (1957)

Complex Salts Containing Ammine and Nitrate Groups. Under this term are known compds of general formula, $[M(NH_3)_n](NO_3)_m$, where M stands for a metal such as Cd, Cr, Co, Cu, Ni or Zn. These complexes are more or less explosive and several of them were listed as Ammines in Vol 1, pp A277 to A282 of this Encycl, together with ammines of azides, bromates, chrorates, iodates and perchlorates

Several of these complexes were prepd and investigated at Picatinny Arsenal, as can be seen from the paper of W.R.Tomlinson, K.G.Ottoson & L.F.Audrieth in JACS **71**, 375 -76 (1946)

Three of the complexes contg ammine and nitrate groups have been recently prepd and investigated at the laboratory of Wasag-Chemie AG at Neumarkt by Preller (Ref). Their prepn and props are given in the table which follows on p C 463

Complex Salt of Styphnic-Hypophosphoric Acids of the formula $[C_6H(NO_2)_3OKO]_2 \cdot Cu.Cu(H_2PO_2)_2$ was patented for use as a priming composition. Its prepn is described in Ref
Ref: J.F.Kenney, USP 2, 663, 627 (1953) & CA **48**, 3692 (1954)

Component of Artillery Ammunition, Tests. Artillery ammunition *components* (such as projectiles, cartridge cases, propellent bags, propelling or bursting charges, primers, boosters, igniters and fuzes) are subjected to various tests in order to det manufg compliance with specifications, satisfactoriness of functioning, or to obtain data for further

TABLE

Properties & Preparation	Tetramminecopper(II) Nitrate $[Cu(NH_3)_4](NO_3)_2$	Hexamminecobalt(III) Nitrate $[Co(NH_3)_6]NO_3)_3$	Hexamminenickel (II) Nitrate $[Ni(CH_3)_6](NO_3)_2$
Color	Dk blue crystals	Yel-orange prisms	Sapphire-blue crystals
Molecular Weight	255.7	347.2	284.2
Impact Sensitivity by BAM Method* with 2 kg Weight, mkg	0.75	1	Partial decomposition
Trauzl Test 10g Sample, cc	240	235	160
Compression Test by Kast Method, mm	3.2	2.8	-
Detonation Rate, by Dautriche Method m/sec	3500 2800	3000 2700	2400 -
Initiation with Cap No 3	Positive	Positive	Negative
Ditto with Cap No 8	Positive	Positive	Partial
Friction Sensitivity by BAM Method	No action at 12 kg, Crackles at 16 kg	No action at 16 kg; Crackles at 24 kg	No action at 16 kg; Crackles at 24 kg
Preparation	By mixing cooled concd solns of ammonia & Cu nitrate; or by passing NH_3 gas thru finely pulverized Cu nitrate	By treating $[Co(NH_3)_6]Cl_3$ with dil nitric acid; the chloride is prepd by passing air thru concd aq soln of $CuCl_2$ contg NH_4Cl, NH_3 & activated carbon	By passing NH_3 gas either thru dry finely pulverized Ni nitrate or thru its concd alcoholic soln.
See also Vol 1 of Encycl	p A281	p A280	Not described

BAM Method = Bundes Anstalt für Materialprüfung (State Institute for Testing Materials), West Berlin. This method is described in Explosivstoffe **1961**, pp 4 & 30. Info courtesy of Freiwald,(Ref 2)

Refs: 1) H.Preller, Explosivst **1964**, 173-74 2) Dr. H. Freiwald, private communication, Bad Godesberg, Germany (Dec 1964)

development and research

In conducting acceptance tests, the components may be tested in complete rounds or as individual items.

Methods of testing of artillery ammunition components are described in the No 8 series of the Ordnance Proof Manual (Refs 1 to 7) and in military specifications listed in Ref 8

Refs: 1)Anon, "Artillery Ammunition Component Tests, General", OPM **8-10** (1947) 2)Ditto, "Projectiles", OPM **8-11** (1943) 3)Ditto, "Propellent Powders", OPM **8-12** (1937) 4)Ditto, "Artillery and Mortar Fuzes", OPM **8-13** (1948) 5)Ditto, "Primers Cannon", OPM **8-14** (1943) 6)Ditto, "Boosters", OPM **8-15** (1942) 7)Ditto, "Cartridge Cases-Powder Bags", OPM **8-16** (1942) 8)Anon, "List of Specifications and Standards", Part 1, Alphabetical Listing, Defense Supply Agency, Washington 25, DC, p 77 (Boosters), pp 122-23 (Cartridge Cases), pp 314-18 (Fuzes), pp 369-70 (Igniters), pp 544-45 (Projectiles), pp 540-41 (Primers) and p 546 (Propellants)

Composite Detonators. See under Detonators

Composite Explosives. See Binary, Ternary and Quaternary Mixtures in Vol 2, p B116-B120

Composite Polyurethane Propellants. See conf rept by E.F.Cuddihy et al, "Reproducibility of Properties of Composite Polyurethane Propellants" (U), CALTECH Tech Rept No **32-481** (1964)

Composite Propellants. These proplnts, also known as *Fuel-Oxidizer Type Propellants,* consist of a finely ground oxidizer (such as an inorganic perchlorate or nitrate) in a matrix of plastic resinous or elastometric material which serves as a fuel. In most cases, the fuel serves also as a binder, but very often some additives are incorporated in order to assist in fabrication of the mixture into grains that will hold their shape. Some additives serve as curing catalysts, others as burning rate accelerator, etc. They might also include inhibitors which are intended to restrict the burning surface of grains (Refs 7, 12 & 16)

The current composite proplnts for use in JATOS or rockets are relatively new, having been developed beginning in 1940. They are nonhomogeneous in structure and in this respect differ from double-base rocket proplnts, which are homogeneous

Oxidizers used in composite proplnts are not as numerous as fuels. Sutton (Ref 16, p 340) lists the following inorganic perchlorates and nitrates which have been used in composite proplnts. Of these Amm perchlorate and AN are more widely used

Oxidizer	Available Wt Percent of Oxygen	Specific Gravity
$KClO_4$	46	2.5
NH_4ClO_4	34	1.9
$LiClO_4$	60	2.4
$NaClO_4$	52	2.54
NO_2ClO_4	66	2.25
NH_4NO_3	20	1.9
KNO_3	39.5	2.1
$NaNO_3$	47	2.26

All perchlorates are potential expls, of which the nitronium salt, $(NO_2)ClO_4$, is the most powerful and sensitive. The exhaust fumes of the perchlorates are toxic & corrosive and, with the exception of Amm and nitronium salts, very smoky. The nitrates are less dangerous to use, but K & Na nitrates produce very smoky exhaust and their oxidizing potential is low. AN possesses the advantage of having a smokeless and a relatively nontoxic exhaust, but its oxidizing potential is only slightly higher than that of K & Na nitrates. The presence of nitrates in composite proplnts induces slow burning

Fuels used in composite proplnts are very numerous, and, accdg to Sutton (Ref 16, pp 343-45), include the following:

a)*Asphalt and asphalt-oil fuels.* Several US composite proplnts developed at the beginning of WWII were prepd by incorporating a perchlorate into asphalt (previously liquefied by heating), and pouring the resulting slurry into rocket chambers. (Some asphalt-perchlorate mixts are discussed below under Cast Composite Propellants). As asphalt in these mixts became brittle, with tendency to crack at low operating temps, it was found advantageous to incorporate in asphalt some oil.

This improved the physical characteristics of mixts at low temps, but it made the material rather soft and easily deformable at higher temps

b)*Plastic fuels.* This include several thermosetting plastics (such as phenol formaldehyde- and phenolfurfural resins), and a few thermoplastics (such as styrene). In the case of proplnts using thermosetting plastics, the various components of plastic are mixed, the oxidizer is added, and the mixture is cast before the plastic sets. With styrene a soft, nonhardening elastic mixt is obtained

c)*Polyvinyl chloride (PVC).* In prepn of proplnts using PVC, $(-H_2C.CHCl-)_n$, it is employed as a plastisol (extremely small particles) mixed with a plasticizer (not specified), heated to 300°F and blended with crysts of Amm perchlorate or nitrate

d)*Polymers.* These include various vinyl-polyesters, polysulfides, polyurethanes, vinyl pyridines, butadiene polymers and several types of synthetic rubbers. They are prepd by polymerization of various monomers which when cross linked form large molecules. They make a strong matrix for the grain, retain some of their rubbery properties in the grain and take some rugged handling without cracking the propellant (Ref 16, pp 343-44)

Inorganic fuels. These include finely powdered easily combustible metals (such as aluminum & experimentally beryllium). These materials are used in conjunction with organic fuels listed above. The addn of ca 15% of Al particles seems to help in three ways: it raises the combustion temp (thus increasing the specific impulse), it increases density of the grain and it appears to alleviate certain types of combustion instability. The addn of Be causes a rise in combustion temp, and also the lowering of molecular wt. This gives products with 5-10% higher theoretical specific impulse than without Be. The disadvantage of Be proplnts is their extreme toxicity which requires special precautions in handling. Proplnts with metallic additives usually contain corrosive condensed phase (liquid or solid) in the exhaust gas and this is undesirable. Metallic hydrides, which have been used on experimental basis only, have the theoretical advantage to contain hydrogen in addn to a metal. This gives proplnts of high combustion temp and of low mol wts. As some of the hydrides react when left in contact with other ingredients of proplnts, the technique has been developed for encapsulating hydride particles with a metal or organic material membrane before incorporating them in proplnts (Ref 16, pp 344-45)

Composite proplnts may be divided into three classes, according to their methods of manuf. These include *cast composite propellants, molded composite propellants* and *solvent-extruded composite propellants* (Ref 1, pp 95, 99 & 101 and Ref 16, pp 336 -37 & 345-52)

Warren (Ref 7, pp 10-11) divides composite propelnts into two classes on the basis of their physical characteristics. When a resin binder is used such as in compn contg: Amm perchlorate 75, resin binder 20 & additives 5%, the resulting grains have a hard structure and are used as "free-standing grains". That is, they are either cast in molds or extruded to shape, and after being inhibited, can be loaded separately into motors in the same manner as double-base proplnt grains. When an elastometric binder is used (such as in compn contg: AN 80, elastometric binder 18 & additives 2%), a flexible grain results which may be bonded directly to the metal case. This can be accomplished by casting directly into the motor chamber

A. *Castable Composite Propellants* are prepd by blending an oxidizer (previously ground to a prescribed particle size to control the casting viscosity, mechanical props and burning rate) with a fuel and additives, in a mixer provided with rotating blades. If the fuel is brittle enough to be ground to a small particle size, mixing can be accomplished in cold and then the mixture is heated to melt the fuel. The resulting slurry is poured either into a special mold or directly into a rocket motor. With fuels like asphalt, mixing is done in hot, by adding a finely pulverized oxidizer to molten asphalt. The technique used in casting composite proplnts varies widely

After casting, the proplnt is cured. With some fuels this involves merely a slow and controled cooling process, while in others

some chemical reactions take place (Ref 16, pp 346-52)

The fuel-binder is low in viscosity during mixing & casting, but during the curing process the viscosity is greatly increased in order to form the solid propellant grain. With asphalt binders, the two viscosity ranges are achieved by first heating the composition during mixing & casting, and then cooling to form the grain. Some composite propellants achieve their low viscosity by the use of relatively low molecular weight liquids which do not react significantly during mixing & casting, but polymerize on heating (Ref 12)

Some castable composite proplnts developed in USA during and after WWII are described in Ref 1, pp 101-02; Ref 8, pp 114-16 & Ref 16, pp 336-37. Following are typical compns:

a)*Asphalt-potassium perchlorate propellants.* The earliest of these were *Calcits* (spelled Galcits in Ref 1, p 101), developed in early 1940's at California Institute of Technology in association with Aerojet-General Corp (Ref 1, p 101 & Ref 8, p 113). The *Calcit 53* consisted of K perchlorate 75 & asphalt (contg a little oil) 25%. It could be fired at temps +40°F & +100°F (Ref 8, pp 113-14)

Accdg to Ref 1, p 101, Calcits were easy to prepare requiring very simple equipment for their manuf. They had the advantage of a low temperature coefficient of the linear burning rate, but their disadvantages were brittleness at low temps and softening at high temps to the point of flowing

Sutton (Ref 16, p 337) gives the following properties for proplnts contg asphalt 22-30 & K perchlorate 78-70%: adiabatic flame temp 3800-3300°F; av mol wt 30lb/moles; specific heat ratio av 1.25; typical sea level specific impulse 180-195sec; characteristic velocity 3700-4200ft/sec; burning rate at 1000psi & 70°F 1.3-1.7in/sec; burning rate exponent, n 0.7-0.8; specific wt 0.063 lb/in^3; max volume impulse at 1000psi 2100lb-sec/ft; temperature sensitivity of pressure av 1%; lower combustion limit 500psi; upper pressure limit 10000psi; probable allowable operating temp limits +20° to 120°F; storage stability good; smoke abundant; mechanical props variable with temps; poor when warm

b)*Asphalt-ammonium perchlorate propellant,* developed by Mace of Aerojet contained: "Texaco 18 asphalt"8, Amm perchlorate 73, cetyl acetamide 3, dibutyl sebacate 5.5, castor oil modified glycerol sebacate 3 & chromium sesquioxide (serving as a combustion accelerator) 2.5 parts (Ref 2a &: Ref 8, p 114)

c)*Ethylcellulose-castor oil-perchlorate propellants.* A compn developed ca 1944 at the ERL (Explosives Research Laboratory) consisted of ethylcellulose- castor oil 25 & K perchlorate 75% showed some advantages over Calcit, but its useful temperature limits were too narrow. This mixture was improved ballistically by the addn of 5% of Al flake (Ref 1, p 101)

d)*Permafil resins-perchlorate propellants.* A typical mixture consisted of Permafil 2851 (one of the resins developed by General Electric Co) 24.6, K perchlorate 74.5, carbon black 0.5 & t-butylperbenzoate (serving as a peroxide catalyst) 0.4%. After blending the ingredients in the usual manner, and pouring the hot slurry into metallic molds, the molds were kept at 60°C for two days. During that time the resin polymerized (due to the presence of peroxide catalyst) to a rubber-like gel. Then the blend was cooled and removed from the molds

The properties of this proplnt are: it remains rubberlike at -40°F and does not flow at +140°F; its specific impulse is 170sec at 1000psi & optimum expansion; density 1.81g/cm^3; an exponent of 0.70 in the burning law; burning rate 0.72 in/sec at 1000psi & 70°F, and a restriction ratio of 182 under the same conditions. The temperature coefficient of pressure, thrust, & burning time at constant restriction ratio is 0.6% per °C (Ref 1, pp 101 -02)

e)*Castable ammonium nitrate composite propellant* was prepd by mixing 58 parts of finely ground crystalline AN, 11.6 ps Nitroguanidine & 7.2 ps dicyandiamide, fusing the mixt at 105°C and adding, while stirring, 8 ps of finely ground crystalline Amm dichromate. The melt was cast into cardboard molds of the required form, from which the casts were removed after cooling (Ref 8, p 115)

f)*Castable composite propellants listed by Sutton* (Ref 16, p 336). The following types of proplnts, including their properties, are discussed. All of them are based on compd

C_2H_4O, which is taken as typical in per cent compositions for many solid proplnt fuels: 1)K perchlorate 50-80 & C_2H_4O 50-20%; 2)Amm perchlorate 50-85 & C_2H_4O 50-15%; and 3)Amm nitrate 80 & C_2H_4O 20% with catalyst added 2%

Of all the composite proplnts employed at present time, the *castable ammonium perchlorate types* are most widely used

Armament Engrg (Ref 1b) gives compn & props of several castable perchlorate proplnts for rockets. The compn of one of them **ALT-161** was given in Vol 1, p A141-R of this Encycl under the name of ALT. Its props are given in Ref 1b as follows: d 0.064 lb/in^3, Isp 180 secs at 2000psi, burning rate 1.53in/sec, temperature sensitivity 0.35%/°F, temp limits -40 to +140°F, smoke -heavy. The compn of **AN-507** is described in Vol 1, p A400-L

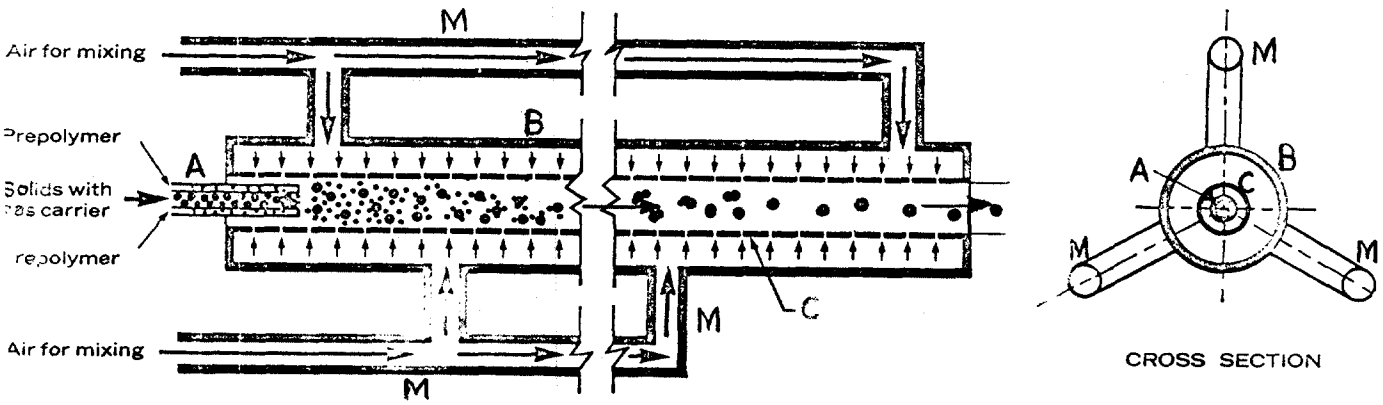
The compn and props of the proplnt called in Ref 1b, **Thiokol**, are as follows: NH_4ClO_4 21.15, $KClO_4$ 47.12, polysulfide rubber (Thiokol) 28.85 & additives 2.88%; its I_{sp} 194 secs, burning rate 0.7in/sec, temp sensitivity 0.17%/°F, temp limits -50 to +160°F and smoke - heavy

A continuous method of mixing castable proplnt was recently developed at the Aerojet General Corp, working under US Navy Contract NOrd 18171. Its description is given by A.S. Cohen in conf progress reports, which were not used by us as a source of info

In the open literature (Ref 17) is briefly described a continuous, remotely controlled, method of mixing ingredients for composite proplnts, which was recently developed at the US Naval Propellant Plant, Indian Head, Maryland. The pilot plant installation at Indian Head, represented schematically in Fig, consists of a mixer B (which is a long cylindrical vessel in which a porous pipe C is inserted); a tube A for introducing into the mixer the solid and liq ingredients ; and a series of manifolds, M, serving for injection of air thru the pores in C

In this process, solid ingredients (previously pulverized and preliminarily mixed in required propn) are injected by means of high velocity carrier gas thru tube A into pipe C. Simultaneously with this, drops of a liq ingredient (prepolymer) are injected, at the mouth of A, prependicular to the gas stream carrying solid particles. This gives an unevenly distributed solid-liquid dispersion and as this moves along the pipe C, air enters from manifold M thru the pores, thus creating a turbulent motion inside C. As result of this, a well-dispersed mixt of solids & liquids, diluted by the carrier gas, is obtained. When the gas is removed from the dispersion by a cyclone separator, a smooth, uniform mixt of ingredients is obtained.

B. *Molded Composite Propellants* are mixts of finely divided, crystalline, self-combustible solids such as ammonium picrate & sodium nitrate, held together by a resinous binder and formed into grains by compression mold-



Air Mixes Propellant Components In Continuous Process

ing. Molded composite propellants posses certain advantages over conventional gun propellants. They burn in a manner which results in a much lower sensitivity of the pressure in a rocket motor to such influences as changes in burning surface & nozzle area, than is the case with earlier types of double -base propellants. They are mechanically strong and, although somewhat brittle, can be placed in a motor so that the powder grain will not be injured by impacts. Composite propellants have great stability and are not altered by long storage at elevated temps. They can be easily coated to prevent burning of a given surface and are well adapted to produce grains of relatively large diameter (Ref 1)

A wide range of burning rates can be obtd by simple changes in the composite propellant compn. The composition & properties of several std composite propellants are reported as follows (Ref 1, p 96)

Molded Composite Propellants

Composition, %	218B	401	404	492
Amm picrate	46.7	72	54	41
Na nitrate	46.7	-	-	-
K nitrate	-	18	36	50
Butyl urea-formaldehyde resin (65% soln in butanol)	5.1	-	-	-
Plasticizer (Santicizer 8)	1.5	-	-	-
Calcium stearate	0.4	-	-	-
Ethyl cellulose	-	4	5	4.5
Liquid chlorinated polyphenyl (Aroclor 1254)	-	6	5	4.5
Ballistic Properties				
Specific impulse, sec	172	174	174	165
Density, g/cc	1.80	1.64	1.72	1.77
Impulse per unit vol of propellant, gsec/cc	310	285	300	292
Exponent in the burning Law $R=bP^n$, n	0.50	0.50	0.45	0.42
Linear burning rate at 1000psi, in/sec	0.70	0.24	0.57	1.0
Ratio of burning surface to nozzle area at steady-state press of 1000psi	170	600	240	155
Temp coefficient	0.005	0.005	0.005	0.003

(See also Ref 4 & Ref 8, p 114)
The burning rate varies considerably with composition, but specific impulse, discharge coefficient & exponent n, are almost the same for the above compns.
All the compositions produce considerable smoke

Armament Engrg (Ref 1b) gives compn & props of molded composite proplnt, designated as **49Z**: Amm picrate 41, K nitrate 50 & resin binder 9%; d 0.065 lb/in^3, I_{sp} 165secs, burning rate 1in/sec, temperature sensitivity 0.17%/°F and smoke - heavy

It should be noted that molded proplnts are not widely used at the present time

C. *Solvent-Extruded Composite Propellants.* Experience gained in the formulation & evaluation of molded composite proplnts led to the development of solvent-extruded compns. Although the extruded proplnts have less satisfactory ballistic props than castable or molded proplnts, they are tough and particularly adaptable to the manuf of long small-web grains needed for some rockets. Solvent-extruded composite propellants are mixed in sigma arm mixer with a sufficient amt of volatile solv (eth-alc, acet or mixts) added to form a dough. This dough is extruded thru dies mounted in hydraulic presses. The strands formed are dried at temps of 130°F or less in a current of air. Adjustment of kind & amt of solv, time & temp of mixing, temp & rate of extrusion, and drying time & temp must all be made for each particular compn. The compn & ballistic props of some solvent-extruded composite propellants are as follows (Ref 1, p 101)

Solvent-Extruded Composite Propellants

Composition, %	EJA	BBP	MJA	EDX
K perchlorate	56	7.8	-	-
K nitrate	-	-	43	43
Carbon black	9	1.2	7	7
Nitrocellulose (12.6%N)	21	-	-	23
Nitrocellulose (13.1%N)	-	54.6	26	-
Nitroglycerin	13	35.5	21.5	22
Ethly centralite	1	0.9	2.5	5
Mg stearate (added)	-	-	-	0.6

Ballistic Properties	EJA	BBP	MJA	EDX
Specific impulse, sec	152	168	130	150
Density, g/cc	2.02	1.68	1.8	1.83
Impulse per unit vol of propellant, g sec/cc	307	280	240	275
Exponent in the burning law $R=bP^n$, n	0.45	0.7	0.46	0.54
Linear burning rate at 200psi, in/sec	1.90	-	0.70	1.45
Temp coefficient	0.003	0.010	-	0.003

It should be noted that solvent-extruded proplnts are not widely used at present time.

More info about properties, manuf, testing and uses of composite propellants developed during WWII can be found in a number of OSRD reports, many of them listed in Ref 1. Many composite proplnts are known by names which associate them with the particular companies that manuf them. For example "Aeroplex" proplnts (See Vol 1, p A108-R of this Encycl) are made by Aerojet-General Corp. They cover a relatively large variety of relatively rigid compns utilizing any of the oxidizers and a number of resinous or plastic binders: "AN" proplnts have been developed at The Johns Hopkins University. One of its variety, AN-507, which is not classified is described in Vol 1, p A400-L of this Encycl). "Arcite" proplnts, products of Atlantic Research Corp, use such materials as vinyl polymers for fuel -binders with any of the oxidizers. "Thiokol" proplnts, products of Thiokol Chemical Corp, are based mostly on polysulfide binders (Ref 7, p 11)

Several of the major oil companies, such as Phillips Petroleum Co, have developed proplnts based on AN with synthetic rubbers & carbon black. Some of these compns have been claimed to be capable of operation over the temp range -75°F to +170°F (Ref 7, p 11 & Ref 8, p 114)

Much effort is being directed towards the production of cheap composite proplnts having flexible rates of burning & ability to withstand rough handling (See Refs 4, 9 & 10)

All the propellants discussed above in this section were taken from unclassified reports, papers or books. There are, however, more than one hundred US and British compns which are classified. The following composite propellants are described in conf "Propellant Mannual" SPIA/M2 (1962).
De

Designation	Unit No
IX-84-A	296
A-15	642
AK-14 Mod I	352
ALT-161	297
AMT-2091AX	473
AN-579Y	356
AN-583AF	358
AN-584J	359
ANP-512DC	480
ANP-2593 Mod 5	590
ANP-2606CD Mod III	605
ANP-2608AF	591
ANP-2639AF	568
ANP-2655AF	569
ANP-2716HL	606
ANP-2803HG	592
ANP-2805HY Mod I	607
ANP-2830BI Mod I	608
ANP-2832HO Mod I	609
ANP-2862JM Mod I	650
ANP-2864HG Mod II	651
Arcite-251	460
Arcite-362	546
Arcite-309	462
Arcite-368	524
Arcite-373	547
Arcite-402	595
B-13	643
BA-107	572
BF-122	570
BF-151	571
BRL-1	601
BSQ	519
C-505	594
CBS-128K	567
CDT(80)	578
CYH	596
CYH(77)	638
CYI(75)	637
DDP	597
DDP(77)	639
DGO	598
DGV	634
E-7	593

EFR	635
EJC	636
GCR-201C	562
GCR-300	566
GCR-518	611
H-3515	549
JPL-131	509
JPL-136	510
JPL-138	541
JPL-300	7
JPL-320	511
JPL-330	512
JPL-532	584
JPL-551	599
JPL-601	612
LFT-3	585
M-20	365
OMAX-S-2b	613
PPL-949	560
QZ_{bn}	600
RD-2312	582
RD-2406	583
RDS-127A	542
RDS-135	543
RDS-176	554
RDS-184	622
RDS-195	623
RDS-223	624
RDS-224	625
RDS-225	626
RDS-233	627
RDS-300	628
RDS-428A	603
RDS-429B	629
RDS-432	630
RDS-436	631
RDS-437	632
RS-1	526
T17E22	580
T24	533
TDX-2	621
TED-402	544
TEX-505	545
TP-G-3013D	614
TP-G-3014A	649
TP-G-8016	588
TP-H-1001	615
TP-H-8009	587
TP-H-8038	645
TP-H-8041	616
TP-H-8067	617
TP-H-8126	646
TP-L-8006	647
TP-L-8049	589
TP-L-8106	618
TP-L-8113	619
TP-L-8119	620
TP-L-2837	648
UR-101	573
XM-29	641

There are also very many classified experimental composite propellants which are listed separately on green sheets inserted in the Manual

Following is the list of US and of British organizations which contributed to development of composite propellants: Aerojet-General Corp Azuza, Calif; Allegany Ballistics Laboratory, Cumberland, Md; Amoco Chemicals Corp, Chicago, Ill; Army Rocket and Guided Missile Agency, Huntsville, Ala; Atlantic Research Corp, Alexandria, Va; Ballistics Research Laboratory, Aberdeen, Md; E.I. du Pont de Nemours and Co, Wilmington 98, Del; Explosives Research Laboratory, Bruceton, Penna; B.F. Goodrich Chemical Co, Cleveland, Ohio; Grand Central Rocket Co (now Lockheed Propulsion Co), Burbank, Calif; Hercules Powder Co, Wilmington 98, Del; Jet Propulsion Laboratory, Pasadena 3, Calif; Naval Ordnance Test Station, Inyokern, China Lake, Calif; Naval Powder Factory (now Naval Propellant Plant), Indian Head, Md; Olin-Mathieson Chemical Corp, East Alton, Ill; Picatinny Arsenal, Dover, N J ; Redstone Arsenal, Huntsville, Ala; Rockedyne Solid Propulsion Operations (North American Aviation Co), El Segundo, Calif; Rohm and Haas, Philadelphia 5, Penna; Thiokol Chemical Corp, Trenton, N J ; United Kingdom Organizations, London; and Wyandotte Chemical Corp, Wyandotte, Mich

The burning of composite proplnts, because of their more recent development, has not been as well explored as the older NC- and NC/NG-based proplnts. As indicated above, several oxidizers & many fuel binders can be used in formulating composite proplnts. The burning process can hardly be considered the same for each compn. However, some studies of the burning mechanism have been reported (Refs 1, 5, 6, 7 & 14). The burning mechanism as it is understood today is believed to occur with the oxidizer particles decompg in the midst of the decompg fuel matrix. Adjacent

streams of fuel-rich & oxidizer-rich gases rise from the surface. When mixing by diffusion is complete, immediate reaction occurs. The effectiveness of the mixing and the manner in which it occurs may account for the distinctive burning characteristics of composite propellants *Uses.* Composite propellants are used as JATO (Jet-Assited Take-Off) & rocket proplnts. They were developed primarily to eliminate the difficulties involved in the manuf of earlier double-base proplnts in large grains, and the changes in ballistic & mechanical props caused by changes in temp. Composite proplnts have been successful in these respects, although they are still affected by exposure to low temps. Further improvements in composite proplnts, and in their resistance to low temp effects, will be made as better fuels & binding agents are developed

It should be noted that a new technique has been developed to cast large grains of solventless double-base proplnts for rocket motors. This means that in future it will not be always necessary to use composite proplnts in prepn of large rocket proplnt grains

Refs: 1)Anon, "The Preparation and Testing of Explosives", Summary Technical Report of Division 8, NDRC Vol 1 (1946), 93ff 1a)W. Ley, "Rockets, Missiles and Space Travel", Chapman & Hall, London (1951), 380 1b)ArmamentEngrg (1954), 40-4 (Composite proplnts for rockets) 2)W.A.Arendale, IEC **48**, 725 -26 (1956) & CA **50**, 10411 (1956) (Fuel-binder requirement for composite proplnts) 2a)H.W. Mace, USP 2740702 (1956) & CA **50**, 9720 (1956) (Combustion accelerators for Amm perchlorate-asphalt proplnts) 3)P.J.Blatz, IEC **48**, 727-29 (1956) & CA **50**, 10410 (1956) (Rheology of composite proplnts) 3a)J.Humphries, "Rockets and Guided Missiles", Benn, London (1956), 27 (Composite proplnts) 4)C.A.Thomas, USP 2742672 (1956) & CA **50**, 11018-20 (1956) (Comp proplnt prepd by coating each grain of finely divided Amm picrate-Na nitrate with liq thermosetting resin, pressure molding & curing) 5)G.S.Sutherland "The Mechanism of Combustion of an Ammonium Perchlorate Polyester Resin Composite Propellant", Princeton Univ, New Jersey (1956), 233 6)A.J.Zaehringer, "Solid Propellant Rockets", AmerRocket Co, Wyandotte, Michigan, 1st Stage (1955) & 2nd Stage (1958) 7)Warren (1958), 10-11 (Characteristics), 60-67 (Manufacture) and 100 (Burning of composite proplnts) 8)Taylor (1959), 112-21 (Composite proplnts) 9)J.Linsk & R.W.Todd, USP 2936225 (1960) & CA **54**, 16834 (1960) [Comp proplnt prepd by pelleting at high pressure a mixt of AN 75-90 & a fuel-binder (PVA or cellulose acetate or butyrate) 25-10%] 10)W.M.St. John, Jr, USP 2941352 (1960) and J.E.Mahan & W.M.Hutchinson, USP 2941878 (1960)½ CA **54**, 21764 (1960); [Comp proplnts prepd by incorporating a burning rate accelerator (Prussian blue or dichromates) & a plasticizer (butoxyethylformal) into a rubbery binder (butadiene-methylvinylpyridine copolymer) before addn of a solid oxidant (AN or Amm perchlorate)] 10a)P.Tavernier & J.Boisson, MP **42**, 305-20 (1960) (A comprehensive description of prepn and props of solid composite proplnts) 11)R.P.Antonelli, Encyclopedia of Explosives, OTIA, OrdLiaison Group, Durham, NC (1960), 31-2 12)A.O.Decker, "Solid Propellants", JChemEduc **37**, 597-602 (1960) 13)Anon, "Safety Requirements for the Manufacture and Loading of Castable Composite Propellants", Ordnance Corps Manual, ORDM **7-230** (1960) 14)A.M.Ball, "Solid Propellants", OrdCorpsPamphlet ORDP **20-175** (1961), 16 (Burning rate of composite proplnts) 14a)F.J.Hendel, "Chemical Rocket Propulsion Systems", ChemEngrg **68**, No 3, 107-10 (1961) (Solid proplnts) 14b)L.Nadaud, "Die Verbrennug der Metallhaltigen Festen Treibstoffe", Explosivst **1962**, 186-93 (The combustion of metal-contg solid proplnts) 15)M. J.Zucrow, "Propulsion and Propellants", Army Materiel Command Pamphlet AMCP **706-282** (1963) (Reprint of ORDP **20-282**), pp 72-3 (Oxidizers, fuels & binders for composite proplnts) 16)G.P.Sutton, "Rocket Propulsion Elements", Wiley, NY (1963), 335 -57 (Composite proplnts) 17)Anon, C &EN **42**, 48-9 (Oct 5, 1964) (Continuous method of manuf of castable comp proplnts)

Addnl Refs: A)A.J.Zaehringer, Weltraumfahrt No **2**, 46-50 (1955) & CA **49**, 9929 (1955) (Discussion on composite & homogeneous proplnts) B)R.L.Hirsch, USP 2780996 (1957) & CA **51**, 6160 (1957) (Comp proplnt consisting of an alkyd-vinyl resin & an inorg perchlorate) C)N.L.Baker, Missiles & Rockets No **9**, 45-7 (1958) & CA **52**, 20984 (1958) (Prepn of cast

comp proplnts consisting of AN, synthetic rubber & C black) D)T.S.Briggs, Particle **2**, No 1, 20-4 (1959) & CA **54**, 5089-90 (1960) (Comp proplnt from amine perchlorate & Ca-base acid sulfite spent liquor from a coniferous wood) E)G.Knöffler & A.Rost, GerP 1059333 (1959) & CA **55**, 9880 (1961) (Castable comp proplnt consisting of AN 55, RDX 20, Ca nitrate 5 & hexamethylenediamine dinitrate 10%) F)J.Linsk, USP 2938778 (1960) & CA **54**, 17890 (1960) [Comp proplnt consisting of AN 70-90, combustible binder (prepd from PVA homopolymer, DNT & 2,4-dinitrodiphenyl ether) 5-25, combustible catalyst (Amm dichromate or Prussian blue) 1-5, gassing inhibitor (1,3-diaminobenzene, DPhA or urea) 0.2-3 & burning rate controlling substance (finely divided C) up to 5%] G)J.W.Perry et al, USP 2929697 (1960) & CA **54**, 15935-36 (1960) [Comp K perchlorate proplnts are cast directly in rocket motor casings w/o use of volatile solvents, during processing by means of a binder matrix consisting of a thermoplastic high molecular polymer of vinyl chloride & vinyl acetate and a plasticizer tri(butoxyethyl) phosphate] H)J.R.Eiszner & W.G. Stanley, USP 2942961 (1960) & CA **54**, 25830 (1960) (Comp proplnt consisting of AN 73-69, cellulose acetate ca 5; 2,4-dinitrodiphenylether 3; acetonyl acetone dioxime 2-3 & finely divided C 1-5%) I)E.F.Morello et al, USP 2942962 (1960) & CA **54**, 25830 (1960) [Addn of an aromatic amine (DPhA or dinaphthylamine) to proplnts of USP 2942961 renders them less susceptible to gassing at elevated temps. A typical compn: AN 71.5, cellulose acetate 4.65; ethyleneglycoldiglycolate 7.85; 2,4-dinitrophenylether 7.85; Amm dichromate (combustion catalyst) 2.00, Prussian blue (catalyst) 0.80, acetonylacetone dioxime 2.30, Norite A 0.80, C black 0.25 & DPhA 2.0%] J)P.O. Marti, Jr, USP 2946671 (1960) & CA **54**, 25831 (1960) [The addn of certain nonionic surfactants (Tergitol XC or Arlacel C) to the molten mix of AN & thermoplastic cellulose esters reduces the consistency of the molten mixt and the extrusion pressure needed to form a proplnt grain) K)P.O.Marti, USP 2946672 (1960) & CA **54**, 25831 (1960) (The use of PVC in the binder of a comp proplnt improves the stability of the grain to thermal shock) L)F.B.Cramer, USP 2949352 (1960) & CA **55**, 3063 (1961) (Comp proplnt consisting of a solid inorg oxidizer dispersed in silicone rubber) M)W.M.St.John, USP 2952530 (1960) & CA **55**, 3063 (1961) [Comp proplnt consisting of chlorinated paraffin (Clorafin or Cerechlor) incorporated with a solid inorg oxidizer (Amm nitrate or perchlorate) in a rubbery binder] N)H.A.Toulmin, Jr, USP 2954284 (1960) & CA **55**, 3064 (1961) [Comp proplnt consisting of a solid oxidizer (AN prills 70%), powdered metallic fuel (Al 15%) and a metal soap (Al stearate 15%)] O)E. D.Guth, USP 2962368 (1960) & CA **55**, 4966 (1961) [Comp proplnt consisting of an inorg oxidizer (AN), a fuel-binder (natural or synthetic rubber) & a burning rate catalyst (a ferrocyanide or chromate)] P)E.D.Guth et al, USP 2963356 (1960) & CA **55**, 5958 (1961) [Proplnt consisting of an oxidizer (Amm nitrate or perchlorate), rubbery binder, & a burning rate catalyst (Milori blue, $NaBH_4$, Zr, ZrH_4 or ZrC)] Q)C.C.Brice, USP 2965465 (1960) & CA **55**, 7844 (1961) (Extrudable comp proplnt consisting of AN, rubbery binder and a complex phosphate compd, which is added to improve extrudability) R)L.L. Weil, USP 2966403 (1960) & CA **55**, 9880 (1961) [Proplnt consisting of an oxidizer (AN) and a HE (Hexanitroethane or Mannitol Hexanitrate) dispersed in a solid rubbery gel] S)B.J.Grabe et al, SwedP 170732 (1960) & CA **55**, 14918 (1961) [Comp proplnt prepd by filling a mold with previously mixed partially unsaturated polyester, NG (or DEGDN) and Amm perchlorate or picrate, etc and leaving the dough to harden] T)G.D.Sammons, USP 2969638 (1961) & CA **55**, 11850 (1961) [Proplnt consisting of an oxidizer (AN), stabilized rubber binder, a salt of Picric or Styphnic acid and a burning rate catalyst (Na dichromate or Cu chromate)] U)H.M. Fox, USP 2970898 (1961) & CA **55**, 12856 (1961) [Castable comp proplnt is prepd by packing the mold with 3/16" pellets consisting of an oxidizer (Amm perchlorate or nitrate) ca 90, high energy element (B, Be, Mg, Li or Al) ca 8 & curable liquid binder (Et cellulose, polyurethan or polybutadiene) ca 2%. After evacuating the gases, the interstitial spaces betw the pellets are filled with a 75:25 mixt of an oxidizer and a curable liquid binder, to comprise ca 40% of the final

proplnt. Then the slurry is cured at 160°F for 24hrs] V)J.R.Eiszner, USP 2973255 (1961) & CA **55**, 15936 (1961) [Proplnt prepd by heating a mixt of PVC 3, DNT 3; 2,4-dinitrodiphenyl ether 3; insol Prussian blue 3, Micromex C beads 3, dintrophenylallyl ether & acetonylacetone dioxime 2 parts,for 20 minutes with steam at 15psi. Then an oxidizer (AN with some Na nitrate) 25 parts is added with stirring and the slurry extruded at 110°C to form grains] W)J.Linsk, USP 2973256 (1961) & CA **55**, 12856 (1961) (Castable comp proplnt consisting of AN 30-60, binder 15-25, burning rate catalyst (Prussian blue, chromates or dichromates or mono-Na-barbiturate) 2-6, finely divided C 0.3-2, org amine (DPhA) 0.2-2 & sorbitol sesquioleate 0.1 parts. The binder consists of cellulose acetate 20-40, DNT 10-30, dinitrophenoxyethanol 10-30 & ethyleneglycol diglycolate 25-40 parts] X)H. A.Toulmin, USP 2989388 (1961) & CA **55**, 19246 (1961) [Comp proplnt prepd by mixing AN ca 40, Guncotton ca 20 (in a suitable solvent), Mg bicarbonate or carbonate ca 30 and powdered metal (Al, Mg, etc) 8 parts, coated with 2 ps of metal soap catalyst (Ca stearate). The slurry is pressed at 2000psi into a block] Y)J.M.Burton, USP 2991167 (1961) & CA **55**, 26448 (1961) [Comp proplnt consisting of Amm nitrate or perchlorate, a rubbery copolymer binder, a reinforcing mixt of C black & SiO_2 and a burning rate controller (such as Prussian or Milori blue) and other ingredients] Z)R.W.Scharf, USP 2995430 (1961) & CA **55**, 26450 (1961) [Comp proplnt consisting of an inorg oxidizer, a polymer of conjugated diene contg four C atoms per mole, a substituted heterocyclic N base (pyridine or quinoline) and a HE (RDX, HMX, or TNT)] AA)M.L.Dickey, USP 2994598 (1961) & CA **55**,26450 (1961) (The performance of a comp proplnt contg C black may be increased by using C that has been activated by heating it for 10 min at 100-250° in dil acids) BB)R.W.Lawrence, USP 2978315 (1961) & CA **55**, 25260 (1961) [Incorporation in comp proplnts of 0.1-4% of Amm trichromate or tetrachromate (or their mixts) increases the burning rate by as much as 45%. For example, a cured proplnt grain contg Amm perchlorate 75, diethyleneglycol-adipic acid-maleic acid polyester 12.4, styrene 12.35, cumene hydroperoxide 0.25, lecithin (wetting agent) 0.05 and $(NH_4)_2 Cr_3O_{10}$ 1 part has a burning rate and burning rate exponent at 60°F & 1000 psi of 0.373 in/sec and 0.40,resp. When the catalyst is omitted, these values become 0.271 in/sec and 0.51,resp] CC)H.R.Ferguson USP 2991166 (1961) & CA **55**, 26449 (1961) [Elastic-gel comp proplnt is prepd by milling oil-extended GR-S rubber (contg 100 parts rubber & 30 ps oil) with 5 ps RPA No 3 (A 36.5% soln of xylyl mercaptan in a hydrocarbon solvent), 5 ps ZnO & 1 part stearic acid. Then 300 ps oil is added, in small increments, followed by Captax 12, Tuads 6, diphenylguanidine 18, C black 20, sulfur 6 and finally AN (50-100 mesh) 1892 parts. The batch is mixed 60 mins, deaerated, cast and cured for 24 hrs at 220°F] DD)C.C.Rice & W.B.Reynolds, USP 2993769 (1961) & CA **55**, 27891 (1961) [Comp proplnt prepd by mixing petroleum pitch with a polymer, a phase-stabilized Amm nitrate & Amm dichromate. The phase-stabilized AN is prepd by mixing 90 parts AN, 10 ps K nitrate & some water, heating the mixt to 140°F, drying and grinding to 40 micron size particles. The proplnt is compressed into grains. Example of compn: AN 82.95, K nitrate 9.22, petroleum pitch 4.11; 90/10 copolymer of 1,3-butadiene/2-methyl-5-vinyl pyridine 1.76 & Amm dichromate 1.96%] EE)R.MacDonald & A.M.Bedard, "Methods of Chemical Analysis of Cardeplex Propellant No 4760/A5 and Its Ingredients", CARDE **TR426/63** (1963) (Cardeplex No 4760/A5 is a composite ammonium perchlorate-polyurethane proplnt. Analysis of fully cured product includes detns of Amm perchlorate, Al, ferric acetylacetonate, phenyl-β-naphthylamine, lithium fluoride & total iron) FF)Anon, C&EN **42**, 50-3 (Sept 28, 1964) [Description of cast composite proplnts manufd at Aerojet-General Plant, near Homestead, Florida, for use in large rocket motors, such as 120" & 156" diam. Ammonium perchlorate, (previously ground to particles ranging from 1 to 2 microns and blended with ca 0.2% of tricalcium phosphate to improve their flow props), is used as an oxidizer. Aluminum powder is used as a fuel and liquid polybutadiene-acrylonitrite as the fuel-binder. To these ingredients are added a catalyst, such as iron oxide (to

achieve high-burning rate), a diepoxide curing agent and a plasticizer] GG)N.L.Coleburn, "The Shock to-Detonation Transition in Composite and Double Base Propellants", NOLTR **64-75** (Sept 1964)

Composition "106". A code designation for 1, 9-Dinitroxy-2,4,6,8-tetranitro-2,4,6,8-tetrazanonane

Composition 146-4-G. One of several resin -bonded expl compns prepd & tested by Benning *Ref:* E.T.Benning, "Development of Resin -Bonded Explosive Compositions", PATR **2062** (Sept 1954) (Conf)

Composition I & II are quarternary explosive eutectic mixts developed at PicArsn: **I** - AN 65.5, Na nitrate 10.0, Ca nitrate 14.5 & dicyandiamide 10.0% and **II** - AN 60.0, Na nitrate 24.0, GuN 8.0 & dicyandamide 8.0%

Both of these Comps can be prepd from cheap materials, which are readily available in time of war. Castable bursting explosive can be prepd by melting Comp II and, while agitating the liquid at ca 100°, adding about 30% of powdered Tetryl. The resulting blend possesses satisfactory stability, sensitivity, and fragmentation properties to serve as a substitute for TNT or other HE's
Ref: F.H.Fogel & S.Sage, "Develop Low Melting Ammonium Nitrate Explosive", PATR **1431** (1944)

Composition A Type Explosives

Composition A Type Explosives (Called *Hexogene/cire d'abeille* in France; *Füllpulver Nr 91-H5, Nr 92-H10, Nr H10.3 & Nr H3 in* Germany; *Angayaku, Koshitsu, Oshitsuyaku* or *Shouyaku Koshitsu* in Japan; and *Flegmatizirovannyi gheksogben* in Russia). The original Composition A was developed by the British during WWII. Composition A type expls are usually white to buff in color, although the German composition was dyed blue & the Italian red. The original British mixture, called **Composition A,** consisted of 91% RDX & 9% beeswax. This was a highly brisant explosive, suitable for press loading, and used as a bursting chge in ammo

When standardized in the US, the beeswax was replaced by a synthetic wax and the mixture was called **Composition A-2.** Subsequent changes in the granulation of the RDX and in the method of incorporating the wax led to the designation **Composition A-3**

Later developed A Type expls include: **Composition A-4**, which contains RDX 97 & desensitizing wax 3% and **Composition A-5**, consisting of RDX 98-99 & desensitizer 2-1% (See also under Analytical Procedures)

Following are preparation and properties of Composition A-3

Preparation. Composition A-3 is prepared by heating a water slurry of RDX to about 100°. The wax, together with a suitable wetting agent, is added to the rapidly agitated slurry, and heating is continued until the wax is melted. The mixture is then allowed to cool, while stirring, to a temperature below the melting point of the wax. The thoroughly coated granules of RDX are collected on a fitter & air dried at 75°C

Properties. Composition A-3 is a granular explosive, 95% of which is retained on a US std sieve 100; oxygen balance to CO_2 -48% and to CO -23%; compressed density 1.47g/cc at 3000psi & 1.65 at 12000psi; booster sensitivity test (See Vol 1, p VIII of Encycl) for pressed material of d 1.62 - 50% detonations are caused by 100g Tetryl pellets separated from Comp A-3 by Acrawax B pellets 1 70'' thick; brisance by sand test 107% TNT & by plate dent test, Method B, 126% TNT at d 1.62; detonation rate 8100 m/sec at d 1.59 & at RT for unconfined chge 1" diam; 7600 m/sec at d 1.51 for a sample kept at −54°C for 16 hrs; explosion temperature - decomposes at 250° in 5 secs; exudation - does not exude at 65° when waxes, melting sharply at 75° or above, are used; flammability index 195 (TNT 100); fragmentation test for 3'' HE M42A1 Shell contg 0 861g Comp A-3 at d 1.64 - 710 fragments vs 514 for the same wt of TNT; fragment velocity 2800ft/sec at 9 ft distance and 2530 ft/sec at 25.5 ft; friction pendulum test - unaffected by steel or fiber shoes; heat of combustion 1210 cal/g; heat of explosion - no info; heat test at 100° - 0.15% loss of wt in 1st 48 hrs; hygroscopicity 0.0% at 30° & 97% RH; impact sensitivity, 2 kg wt - 100+ cm BurMinesApp and 16'' (for 17mg sample) PicArsnApp (TNT 14''); power by ballistic mortar test 135% of TNT; rifle bullet impact test - unaffected; sensitivity to initiation - minimum detonating charge 0.25g

of LA; stability at high temps - see Heat test at 100° & Vacuum stability test; storage - dry, hazard class 9 compatibility group 1; vacuum stability at 100° - 0.3 cc gas evolved in 48hrs (0.1cc for TNT); at 120° - 0.6cc (vs 0.23cc for TNT); velocity of detonation - see detonation rate; volatility - % loss of wt 0.03 when kept for 15 days at 50° (Ref 19)

Uses: Composition A-3 has a combination of sensitivity characteristics which makes it particularly useful as a press-loaded HE filler for AA & SAP shells, in grenades, and it can be used as a booster chge (low wax content) in armor piercing shells (Ref 19). Various RDX (wax mixts were used during WWII by the British,Italians & Japanese (Ref 9)

Refs: 1)W.R.Tomlinson, Jr, PATR **1175** (1942) 2)H.Perry, PATR **1184**(1942) 3)J.Rubin, PATR **1424** (1944) 4)Blatt, OSRD **2014** (1944) 5)Anonymos, "Table of Military Explosives", Explosives Research Memorandum **10**, US Bureau of Ordnance (1944) 6)E.H.Eyster & C.A. Weltman,"The Preparation and Properties of RDX Composition A", OSRD Report **5626** (1945) (PB No 34803) 7)W.R.Tomlinson, Jr, PATR **1492** (1945) 8)Anonymous, Ordn **30**, 102 (1946) 9)All & En Expls (1946), 121-23 10)S.Fleischnick, PATR **1614** (1946) 11)H. A.Aaronson, PATR **1634** (1946) 12)S.Fleischnick, PATR **1639** (1947) 13)H.A.Aaronson, "Desensitization of High Explosives by Waxes. Semi-Plastic RDX Compositions", PATR **1761** (1950) 14)A.L.Forchielli, ibid, PATR **1787** (1950) 15)C.E.Jacobson, PATR **2112** (1955) 16)A.Leschinsky, "Development of Processes for Recovering RDX from Compositions A-3 and C-3" PATR **2154** (1955) 17)TM **9-1910** (1955), 199-201 18)USMilitary Specification **MIL-C-440B** (1961) (Compositions A-3 and A-4) and Picatinny Arsenal Purchase Description **PA-PD-2321** (1961) (Explosive Composition A5) 19)Anon, "Properties of Explosives of Military Interest", Section 1, (Army Material Command AMC Pamphlet **706-177** (1963), pp 40-1 (Revised edition of W.R.Tomlinson & O. E.Sheffield's PATR **1740**) (Reprint of ORDP **20-177**)

Composition A, Aluminized. An experimental expl consisting of Composition A 92% & aluminum 8% was developed, during WWII, at Picatinny Arsenal. This mixture was designated as *APX-4A* and was intended as a press-loaded filler for AP ammunition. It was found to offer considerable advantage over Explosive D in fragmentation & incendiary action (Ref 1). However, it was found in later tests that aluminized Composition A was too sensitive to withstand the shock encountered in armor plate impact tests (Ref 2 & 3)

Refs: 1)W.R.Tomlinson, "Develop New High Explosive Filler for AP Shot", PATR **1380** (1944) 2)W.R.Tomlinson, ibid, PATR **1417** (1944) 3)W.R.Tomlinson, ibid, PATR **1492** (1945) 4)ADL Pure Expl Compds Part **4** (1952), 588-89

Note: See also **APX Explosives** listing in Vol 1, p A475-L of this Encyclopedia

Composition A Type Explosives, Analytical Procedures. *Composition A-3.* Colorimetric method of identification is described in this volume of Encyclopedia under Color Reactions and Color Reagents and also in Refs 2 & 7

Quantitative method of analysis of Comp A-3, contg Stanolid wax, devised at the US Naval Ordnance Laboratory is described in Ref 1

Quantitative method of analysis of *Compositions A-3 and A-4* is described in Ref 5, which also lists the following US Military specification requirements for them:

	Comp A-3	Comp A-4
RDX content, %	91.0+0.7	97.0+0.5
Desensitizer, % (which shall completely coat RDX crysts)	9.0+0.7	3.0+0.5
Foreign matter	None	None
Moisture, %	0.10, max	0.10 max
Acidity (as % AcOH), when RDX is Type A(made by nitric acid process)	0.05, max	0.05, max
Acidity (as % HNO_3), when RDX is Type B (made by acetic anhydride process)	0.02, max	0.02, max
Insoluble particles	There shall be no insol particles retained on a USStd Sieve No 40 & not	

	more than 5 insol particles retained on sieve No 60	

Granulation:

Thru US Std Sieves

No 6	100%, min	100%, min
No 50	-	95%, max
No 100	5%, max	25%, max

Tests for Comp A-3 and Comp A-4

a)Foreign matter is detd by visual inspection

b)Moisture is detd by Karl Fischer Reagent Method as described in Ref 5, pp 5-7 & 11-13 and Refs 3 & 4

c)Acidity, for Comps A-3 or A-4 using Type A RDX is detd by titrating with 0.01N NaOH soln and for those using Type B RDX by titrating with 0.05N NaOH soln. The procedures are described in Ref 5, pp 8-9

d)Insoluble particles are detd by heating on a steam bath a 50g sample with 75ml of carbon tetrachloride in 400ml beaker, provided with a cover,until all sol matter appears to be dissolved. The soln is then decanted thru a 3" No 40 sieve placed upon a No 60 sieve and the residue in the beaker is heated with 24ml of CCl_4. After decanting this soln thru the same sieves, the residue in the beaker is heated with two 100ml portions of acetone, followed by filtering thru sieves. Finally the material left on the sieves is washed with acetone, dried on a closed steam bath and the insol particles counted

e)Granulation is detd by shaking a 100g sample on a nest of three superimposed sieves, placed on a Ro-Tap machine. The upper sieve is provided with a cover, while the lower one is assembled with a bottom pan. After shaking the assembly of sieves for 3 mins at 300±15 gyrations and 150±10 taps of the striker per min, the portions retained on each sieve are weighed and the results calcd to the percentage basis

f)RDX content is detd as follows: Place a 5g sample, accurately weighed,in a 50ml beaker, add 20ml of benzene satd with RDX, cover the beaker and place it on a steam bath. Occasionally lift the cover and crush the lumps by means of a glass rod. After about 30 mins of heating, cool the mixt and decant the sol portion thru a tared sintered glass crucible. Transfer the insol portion in the beaker into crucible using a "policeman" and four 5ml portions of benz satd with RDX. Remove the benz by drawing warm air thru the crucible and then heat it in oven for 15 mins at 100±5° until const wt

Percent RDX = 100A/W, where A=increase in wt of crucible and W=wt of sample on the dry basis

g)An alternate (chromous chloride) method for the detn of RDX in Comp A-3 is as follows: Dissolve by stirring and heating an accurately weighed 0.5g sample in 100ml AcOH contained in a 150ml beaker. Cool the soln, transfer it quantitatively into a 250ml volumetric flask, shake and fill to the mark with AcOH. Pipet a 25ml aliquot into a 500ml carbon dioxide titration flask from which the air has been removed by a stream of CO_2. While the flow of CO_2 thru the flask continues add 75ml of concd HCl and stir the contents by means of a magnetic stirrer for 10mins. Then, add exactly 100ml of 0.2N chromous chloride soln and, after stirring again for 10mins, 15 drops of a 2% aq soln of phenosafranin indicator. Titrate with 0.15N ferric ammonium sulfate soln to a sharp color change from green to deep red. Run a blank simultaneously with sample

$\%RDX = [12.340 \times N \times (V_1 - V_2)] / W$ where

V_1 = ml of ferric ammonium sulfate used to titrate the blank

V_2 = ml of above soln used to titrate the sample

N = normality of the above soln

W = wt of sample (See also Ref 4a)

h)Desensitizer is calcd by subtracting from 100 percent the precentage of RDX detd on a moisture-free basis

i)A nonaqueous titration method for detn of RDX in Comp A-3, was developed at Pic-Arsn (Ref 1a). It employs dimethylformamide as a solvent, a soln of Na methoxide in benzene-methanol as a titrant, and a satd soln of p-nitrobenzeneazoresorcinol in benz as an indicator. The method is the same as described under Cyclotrimethylenetrinitramine (RDX), Analytical Procedures (qv).(See also under Composition C)

Composition A-5. Quantitative methods of analysis and US Military Specification requirements are listed in Ref 6. There are two types of Comp A-5, each using RDX, Type A or B, Class C, which shall comply with Military Spec MIL-R-398C. Both types (I & II) of Comp A-5

are prepd by coating particles of RDX with desensitizer, using wet slurry process

Requirements for Composition A-5

	Type I	Type II
RDX content, %	98.5,min 99.0,max	98.0,min
Stearic acid, % (Spec MIL-A-271)	1.0,min 1.5,max	1.6,min
Lubricant (such as graphite with or w/o Ca resinate)		0.4,max
Foreign matter	None	None
Moisture, %	0.10,max	0.10,max
Acidity	Not specified	Not specified
Granulation: Thru US Std Sieve No 12	99.0%, min	

Tests for Composition A-5

a)Foreign matter is detd by visual inspection

b)Moisture is detd by titration method described either in Ref 3 or 4

Moisture can also be calcd by detg loss of wt of a 5g sample (weighed accurately),on heating it at 100-105° for 1 hr

c)Granulation is detd by shaking on a Ro-Tap a 50.0g sample on a No 12 sieve for 3mins, as described under Compositions A-3 & A-4, and weighing the portion retained on sieve

d)Stearic acid is detd by extracting a 5g sample in a tared, medium porosity, sintered glass crucible with three 20ml portions of chloroform (previously satd with RDX), maintaining each portion in contact with contents of crucible for ca 5mins with intermittent stirring before applying suction. Any lumps present in crucible should be crushed during extraction. The final suction should be conducted until complete disappearance of chlf odor and then the crucible with contents is dried for 30mins at 100-105°, cooled in a desiccator and weighed

% Stearic acid = 100A/W, where A = loss in weight of crucible + sample and B = weight of sample on a moisture-free basis

e)Lubricant content in Type II Comp A-5 is detd by extracting the residue remaining in the crucible (See proc d), with two 20-ml portions of DMF (dimethylformamide), maintaining each portion in contact with the residue for ca 5 mins with intermittent stirring before applying suction. This is followed by washing the residue with three or four 20ml portions of acetone, maintaining each portion in contact with the residue for ca 1min with continuous stirring before applying suction. Acetone is used to remove DMF (which is not very volatile), as well as RDX which remains after DMF extraction. The last washing with acetone should contain no RDX and this can be shown by evapg the acetone in a small dish and adding to the residue several drops of a soln of chromotropic acid in 36N sulfuric acid. If no reddish-brn color is produced, the residue in crucible is dried, first by aspiration and then by heating the crucible at 100-105° for 30mins, followed by cooling in a desiccator & weighing

% Lubricant = 100B/W, where B = wt of residue in the crucible and W = wt of sample on a moisture-free basis

f)RDX content is detd by subtracting the % of stearic acid and of lubricant (for Type II) from 100% (Ref 6)

Refs: 1)K.Van Keuren, "A Procedure for Chemical Analysis of Composition A-3", NAVORD Report **1781**, US Naval Ordnance Laboratory, White Oak, Md. (1951) 1a)S.M. Kaye, PATR **1936** (1953) (Nonaqueous titration method for detn of RDX content in Comp A-3) 2)Anon, "Military Explosives", **TM 9-1910** (1955), 271 (Identification of Composition A-3) 3)Purchase Description **X-PA-PD-940** (1956) (Karl Fischer moisture detn) 4)Federal Test Method Std No **141** (1958), Method **4082** (Karl Fischer moisture detn) 4a)C.C.Jamison, "Determination of Nitrogenous Coumpounds of Ordnance Interest by Chromous Chloride Reduction", PicArsn, FRL TechMemo **ACS-3-60** (1960) 5)U.S. Military Spec **MIL-C-440B** (1961) (Compositions A-3 and A-4) 6)Purchase Description **PA-PD -2321** (1961) (Explosive Composition A-5) 7)StdMethodsChemAnalysis **2B** (1963), 1347 (Identification of Composition A-3)

Composition B Type Explosives and Cyclotols

Composition B Type Explosives (called Hexolite or HT in France; Füllpulver Nr 18 & Füllpulver Nr 95 in Germany; Tritolite in Italy; Chauyaku or Nigotanoyaku Mk 2 in Japan; Tritolita in Spain; and Bonit or Hexotol in Sweden).

They were originally developed, before WWII, by the Germans and by the British. These mixtures are yel-brownish or yel-buff in color, and are castable explosives based on RDX & TNT in various proportions. Some of them contain wax

As standardized in the US, early in WWII, Composition B consisted of a mixture of RDX, TNT & a desensitizing wax. Compositions consisting of only RDX & TNT in proportions varying from 75/25 to 20/80 are called **Cyclotols.** Properties of Cyclotols and Composition B type expls are described after the preparation

Preparation. Composition B type explosives, including Cyclotols, are manufactured from TNT & water-wet RDX. The TNT is melted in a steam-jacketed kettle, equipped with a stirrer, and brought to a temp of ca 100°. The wet RDX is added slowly and heating & stirring are continued until most of the water is evaporated. The appropriate desensitizing wax or other additive is then thoroughly mixed with the other ingredients. On cooling the composition until it is of satisfactory fluidity for casting, it is then cast directly into the desired ammo, or so as to form chips. Chips of Composition B are prepared when it is to be stored or shipped for use in another location

The table which follows gives compositions and properties of Composition B and of Cyclotols used in the USA and in some foreign countries

Uses of Cyclotols in Foreign Countries:

Great Britain: Bursting and booster charges in various bombs, such as fragmentation, parachute and antisubmarine (Ref 12)

Germany: Cyclotols 50/50 & 53/47 as bursting chges in 4000kg bombs; shaped chge shells and as demolition chges. Cyclotol 60/40 (Filler No 95) as bursting chge is some shells. Cyclotol 20/80, plus 1% Montan was, known as Filler No 18 was used in some shells (Ref 12 & 29a)

Italy: Cyclotol 50/50 as bursting chge in shaped chge shells; also in land mines. Cyclotol 60/40 contg 2% wax as bursting chge in A/T bombs (Ref 12)

Japan: Cyclotol 50/50 (Chauyaku) and 70/30 & 60/40 (Nigrotanoyaku Mk 2) as demolition chges and as bursting chges in shells, bombs, land mines & bangalore torpedoes (Refs 10a, 11a & 12)

In addition to the mixts described in the above table the following Composition B type explosives were developed in USA:

Composition B-3. It is Composition B-2 (Cyclotol 60/40) supplied in the form of buds or strips, 1.5" in width & 3" in length (Ref 34c)

Composition B-4. It consists of RDX 60, TNT 39.5 & Ca silicate 0.5% (Ref 34d)

Composition B, Desensitized. Several expl mixtures of reduced sensitivity to mechanical action were developed at PicArsn after WWII (Refs 14 & 16)

The following two Comp Tupe B mixts are described in Ref 35, pp 48-50:

Mixture I: RDX 60 & TNT 40% with added 5 parts of emulsified wax (Stanolid or Aristowax, each of mp 165-170°F) & 2 parts of Vinylseal (MA 28-14)

Mixture II: RDX 55.2, TNT 40.0, Vistanex (B120) 1.2 & Albacer wax 3.6% (RDX is coated with combination of Vistanex & wax). Both Expls can be cast-loaded at d 1.65

Properties: brisance, by sand test (TNT 100) -110 for I & 115 for II; detonation rate - no info; expln temp - decomp in 5 secs at 260° for I & 270°for II; fragmentation test in 3" HE M42AI Shell loaded with 0.87lb of I or 0.87lb of II - total number of fragments 609 for I & 659 for II (TNT 514); friction pendulum test - both expls unaffected by fiber or steel shoe; heat of combustion - no info; heat of explosion - no info; heat test at 100°, % loss - in 1st 48 hrs 0.05 for Mixt I & 0.12 for Mixt II; in 2nd 48 hrs 0.19 for Mixt I & 0.18 for II; no expln in 100 hrs for I or II; hygroscopicity - nil at 30° & 90%RH for both expls; impact sensitivity, 2kg wt - 95cm BurMinesApp & 14" (for sample 17mg) Pic - ArsnApp for I; for expl II 13" (for sample 17mg) PicArsnApp (TNT 14"); power - no info; rifle bullet test - for expl I 95% unaffected & 5% burned and for expl II 100% unaffected; sensitivity to initiation - minimum detonating chge of LA-0.22g for I & 0.26g for II; vacuum stability test at 120 - cc of gas evolved in 48hrs 0.99 for I & 0.92 for II (TNT 0.23); at 150° 11+ for both expls (TNT 0.67); viscosity, poises - at 83° 3.5 for I & 3.1 for II; at 95° 2.6 for I & 2.7 for II; volatility - nil for both expls

Table
Composition and Properties of Compositions B & B-2 and of Some Cyclotols

Composition and Properties	Composition B	Cyclotols 75/25	Cyclotols 70/30	Cyclotols 65/35	Composition B-2
% RDX (US Spec MIL-R-398C)	60	75	70	65	60
% TNT (US Spec MIL-T-284)	40	25	30	35	40
% Wax, synthetic (US Spec MIL-W-20553A) added	1	-	-	-	-
Oxygen Balance to CO_2, %	-43	-35	-37	-40	-43
Color of material	Yel-brown	Yel-buff	Yel-buff	Yel-buff	Yel-brown
Density, cast, g/cc	1.68	1.71	1.71	1.71	1.68
Castability at 90°	Easily poured	Difficultly poured	Difficulty poured	Easily poured	Easily poured
Blast Effect in Air, TNT100:					
Peak pressure	110	111	110	-	104
Impulse	110	126	120	-	116
Energy	116	-	-	-	-
Booster Sensitivity (thickness in inches of Wax pellet). (See Vol 1, p VIII of Encycl) (For TNT = 0.82" for cast material at d 1.60)	1.40 at d 1.69	-	-	-	-
Brisance by Plate Dent Test, Method B, % TNT	132 (unconfined sample)	-	136 (unconfined sample)	-	132 (unconfined sample)
Brisance by Sand Test, % TNT	113	-	118	115	114
Compatibility with metals	Cause slight corrosion of Cu, stainless steel, Ni, Cd & Zn				
Compressive Strength-lb/in²	1610-2580	-	-	-	2200-3000
Detonation Rate, m/sec, at RT, for unconfined charge 1" dia.	7840 at d 1.68	8035 at d 1.70	8060 at d 1.73	7975 at d 1.72	7900 at d 1.72
Ditto at -54°	7720	-	-	-	-
Explosion Temperature (decompn in 5 secs)		-	265	-	
Exudation at 71°	Very slight	No info at our disposal			
Flammability Index	No info at our disposal				
Fragmentation Test:					
Number of fragments from 90mm HE, M71 Shell	998	1514	1165	1153	998
Wt of bursting chge, lb	2.187	2.22	2.213	2.253	2.187
No of fragments, from Shell contg TNT	703	703	703	703	703
Fragment Velocity, ft/sec:					
at distance 9 ft	2940	-	-	-	2965
at distance 25.5 ft	2680	-	-	-	2800
(For TNT at dist 9 ft 2600 and at 25.5 ft 2360 ft/sec)					
Friction Pendulum Test	Unaffected with fiber or steel shoe				
Gas Volume, cc/g	-	862	854	845	845
Heat of Combustion, cal/g	-	2625	2685	2755	2820
Heat of Explosion, cal/g	-	1225	1213	1205	1195
Heat of Fusion, cal/g	-	5.0	-	-	-

Table
Composition and Properties of Compositions B & B-2 and of Some Cyclotols

Composition and Properties	Composition B	75/25	Cyclotols 70/30	65/35	Composition B-2
% RDX (US Spec MIL-R-398C)	60	75	70	65	60
% TNT (US Spec MIL-T-284)	40	25	30	35	40
% Wax, synthetic (US Spec MIL-W-20553A) added					
Heat Test at 100°:					
% Loss in 1st 48 hrs.	0.2	-	0.07	-	-
% Loss in 2nd 48 hrs.	0.2	-	0.08	-	-
Explosion in 100 hrs.	None	-	None	-	-
Hygroscopicity, %, at 30° and 90% RH	0.02	-	Nil	Nil	Nil
Impact Sensitivity:					
Bur Mines App, 2kg wt, cm	75	-	60	-	75
Pic Arsn App, 2kg wt, in	14	-	14	-	14
(Wt of Sample in mg)	(19)	-	(20)	-	(19)
Power, by Ballistic Mortar Test, % TNT	133	-	135	134	133
Power, by Trauzl Test, % TNT	130	No info at our disposal			
Rifle Bullet Impact Test:					
% Explosions	3	30	30	-	5
% Partials (Smokes)	13	40	30	-	55
% Burned	4	0	0	-	25
% Unaffected	80	30	40	-	15
Sensitivity to Initiation (Minimum detonating charge in g)	LA 0.20; MF 0.22	-	LA 0.20 MF 0.21	-	LA 0.20 MF 0.22
Shaped Charge Effectiveness, % TNT:					
Hole volume with glass cones	178	-	-	-	178
Hole volume with steel cones	162	-	-	-	162
Hole depth with glass cones	125	-	-	-	125
Hole depth with steel cones	148	-	130	130	148
Specific Heats, cal/g/°C; at: -75°	0.235	0.220	No info at our disposal		
0°	0.220	0.225			
25°	0.254	0.254			
50°	0.305	0.296			
75°	0.376	0.352			
85°	0.354	0.325			
90°	0.341	0.332			
100°	0.312	0.351			
Storage (for every expl)	Dry; hazard class (quantity - distance) 9; and compatibility group I				
Vacuum Stability Test at 100°	0.7cc/48 hrs	0.23cc/48 hrs	-	-	-
Ditto at 120°	0.9cc/48 hrs	0.41cc/48 hrs	0.86cc/48 hrs	-	0.29cc/48 hrs
Ditto at 150°	11+cc	-	-	-	-
Viscosity, Absolute, in poises	3.1 at 83° 2.7 at 90°	210 at 85°	53.2 at 90°	30.2 at 85° 26.0 at 90°	12.3 at 85°
Volatility	-	-	Nil	Nil	Nil
Uses	Fragmentation boms; HE shells; grenades; shaped charges		Shaped charge bombs especially fragmentation; HE shells; grenades		

Uses: Mostly in bombs (Ref 35, pp 48-50)

Another Comp B type mixt consisted of RDX 57.0, TNT 38.0, wax (Stanolind or Aristowax, mp 170-175°F) 4.8 & polyvinyl acetate/resin adhesive 0.2%. In prepn of this expl mixt, the wax was emulsified in TNT by polyvinyl acetate/resin adhesive. The expl props of this compn are somewhat similar to the above mixtures, but it was not considered suitable due to the instability of the wax/TNT emulsion on standing (Ref 14)

A laboratory investigation led to the development of a composition consisting of RDX 60, TNT 32 & N-octadecylphthalimide 8%. This mixture was found to be much less sensitive to mechanical shock than std Composition B. Its brisance value by sand test is equivalent to Composition B, but its fragmenting power, ballistic mortar and rate of detonation values are lower than the values for Composition B. The composition is castable at the same temp used for std Composition B and shows no evidence of exudation after six months storage at elevated temps (Ref 16)

Refs: 1)H.Perry, "Study Loading Characteristics of RDX Compositions", PATR **1211** (1942) 2)L.Zapf, "Drilling of RDX Composition B" PATR **1224** (1942) 3)J.Rubin, "Properties of RDX Composition B without Desensitizing Wax", PATR **1313** (1943) 4)E.H.Evster & M. A.Paul, "Composition B (Cyclotol)", OSRD Report **1167** (1943) 5)J.Rubin, Evaluation of Waxes as Substitutes for Aristowax in RDX Compositions A and B", PATR **1424** (1944) 6)S.Kogan, "Effect of RDX Granulation on the Properties of Composition B", PATR **1433** (1944) 7)J.Rubin, "Properties of RDX Composition B without Desensitizing Wax", PATR **1435** (1944) 8)P.B.Tweed, "Effect of RDX Granulation on the Properties of Composition B", PATR **1458** (1944) 9)L.H.Eriksen, "Properties of 70/30 Cyclotol", PATR **1476** (1944) 9a)A.T.Blomquist OSRD Rept **3014** (1944) (Microscopic examination of RDX-TNT systems) 10)G.M.Hopkins, "Examination of Explosives for Shaped Charges", PATR **1482** (1945) 10a)Anon, "Handbook of Japanese Explosive Ordnance", OPNAV **30-3M** (1945), 27 (Nigotanoyaku Mk 2) 11)S.Fleischnick, "Investigation of Malfunctioning of Composition B Loaded 105mm 130E1 Shell", PATR **1592** (1946) 11a)G.C.Tibbetts, et al, "Japanese Powders and Explosives", PBRept **50394** (1946) p 91 (Chauyaku) 12)All & En Expls (1946), 123-25 13)S.Fleischnick "Evaluation of Explosives Based on Shell Fragmentation", PATR **1688** (1948) 13a)O.E.Sheffield, PATR **1709** (1949) (Establishment of optimum granulation of RDX for use in Cyclotols) 13b)M. C.Epton, PATR **1715** (1949) (Development of process for reworking RDX composition, such as Comp B) 14)A.L.Forchielli & H.A. Aaronson, PATR **1756** (1950) (RDX Comp B of reduced mechanical sensitivity) 14a)D.C.McLean, PATR **1793** (1950) (A study of various solvents and solvent mixts for RDX and investigation of the effect of surface active agents on the viscosity of high RDX content Cyclotols) 14b)W.M.Evans ProcRoySoc **A204**, 14 (1950) (Detonation rate of 50/50 Cyclotol) 15)M.Baer, PATR **1803** (1951) (Investigation of methods for detn of viscosity of Comp B & other binary expls) 16)A.L.Forchielli PATR **1865** (1952) (Comp B of reduced mechanical sensitivity) 17)S.J. Lowell, PATR **1978** (1953) (Cast loading of Comp B with heated probes) (C) 18)S.J.Lowell & P.B.Tweed, "Investigation of Surface-Active Agents for Reducing the Viscosity of Molten Composition B and Cyclotols", PATR **1983** (1953) (C) 19)S.Fleischnick & C.E.Jacobson, "Development of Improved Methods for Cast-Loading RDX/TNT Compositions and Comparison of Composition B and 50/50 Pentolite as HE Filler for 2.36 Inch M6 Rocket Heads," PATR **2008** (1954) 20)L.Jablanski, "Investigation of Segregation in Composition B", PATR **2019** (1954) 21)N.D.Baron & E.A.Skettini, "Evaluation of Composition B for Use as the High Explosive Filler in 90mm M71 HE Shell", PATR **2063** (1954) 22)L. Jablansky, "Heat Study of Composition B-Loaded 155mm M101 HE Shell", PATR **2103** (1954) 23)I.L.Kintish, "Effect of a Hot Weapon on Composition B-Loaded 105mm HE Shell", PATR **2131** (1955) 24)D.Zauder et al, "Effects of Storage on Composition B-Loaded and TNT-Loaded 76mm M42AI HE Shell", PATR **2175** (1955) 25)A.Nordio, "The Cooling and Solidification of Molten Composition B and the Causes of Shrinkage Cavitations in Cast-Loaded Shell", PATR **2190** (1955) 26)R.J.Heredia et al, "Effects of Storage on Composition B-Loaded

and TNT-Loaded 90mm M71 HE Shell", PATR **2380** (1956) 27)C.E.Jacobson et al "Evaluation of Materials for Use as Desensitizers in Composition B", PATR **2425** (1957) 28)L. Jablansky, "Factors Affecting Sensitivity of Composition B to Setback", PATR **2433** (1957) 28a)A. M.Anzalone, SFAL, TechInfoSect, PicArsn, Bibliography No 4, Part 1, "Bibliography on the Flow and Viscosity Properties of Cyclotols, Composition B and TNT" (1957) 29)P.B.Tweed & S.D.Stein, "Loading and Safety Testing of Composition B", PA ExplsDevelopSect, SFAL Report **12** (June 1957) 29a)B.T.Fedoroff et al, PATR **2510** (1958), pp Ger 30 & Ger 47-48 30)R.W.Heinemann, "Improvement of Composition B and HBX Compositions", PATR **2513** (1958) 31)R.W.Heinemann, "Safety Firings of Composition B-Loaded 175mm T203E3 High Explosive Shell," PATR **2590** (1958) 32)R. L.Jameson, "Electrical Measurements in Detonating Pentolite and Composition B", BRL Memo Rept **1371** (September 1961) 33)W.Kegler, Explosivst **1961**, 2, 27-30 (Examination of vacuum -cast RDX-TNT charges) 34)US Military Specifications:
a)MIL-C-13477 (Ord) (1954) & Engineering Change Order ECO No 26973-S ANR (Cyclotols)
b)MIL-C-401C (1963) (Composition B)
c)MIL-C-45113A (MU) (1963) (Composition B-3)
d)MIL-C-46652 (MU) (1962) & Ammendment 1 (Composition B-4) 35)Anon, "Properties of Explosives of Military Interest," Section 1, AMCP **706-111** (1963) (Revised edition of W. R.Tomlinson & O.E.Sheffield's PATR **1740**) (Reprint of ORDP **20-177**)

Composition B Type Explosives and Cyclotols, Analytical Procedures. *Composition B.* Colorimetric method of identification is described in this volume of Encyclopedia under Color Reactions and Color Reagents and also in Ref 6

Quantitative method of analysis of Comp B is given in Ref 2, which also lists the following US Military Spec requirements for Comp B:

RDX, % (Spec MIL-R-398C)	59.5±2.0
TNT, Grade I, % (Spec MIL-T-248)	39.5±2.3
Desensitizer, % (Spec MIL-W-20553)	1.0+0.3
Moisture, %	0.25, max
Viscosity for Grade A	7.0 efflux secs, min, 17.0 max
Viscosity for Grade B	7 1 efflux secs, min 17.0 Efflux secs, max
Insoluble particles	Max 5 particles retained on No 60 sieve
Granulation	Unless otherwise stated Comp B shall be supplied in the form of buds or as strips ca 1.5" wide & 3" long

RDX shall be thoroughly and uniformly incorporated with molten TNT

Tests:

a) Moisture is detd either by conductometric method (Ref 2, pp 10-13) or by Karl Fischer method, described in Ref 1, Method **101.4**, using 1:1-methanol/chloroform as a solvent

b) Viscosity is detd by efflux method as described in Ref 1, Method **212.1**

c) Insoluble particles are detd by solvent extraction method as described in Ref 1, Method **106.1**

d) RDX content is detd in the manner described under Compositions A-3 & A-4, test f

e) Desensitizer content is detd as follows: Weigh accurately ca 5g of finely ground sample in a tared, coarse porosity, sintered glass crucible and add 15ml of reagent grade acetone, previously cooled to 5°. After swirling the contents for 5 secs apply suction. Repeat washing & aspirating for an addnl nine times, using a total of 150ml acetone. Before removing the last portions of acetone by suction, see that the sides of the crucible are washed down. Then aspirate the crucible for 5 mins, dry it in a desiccator for 5 mins and weigh
%Desensitizer = [100×(A+0.009W)] /W, where A = wt of desensitizer; W = wt of sample on a moisture-proof basis; and 0.009 = compensating factor for soly of desensitizer in acetone

f) TNT content is detd by subtracting from 100 the sum of the percentages of RDX & desensitizer

g) Alternate (chromons chloride) method for detn of TNT in Comp B is described in Ref 2, p 9

h) Alternate (chromous chloride) method for detn of RDX in Comp B is described in Ref 2, pp 9-10

i) Alternate method for detn of desensitizer is by subtracting from 100 the sum of percentages of TNT & RDX

Composition B-3. Same as 60/40 Cyclotol

Composition B-4

Following are US Military Spec (Ref 4) requirements:

RDX, Type B, % (Spec MIL-R-398C)	60±2.0
TNT, Grade I (Spec MIL-T-248)	39.5±2.0
Calcium silicate, % (Spec MIL-C-51077)	0.5±0.1
Moisture, %	0.25, max
Viscosity (efflux secs, max)	7.0
Insoluble particles	Max 5 particles retained on No 60 sieve
Form	Comb B-4 shall be supplied in the form of buds or strips ca 1.5" wide and 3" long

Tests:

a)Moisture is detd either by conductometric method described in Ref 4, pp 11-14 or by Karl Fischer method described in FED-STD-791, Method **3253**, except that the sample shall be 8 to 10g

b)Viscosity is detd by efflux method as described in Ref 4, p 14

c)Insoluble particles are detd by solvent extraction method, as described in Ref 1, Method **106.1**

d)Form is detd by visual inspection

e)TNT content is detd as follows: Heat on a steam bath, a 50ml beaker contg a 5g sample, accurately weighed, and 15ml of benzene satd with RDX. Crush the lumps and then keep the beaker covered. Agitate occasionally by swirling. After 30mins of heating, cool to RT and filter the supernatant liquid thru a tared sintered glass crucible of medium porosity. Remove the residue from the beaker by washing it with four (or more) 2-3ml portions of benz (satd with RDX) into the crucible. Aspirate until disappearance of odor of benzene in the crucible, dry it for 1 hr at 100±5°, cool in a desiccator and weigh:

%TNT = 100(A-B)/W, where A = wt of crucible & sample; B = wt of crucible & residue after benz extraction and W = wt of sample on the dry basis

f)RDX content is detd as follows: Extract the residue in the above crucible with eight 10ml portions of hot acetone, allowing each portion to remain in contact with the residue for 1min, before applying suction. Aspirate until disappearance of odor of acetone in the crucible, dry for 1hr at 100±5°, cool in a desiccator and weigh:

%RDX = 100B/W, where B = the difference in wt of crucible before & after extraction with acetone and W = wt of sample on the dry basis

g)Calcium silicate content is calcd as follows:

%Ca silicate = [100(C-D)]/W, where C = wt of crucible with residue after acetone extraction; D = wt of empty crucible and W = wt of sample on the dry basis

h)Alternate (chromous chloride) method for detn of TNT is described in Ref 4, pp 8-9

i)Alternate (chromous chloride) method for detn of RDX is described in Ref 4, pp 9-10

j)Alternate method for detn of Ca silicate is as follows: Place on a water bath a 100ml beaker contg a 5g sample (accurately weighed) & 50ml of acetone. Crush the lumps with a glass rod, cover the beaker and swirl it occasionally during warming. After ca 30mins, decant the soln thru a tared sintered glass crucible of medium porosity, and warm the residue in the beaker with 20ml of acetone. Filter the soln thru the same crucible and remove the residue from the beaker by washing it with several portions of acetone into the crucible. Aspirate to the disappearance of odor of acetone, dry the crucible with residue for 1hr at 100±5°, cool in a desiccator and weigh:

%Ca silicate = [100(C-D)]/W, where C = wt of crucible with residue after acetone extraction; D = wt of empty crucible and W = wt of sample on the dry basis

Cyclotols are mixts in various proportions of RDX & TNT. Only the specs for 60/40 and 75/25 Cyclotols are available (Refs 3 & 5)

Following are US Military Spec requirements for Cyclotols:

	60/40 Cyclotol or Comp B-3	75/25 Cyclotol
RDX, %(Spec	59.5±1.0	75.0±2.0

MIL-R-398C),		
TNT, Grade I, % (Spec MIL-T-248)	40.5 ± 1.0	25.0 ± 2.0
Moisture, %	0.25, max	0.25, max
Viscosity, efflux, secs, max	20	15
Insoluble particles	Max of 5 particles retained on No 60 sieve	
Granulation	See item e under tests	Not specified

Tests:

a)Moisture is detd by conductometric methods described in Ref 3, pp 7-10 & Ref 5, pp 3-5

b)Viscosity is detd by efflux method described in Ref 1, Method **212.1**

c)Insoluble particles are detd as described in Ref 1, Method **106.1**

d)RDX content is detd by benzene extraction method, such as described under Compositions A-3 & A-4, test f

e)TNT content is detd by subtracting from 100 the percentage of RDX

f)Granulation of RDX particles extracted from Comp B-3 is detd by the method described in Ref 3, pp 11-16. RDX should have a median particle diam of 65 to 80 microns

Refs: 1)USMilitary Standard, "Explosive: Sampling, Inspection and Testing," **MIL-STD-650** (1962) 2)US Military Specification **MIL-C-401C** (1963) (Composition B) 3)Ditto, **MIL-C-45113A(MU)** (Composition B-3) 4) Ditto, **MIL-C-46652(Ord)** (1962) (Composition B-4) 5)Ditto, **MIL-C-13477(Ord)** (1955) (Cyclotol) 6)StdMethodsChemAnalysis **2B** (1963), 1347-48

COMPOSITION C TYPE EXPLOSIVES

Composition C Type Explosives (called *Plastic Explosives* in Gr Britain; *Explosifs plastiques* in France; *Hexoplast 75 & Plastit* in Germany; *Koshitsu Oshitsuyaku & Shouyaku Koshitsu* in Japan; and *Exógeno plastico & Plastex* In Spain) The original Composition C was first used by the British, during WWII, as a plastic demolition charge. These explosives could be shaped by hand and had great shattering power. As standardized in the USA, a composition designated as **Composition C** was a white plastic which consisted of RDX 88.3% & a nonexplosive oily plasticizer 11.3%. The plasticizer contained 0.6% lecithin which helped to prevent the formation of large RDX crysts. Composition C, while plastic from 0-40°, became brittle & less sensitive below 0° and tended to become gummy & exude oil above 40°C. Its expl properties are given in Ref 19, pp 51-2

Composition C, therefore, was replaced in the US by **Composition C-2** which was a white plastic consisting of RDX 78.7% & explosive plasticizer 21.3%. The expl plasticizer was composed of DNT 12.0%, TNT 5%, MNT 2.7%, NC (pyroxylin) 0.3% & solvent 1.0%. Composition C-2 was plastic from -30° to 50°, but became less plastic at higher temp storage because of evapn of the volatile matter. Expl props of Comp C-2 are given in Ref 19, pp 53-4

Comp C-2 was replaced by **Composition C-3**, which consisted of RDX 77, DNT 10, MNT 5, TNT 4, Tetryl 3 & NC 1%. The mixt of the last five ingredients served as an explosive plasticizer. This yel puttylike solid was satisfactory as a demolition explosive except that it became hard at -29° & below and exuded considerably when stored at 77° & above

Properties of Composition C-3: blast (relative to TNT), in air-peak pressure 105 & impulse 109; booster sensitivity test = 50% detonations produced by 100g Tetryl separated from pressed Comp C-3 of d 1.62 by Acrawax B pellet 1.36" thick; brisance = by sand test 111% TNT and 118% by plate dent test, method B; detonation rate 7625m/sec for hand tamped unconfined charge 1" diam at d 1.60 at RT; explosion temp = decomp at 280° in 5secs; fragmentation test for 90mm HE, M71 Shell, charged with 2.045 lb of Comp C-3 at d 1.58 = number of fragments 944 (TNT 703); friction pendulum test = unaffected by fiber or steel shoe; heat test at 120°, % loss = 3.20 in 1st 48hrs, 1.63 in 2nd 48hrs and no expln in 100hrs; hygroscopicity 2.4% at 30° & 95% RH; impact sensitivity, 2kg wt = 100+cm, BurMinesApp & 14" (for 33mg sample), PicArsnApp (TNT 14");power = by ballistic mort 126% TNT & by Trauzl test 117%; rifle bullet impact test = unaffected 60, and partials 40%; sensitivity to initiation = min detonating chge 0.20g LA & 0.08g Tetryl; storage = dry, class 9, group 1; vacuum stability test, cc evolved in 48hrs = 1.21cc at 100° (TNT 0.1cc) and 11+cc at 120° (TNT 0.65cc); volatility = 1.15% loss at 25° in 5 days

Use: Plastic demolition charges (Ref 19, pp 55-6)

Composition C-4, also called **Harrisite**, was developed before 1950 at Picatinny Arsenal by Ottoson (Ref 10) to serve as replacement for Comp C-3 which possessed some undesirable props. A semiplant method for manufg Comp C-4 was developed in 1956 by Lerner (Ref 14). This light brown putty-like substance contains RDX 91 & nonexplosive plasticizer 9%, consisting of di-(2-ethylhexyl)sebacate 5.3, polyisobutylene 2.1 & motor oil 1.6 parts. It remains plastic between -57° and +77° and is considered as a very satisfactory demolition explosive which almost entirely replaced all other Comp C type expls. It is odorless and nontoxic because it contains no aryl nitrocompds

Preparation. In the laboratory prepn of Composition C-4, dry RDX is added to a petroleum ether soln of di(2-ethylhexyl)sebacate, polyisobutylene & SAE 10 engine oil. The solvent is evaporated and the residue worked by hand into a homogenous dough-like mass. Since the use of a volatile flammable solv is undesirable in plant production, a solventless process was developed. In the plant process two different granulations of RDX are used: 61 parts of Class (or Class B) & 30 parts of Class 3 (See description of RDX classes under Cyclotrimethylenetrinitramine in this vol of Encyclopedia). The water-wet mixture of both is placed in a stainless steel mixing kettle and 9 parts of "polyisobutylene" binder are added. The mass is blended by tumbling the kettle until completely homogeneous. The resulting dough is transferred to trays where it is dried by forced air at 50-60°. About 16hrs are required to reduce the moisture content to below 0.25%

Properties of Composition C-4. It is a light -brown soft putty-like plastic explosive which can be hand-tamped to a density of 1.58 - 1.60 g/cc. Its *brisance* by sand test is 116% of TNT and by plate dent test, method B 115% (for unconfined hand-tamped material at d 1.60); detonation rate at RT, 8040m/sec, for unconfined, hand -tamped chge,diam 1" & d 1.59; Ditto - for sample kept at -54° for 16hrs 7020m/sec at d 1.36, vs 7040m/sec for sample at +21°; explosion temperature - 290° in 5secs; exudation - none at 77°; friction pendulum test - unaffected by fiber or steel shoes; heat test at 100°, % loss - 1st 48hrs 0.13, 2nd 48hrs 0.00 & no expln in 100hrs; hygroscopicity, % gain at 30° & 95% RH - nil; impact sensitivity, 2kg wt - BurMines App 100+cm (sample 20mg) and PicArsnApp 19" (sample 27mg) (TNT 14"); power, by ballistic mortar test 130% TNT; rifle bullet test - unaffected 80 & burned 20%; sensitivity to initiation - LA 0.20g & Tetryl 0.10g; storage - dry, hazard class 9 (quantity - distance), and compatibility group I; vacuum stability test at 100° - 0.20cc/40hrs (TNT 0.10) (Ref 19, pp 57 -8)

Uses: Composition C type explosives, of which Composition C-4 is the principal type, are used primarily as demolition blocks, in some underwater charges and a bursting charge in some rocket shells

Refs: 1)F.R.Benson, "Study Properties of RDX Composition C-2", PATR **1293** (1943) 2)F.R. Benson, "Suitability of Composition C-3 as a Plastic Explosive," PATR **1416** (1944) 3)Anon "Data on Foreign Explosives", US Chemical Warfare Service Field Lab Memo **4-6-2**, US Office of Technical Services PB Report **11544** (1944) 4)L.H.Eriksen, "Effect of Storage on the Sensitivity of Compositions C, C-2, C-3 and PEP-2D" PATR **1518** (1945) 5)All & En Expls (1946), 125-28 6)Anonymous, Ordn **30**, 102 (1946) 7)S. Fleischnick, "Evaluation of Explosives Based on Shell Fragmentation," PATR **1595** (1946) 8)S.Livingston, "Plastic Explosives. Comprehensive Study of the Plastic Explosives RIPE, PEP-3 and Composition C-3," PATR **1695**(1948) 9)R.C.Grass, "Development of an RDX Plastic Explosive of the C-3 Type," PATR **1713** (1949) 10)K.G.Ottoson, "Plastic Explosives. Development of an RDX Plastic Explosive of the C-3 Type", PATR **1766** (1950) 11)S.Axelrod, "Suitability of Plastics for Use in Contact with RDX Composition C-4 (Harrisite)", PATR **1907** (1953) 12)H.W.Adam, W.P.Morton & E.J. Murray, "Development of an RDX Plastic Explosive of the C-3 Type", PATR **2028** (1954) 13)A.Leschinsky, "Development of a Process for Recovering RDX from Compositions A-3 and C-3", PATR **2154** (1955) 14)S.Lerner, "Development of a Semiplant Process to Manufacture Composition C-4 (Harrisite)", PATR **2292** (1956) 15)T.W.Stevens, D.E.Seeger & D. H.Stone, "Development of the M5 and M5A1

Demolition Blocks", PATR **2332** (1956) 16) PATR **1740**, Rev 1 (1958), 54-67 17)US Joint Army-Navy Specification **JAN-C-427**, "Composition C-3" (12 Dec 1946) 18)U.S.Military Specification **MIL-C-45010A(MU)**, "Composition C-4" (26 Sept 1963) 19)Anon, "Properties of Explosives of Military Interest", Section 1, AMCP **706-111** (1963), pp 51-9 (Revised edition of W.R.Tomlinson, Jr & O.E.Sheffield's PATR **1740**) (Reprint of ORDP **20-177**)

Composition C Type Explosives, Analytical Procedures. *Composition C-3.* Colorimetric method of identification is described in this Vol of Encycl under Color Reactions and Color Reagents and also in Ref 5

Quantitative method of analysis of Comp C-3 is given in Ref 1, which also lists the following US Joint Army-Navy Spec requirements for Comp C-3:

	Class A	Class B
RDX, plus NC, %	78.0±2.0	78.0±2.0
C-3 plasticizer, minus NC, %	22.0±2.0	22.0±2.0

Note: Comp C-3 shall consist of RDX crysts, conforming to the requirements of Spec MIL-R-398C, completely coated with a plasticizer approved by the agency concerned. It shall have a soft putty-like consistency and shall not crumble when kneaded by hand

Moisture, %	0.25,max	0.25,max
Acetone insol material, org & inorg, %	0.15,max	0.15,max
Acetone, insol material, inorg, %	0.05,max	0.05,max
Grit	See under Tests	
Acidity, %	0.064, max	0.05, max
Plasticity	See under Tests	
Detonation	See under Tests	
Uses	Demolition blocks	Demolition blocks and ammunition

Tests:

a)Moisture is detd in Ref 1, p 3 by distn with carbon tetrachloride, but it can also be detd by conductometric method & Karl Fischer method which are listed under Compositions A and B, Analytical Procedures

b)Acetone insoluble material, total is detd in the following manner: Warm a 10g sample with 200ml of acetone in a 400ml beaker, provided with a cover. Crush the lumps & occasionally stir and after ca 15mins, filter the soln thru a tared sintered glass crucible. Transfer the insol portion, by washing with acet, into the crucible and rinse the residue in the crucible with hot acetone. Aspirate, heat at 100±5°, cool in a desiccator and weigh:

%Insolubles = 100A/W, where A = wt of residue in the crucible and W = wt of sample on the dry basis

If the percent of insol is less than 0.05, no test for inorganic insol is required. If greater than 0.05%, ignite the material in the crucible, cool in a desiccator and weigh:

%Insol, inorganic = 100B/W, where B = wt of residue after ignition and W = wt of sample on the dry basis

c)Grit is detd in the following manner: Heat on a steam bath, a 50g sample with 100ml of carbon tetrachloride in a 400ml beaker provided with a cover. Crush the lumps and stir ocassionally until complete disintegration. Decant the liquid thru a small No 40 sieve placed on top of No 60 sieve and heat the residue in beaker with two 50ml portions of acetone and then transfer the insol in the beaker to the sieves. Wash any residue on the sieves with acetone and dry them. Count and examine any particles retained. No particles shall be on No 40 sieve and max of five particles on No 60 sieve. Note if the particles produce scratching noise when pressed and rubbed with a smooth steel spatula on a smooth glass slide

d)Acidity is detd in the following manner: Warm on a water bath, a 10g sample with 100ml of acetone in a 1000ml beaker, provided with a cover. Crush the lumps and stir until the sample is dissolved. Add, with vigorous stirring, 100ml of CCl_4 and then, slowly, 200ml of water. Finally add, more rapidly, 300ml of w and cool. Add 15 drops of mixed indicator, which is prepd by dissolving 0.15g of bromocresol green & 0.10g of methyl red in NaOH soln, neutralizing the soln and making it up to 250ml with w. Titrate with 0.05N NaOH soln to a blue green end point. Run a blank and correct for acidity or alkalinity of reagents:

%Acidity as HNO_3 = [6.302(V-v)N]/W, where V = ml NaOH used in titration of sample; v = ml NaOH used in blank titration; N = Normality of NaOH soln and W = wt of sample on a dry basis

e)RDX content is detd in the following manner: Treat a 5g sample, accurately weighed, with 15ml of benzene (satd with RDX) in a tared 50ml beaker, provided with a cover. Crush the lumps with a glass rod and when the sample has been completely disintegrated, add another 15ml of benz (satd with RDX) and stir for a few mins. Allow to settle and filter the liquid thru a sintered glass crucible of medium porosity. Transfer the bulk of the residue to the crucible, and wash the beaker, crucible, and residue with five 15ml portions of benz (satd with RDX), stirring each time the contents of the crucible while the wash-liquor runs thru by gravity. When dripping stops, apply suction to remove the bulk of residual wash-liquor and break vacuum before adding a fresh portion of solvent to prevent pptn of RDX in the benz-RDX soln by evapn & cooling. Dry the beaker and crucible for 2hrs at 70°, cool in a desiccator and weigh each of them:

%RDX plus NC = [100(A+B)]/W, where A = wt of residue in the crucible; B = wt of the residue in the beaker and W = wt of sample on a dry basis

f)Plasticizer minus NC is calcd by subtracting from 100 the percent of RDX plus NC

g)Plasticity test is conducted as described in Ref 1, p 5. Comp C-3 shall undergo a minimum extension of 0.9" prior to fracture of test piece of dimensions indicated in Ref 1, pp 5 & 10

h)Detonation is detd as described in Ref 1, pp 5-6. Comp C-3 shall detonate completely and shall effect perforation of the test plate immediately beneath the charge

Composition C-4

The following US Military Spec requirements and tests are described in Ref 4

	Class 1	Class 2	Class 3
RDX, Type A or B, %	91.0±1.0	91.0±1.0	90.5±0.7
Polyisobutylene, % (Desensitizer-binder)	9.0±1.0	9.0±1.0	9.5±0.7

Note: RDX complying with the requirements of MIL-R-398C, shall be thoroughly and uniformly incorporated with a binder complying with the requirements of MIL-P-14536 to form a homogeneous compn having a soft, putty-like consistency

Moisture, % maximum	0.25	0.25	0.25
Insoluble particles	No particles shall be retained on a No 40 sieve and not more than 5 particles on a No 60 sieve from a 50g portion of the sample		
Plasticity, units, max	0.030	0.018	0.080
Specific gravity, g/cc		1.50,min	-

Tests:

a)Moisture content is detd in accordance with Method 101.4, described in Ref 2

b)Insoluble particles are detd as follows: Warm a 50g sample with 400ml petroleum ether in a 600ml beaker, provided with a cover. Crush the lumps and occasionally stir until complete disintegration. Then decant the soln thru a small No 40 sieve placed on top of a No 60 sieve and add to the residue in the beaker ca 100ml of acetone. Warm on a water-bath until all sol material dissolves and pour the soln, together with all insol particles, thru the above sieves. Wash the residue on the sieves with ca 50ml of acetone, dry them and count the particles retained on No 60 sieve. No particles shall be retained on No 40 sieve

c)Plasticity (modulus of compressibility) shall be detd in accordance with Method 211.2 described in Ref 2

d)Specific gravity detn is conducted only for class 2 and the method is described in Ref 4, p 9

e)RDX content, by gravimetric method is conducted as follows: Heat, on a steam bath, 2g sample, accurately weighed, with ca 35ml of carbon tetrachloride (satd with RDX) in a 100ml beaker, provided with a cover. Stir occasionally, without crushing the lumps, until the binder has dissolved which takes ca 30mins. Cool the beaker to RT and filter the contents thru a sintered glass crucible of medium porosity. Transfer the residue from the beaker to the crucible and wash with three 10ml portions of CCl_4, satd with RDX. Aspirate the crucible until complete removal of CCl_4, dry in an oven at 100±5° for 1hr, cool in a desiccator and weigh

%RDX = 100A/W, where A = wt of residue in the crucible and W = wt of sample on a dry basis

Note: The RDX particles recovered from Class

3 shall consist of 1 part fine and 3 parts coarse. Fine RDX shall comply with Class E of Spec MIL-R-398C. Coarse RDX shall meet the following granulation requirements: 98±2 thru a No 20 sieve, 90±10 thru a No 50 , 35±5 thru a No 100 and 18%, max thru a No 200 f)Polyisobutylene (desensitizer) is det by subtracting from 100 the percentage of RDX g)RDX content by nonaqueous titration method is conducted by the method developed at Pic Arsn and described in Ref 2. It differs slightly from the method used in detn of RDX in Comp A-3 and described in PATR **1936** (1953). The method included in the Spec (Ref 4, pp 7-8) is as follows:

Heat, on a steam bath, a 1.5g sample, accurately weighed, with 25ml of carbon tetrachloride in a 150ml beaker provided with a cover. Swirl until all polyisobutylene dissolves. Cool to RT and remove all liquid by suction thru a fine porosity filter stick. Add 50ml of DMF (dimethylformamide), tech grade, to the beaker and stir the mixt, using the filter stick, until all of the RDX is dissolved. Transfer the soln quantitatively to a 100ml volumetric flask and make up to the mark with DMF. Prepare a blank consisting of DMF. Pipet 20ml of soln into 100ml tall beaker, add 5 drops of azo violet indicator (a satd soln of p -nitrobenzeneazoresorcinol in benzene) and introduce a magnetic stirrer. Cover the beaker with a round glass provided with a hole in the center to admit a buret tip. Titrate (while stirring) with 0.1N sodium methoxide to a green end point which persists for 30secs. Treat the blank in the same manner

%RDX= [7.40(V-v)×N] /W, where V=ml of 0.1N Na methoxide used for sample; v=ml of the same reagent used for blank and W=wt of sample on the dry basis (Refs 2 & 4)

Note: Prepn of Na methoxide reagent is described in Ref 2, p 4 and under Cyclotrimethylenetrinitramine, Analytical Procedures

Refs: 1)US Joint Army-Navy Spec, JAN-C-427 (1946) (Composition C-3) 2)S.M.Kaye, "The Development of Nonaqueous Titration Method for the Determination of the RDX Content of Composition C-4", PATR **1958** (1953) 3)Anon, "Military Standard Explosive: Sampling Inspection and Testing", MIL-STD-650 (1962) 4)US Military Spec MIL-C-45010A(MU) (1963) (Comp C-4) 5)StdMethodsChemAnalysis **2B** (1963), 1349 (Comp C-3)

Composition D-2. This composition consists of a mixture of desensitizing wax 84, lecithin 2 & Nitrocellulose 14%. It is prepared by melting & mixing the ingredients in the above amounts. When the mixture is uniform and free from any visible impurities, it is cast in the form of slabs not more than 2 inches thick, or in the form of pellets not more than 2"× 2" in size. Composition D-2 is used to emulsify & desensitize solid explosives or mixtures of explosives. The detailed US Military requirements of Composition D-2 are given in Ref 2, where the tests are also described

An example of use of Comp D-2 in expls is given in Ref 1. The expl, designated **HBX-1,** contains 5% of Comp D-2; other ingredients are: Comp B 70, TNT 12 & powdered Al 18%

Refs: 1)Anon, "Military Explosives", TM **9-1910** (1955) 2)US Military Specification **MIL-C-18164(NORD),** "Composition D-2" (4 November 1954 with Amendment 1, 18 July 1957)

Composition d'amorçage (Fr). Primary composition

Composition, Delay. See Delay Composition

Composition EL-387A & EL-387B. The Eastern Laboratory of the Du Pont Company developed two slurry-type expls which could be poured on or pumped into continuous tubes of varying sizes down to 1" in diam. The compn & props of these expls are as follows:

Composition, %	**EL-387A**	**EL-387B**
50/50 Pentolite	30.0	24.0
Ammonium nitrate	54.0	43.2
Water	13.5	10.8
Starch	2.5	2.0
Aluminum (MD101)	-	20.0
Properties:		
Density, g/cc	1.36	1.50
Triton value (see Note)	10.9	13.4
TNT equiv (underwater blast measurements), lb	0.856	1.207

Prapagation in tubes 1" by 50'	OK	OK
Sensitiveness:		
Air gap, 1¾" diam	12"	11"
1" diam	2"	0"
Water gap, 1¾" diam	4"	4"
1" diam	½"	0"
Cap sensitivity		
Eng Spec No 8	Detonated	Detonated
No 6 EBC	-	Failed
Thermal stability	Brown fumes at 150°C Ignited at 170°-180°C	Brown fumes at 150°C Ignited at 170°-180°C

Note: Accdg to C.H.Winning of E.I. du Pont de Nemours & Co., **Triton Value** (called in literature TNT value) indicates the number of grams of TNT that will impart the same amount of swing to the Du Pont ballistic pendulum as 10g of a test explosive. Thus a Triton value of 10.9 signifies that 10g of a test explosive is equivalent in effect to 10.9g of TNT. On dividing 10 by 10.9 and multiplying the result by 100, the value of 92.7 is obtained, which indicates that the tested expl is 92.7% as strong as TNT

Pentolite was used as the sensitizer for the expl slurry because it detonates dependably in water. Ammonium nitrate was used as a source of strength, and also because its solubility in water increases very rapidly as the temp is raised. Both of the above compns were mixed & poured at 60-70°, or just below the mp of pentolite. On cooling to RT both slurries stiffened due to crystn of AN. The column of expl was flexible to -30°, but sufficiently solid so that settling of the solid ingredients did not occur. Addition of starch prevented seepage of liq at RT & above

These water-slurry compns were satisfactory with respect to propagation, strength, stability, and failure to be exploded by rifle bullet impacts. Hoses loaded with them withstood rough handling and were flexible & easily manipulated. Explosive streamers, for the Mark 29 Mine, loaded with these expl slurries were unsatisfactory only with respect to their densities. The overall density of a Mark 29 streamer has to be 1.04g/cc for proper buoyancy. However, nonbuoyant streamers loaded with Compositions EL-387A & EL-387B might be useful line-demolition chges, particularly for underwater purposes. Also these expls might be well suited for use as "drone" (qv) chges, where the max wt of expl could be loaded in min time in some type of carrier which is to be run up to the firing point by remote control & detonated *en masse*

Ref: Anon, "The Preparation and Testing of Explosives", Summary Technical Report of Division 8, NDRC Vol 1 (1946), 61-62

Composition, Explosive. See Explosive Compositions; Black Powder, Vol 2, p B165-R; Blasting Explosives, Vol 2, pB202-L; and under individual explosive compositions

Composition, Flash. See Flash Compositions

Composition fusante (Fr). Time Train

Composition H-6 Explosive. See H-6 Explosive

Composition, Igniting. See under Igniting Compositions

Composition incendiaire (Fr). Incendiary Composition

Composition, Incendiary. See Incendiary Compositions

Composition, Initiating. See Initiating Compositions

Composition "K" Igniter. See under Igniters for Tracers and Their Compositions

Composition, Propellant. See CANNON PROPELLANTS, Vol 2, p C29-Lff; COMPOSITE PROPELLANTS in this Vol; and others

Composition, Pyrotechnic. See Pyrotechnic Compositions

Compositions, Explosive (British). Explosive compositions designated Type A-PN & SR; Type B-PN & SR; Type C-PN & SR; Type D-PN & SR; and Type E-PN are listed, but their formulations are not reported, in classified British Report "Comprehensive List of

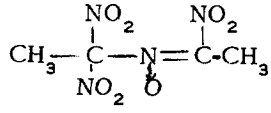
Government Explosives 1955", Admiralty BR 819 (IB/54)

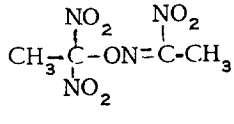
Composition T-9. A Composite Propellant, also called **CP-492.** See under Composite Propellants, p C468-L, Table
Refs: 1)Anon, "Summary Technical Report of Division 8", NDRC Vol 1, (1946), 96 2)Anon TM 9-1910 (1955), 260 3)R.P.Antonelli, "Encyclopedia of Explosives", OTIA, Durham, NC (1960), 31

Composition, Tracer. See Tracer Compositions

Compound 335, $C_4H_6N_4O_7$; mw 222.12, N 25.23%, O bal to CO_2 - 29%; expl crysts, mp 122°, d 1.62 g/cc; heat of comb 2495 cal/g & 545 kcal/mole with w liq at const vol; ignition temp for 50% value by Hot Bar 222°; impact sensitivity for 50% explosion with 2 kg wt 21 cm; power by Ballistic Mortar 144% TNT & by Lead Block 151% TNT; and vac stability value at 100° 0.98 cc/g/48hrs

This very interesting expl substance was previously misinterpreted by several workers as a tetranitrobutane. Its structure & reactions were the chief concern to Dr. W.L. Gilliland & Dr W.E.Taft of the Purdue Univ Research Foundation. Its crystallography was explored by Dr. W.C.McCrone at Armour Research Foundation; and its prepn was studied by Dr. C.R.Morgan & Mr. T.U.Hall, of Arthur D. Little, Inc. All of these investigators report a structure which is consistent with one of the following:





Compound 335 has been prepd in 25-30% yield by several laboratories from either an aq soln of the K salt of 1,1-dinitroethane or a mixt of the K salt with ammonium persulfate. Further details of the prepn of Compound 335, its reactions with anions, and other props are given in the Ref

This substance is considered to be a powerful expl and of excellent stability. It may be of interest to workers in the field of pure high explosives
Ref: Anonymous, "Second Report on Synthesis and Testing of High Explosives", Arthur D. Little, Inc Contract Nos W-19-020-ORD-6436 & DA-19-020-ORD-12 (March 1951), pages 17, 30, 65, 91, 255, 274 & 345

Compound B. See S. Marantz et al, NBS Report **7 B 101** (March 1964) (Conf)

Compound $C_6H_5N_3O_3$; mw 167.12, N 25.15% was obtd by Quilico & Fusco (Ref 2) as a white flocculent ppt, on treating the basic compd of probable formula $O.N:C(CH_3).CH:C.NH.CO.CH_2.NH_2$ with Na nitrite in AcOH. The basic compd was one the products obtained by Q & F during investigation of reactions betw citraconic (methylmaleic) acid and nitric acid. The dried compd $C_6H_5N_3O_3$ exploded violently on heating. It is sol in cold aq alkalias & alk carbonates and is decompd by hot alkalies & mineral acids with the evoln of gas
Refs: 1)Beil - not found 2)A.Quilico & R. Fusco, Gazz **66,** 286 (1936) & CA **31,** 1806 (1937)

Compound or Composite Detonators. See under Detonators

Compressed Air Blasting Devices. The possible use of blasting devices, activated by compressed air, to improve safety in blasting coal was considered long before many other devices (Ref). The two systems in use *Airdox* & *Armstrong Airbreaker* are listed in Vol 1 of this Encycl, pages A117-R & A485-L. See also *Cardox* in Vol 2, p C67-R

See under COMMERCIAL OR INDUSTRIAL EXPLOSIVES
Ref: Taylor & Gay (1958), 137

Compressed Nitrocellulose. Guncotton, molded into blocks of regular shape by means of high pressure, was formerly used for military pur-

poses. NC containing about 18% moisture was used in Russia beginning in 1885 as a bursting chge in shells, mines & torpedoes. Later the use of NC was extended to the prepn of small demolition chges intended for the destruction of rails, bridges & small structures. Moisture was added to NC because the dry material is extremely sensitive to shock & friction. However, when the moist NC blocks froze, they became very sensitive and dangerous to handle. The highly compressed NC (d=1.6 g/cc) was known in Russia as *Slonit.* This Slonit was also used as a propellant. With the development of gelatinized NC proplnts, Slonit proved inferior and its use was discontinued. Compressed NC blocks as HE charges were gradually replaced by compressed Picric Acid (in Russia about 1896) and then by TNT (beginning about 1907). However, some NC-compressed shells were still used during the Russo-Japanese War of 1904-1905, and in some Whitehead torpedoes & demolition chges as late as WWI (Ref 2)

See also CELLULOSE NITRATES in Vol 2, p C100-L

Refs: 1)O.Guttmann, "The Manufacture of Explosives", Whittaker & Co, London, Vol 2 (1895), 33ff 2)B.T.Fedoroff et al, "Dictionary of Russian Ammunition and Weapons", PATR **2145** (Feb 1955), pp 12 & 21

Compressibility of Explosives. The compressibility of a solid explosive is a measure of the change in vol caused by the application of pressure. Compressibility is the relationship between the density and the pressure applied to a granular or powdered expl. The resulting curve is useful where the loading of ammunition or of pellets is done by compression

The procedure to determine this property is to weigh to the nearest 0.1g a sample of the granular expl and transfer it to a steel mold. By means of a hydraulic press, the sample is compressed at a predetermined pressure, and its density is determined. Repeat this operation using new samples and varying pressures. The data are plotted as pressure vs density and a line is drawn between the points to show the compressibility relationship

Refs: 1)A. LeRoux, MP **32**, 122 (1950) 2)M. Ya. Vasil'ev et al, ZhFizKhim **34**, 2454-59 (1960) & CA **55**, 10889 (1961) (Isothermal compressibility of explosives under pressure) and Explosivst **1963**, 275-R

Compressibility of Propellants. When investigating the phenomenon of burning of propellants under high pressure (as occurs in the bore of a weapon), no consideration was made, until recently, of the fact that surfaces of proplnt grains are sl smaller at high pressure than at atmospheric pressure. This is due to shrinkage of the grains. If the surface shrinks appreciably, such as 12-12% at 3000 kg/sq cm, as claimed byCorner (Ref 1) for British SC Propellant, then it is necessary to introduce corrections in formulas where surface is one of the variables

Tavernier & Prache (Ref 2) investigated the compressibility of French solventless powders (poudres SD) and of Ballistites. Their shrinkage was only 3-4% at pressures up to 3000 kg/sq cm. This value is considered sufficiently small so as not to affect the calculation of vivacity of proplnts. See also Ref 4

The compressibilities & specific heat ratios of some rocket propellants liquids such as hydrazine, JP-3, JP-4 & WFNA (white fuming nitric acid)] are reported by Kretschmar (Ref 3)

The term compressibility is also applied to a std test method for detg the percentage of compressibility of proplnt grains (Ref 5). The method consists of removing both ends of several grains (10), compressing the center portions until disintegration begins, and calculation the average percentage of compression as follows:

$$\% \text{ Compressibility} = \frac{100(A-B)}{A}$$

where A = av length of uncompressed grains and B = av length of compressed grains

Refs: 1)Corner, Ballistics (1950), 66ff 2)P. Tavernier & P. Prache, MP **35**, 223-31 (1953) & CA **49**, 12831 (1955) (Compressibility of solventless propellants under high pressure) 3)G. Kretschmar, Jet Propn (now ARS) **24**, 175-79 (1954) & CA **48**, 11062 (1954) (Isothermal compressibilities of some rocket proplnt liquids) 4)A.Louiton, MP **38**, 243-65 (1956) & CA **51**, 15131 (1957) (Study of the compressibility of SD propellants) 5)Anonymous, "Propellants, Solid: Sampling, Examination and Testing", Military Standard **MIL-STD-286A** (April 1960) method 505.1

Compression, Effect on Sensitiveness of Initiators. This subject was studied by Ubbelohde & Woodward (Ref) who found that compression reduces the "induction period", but the values E and B of the equation:

$$\log Y = \frac{E}{4.57T} + B$$

where Y = induction period, E = activation energy, B = constant, and T = absolute temp, are affected differently by different initiators. Compression also lowers the "Threshold temperature" below which a given mass of initiator will not detonate. Thus in all cases the sensitiveness to heat is increased by compression. On the other hand compression increases the sensitiveness of LA to flash & percussion, but it decreases these properties for MF

See also Delay to Ignition and Its Temperature Coefficient

Ref: A.R.Ubbelohde & P.Woodward, PhilTr **241A**, 222-37 (1948)

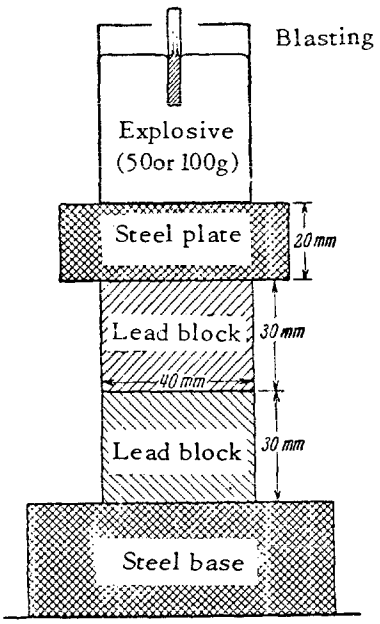
Compression of Solids by Strong Shock Waves. Title of an article in "Solid State Physics", Vol **6**, Academic Press, NY (1958), pp 1-63

Compression (or Chrusher) Test for Determination of Efficiency of Detonators. See Esop's Test in Vol 1, p XI of this Encycl.

Compression (or Crusher) Tests for Determination of Brisance. The tests described here are designed to measure the violence of an explosion effect, called brisance or shattering effect. A brief discussion on brisance is given in Vol 2, p B265ff of this Encyclopedia and a brief description of test is given in Vol 2, p B299

In addition to this information, a more detailed description, including illustrations, of tests utilizing compression of soft metals (such as lead & copper) is given below,

A. *Lead Block Compression Test* **(LBCT)**, known also as *Hess' Crusher Test* (Stauchprobe nach Hess, in Ger). The apparatus used for this test, called "Brisance Meter of Hess", (Stauchmesser nach Hess in Ger), was developed in 1879 by an Austrian scienties, named Hess. The apparatus (See Fig, which is a reproduction of Fig 236, p 365, Ref 10), consists of two cylindrical lead blocks 40mm in diam & 30mm high. The lower block rests on a steel base, while the upper



BRISANCE METER OF HESS

one is covered with a steel plate, 20mm high. On top of this is placed a charge (50 or 100g) of explosive to test, provided with a blasting cap. No tamping was used in Austria

After firing the charge, the height of each lead block is measured and subtracted from 30mm. The more the cylinder is compressed, the higher is the brisance. For example, Stettbacher (Ref 10, p 367), gives the following values:

	Compression in mm			
Explosive	Upper Block	Lower Block	Total	Mean
TNT	19.1	12.9	32.0	16.0
Gelatin-Dynamite (65%)	23.1	18.0	41.0	20.6

Lead block compression test (LBCT) values of various expls, expressed as percentages of TNT value, are listed in Vol 2, Table 1, pp B266 to B 295 of this Encycl

The German modification of Hess' apparatus was developed by the Dynamit AG at Schlebusch. It contains only one lead block, 40mm in diam & 65mm high. Here the charge of expl varies betw 100 & 300g (Ref 10, p 365) (See also Refs 3, 5, 7, 13, 14 & 15)

In the French version of Hess' test, known as *epreuve des petits plombs*, which was developed by the CSE (Commission des Substances Ex-

plosives), one lead block 40mm in diam & 50mm high is used. The charge of expl is 45g and it is initiated by a No 7 cap (wich is equal to 1.5g MF) (Ref 9, p 190)

Another Fr modification of Hess' app is *l'appareil de Chalon* Ref 1, p 452). It is quite similar to Hess' apparatus, except that the steel disk covering the upper block is 3mm thick (instead of 20mm). A charge of Expl 40 to 50g, contained in a zinc box, (provided with a perforated cover) is exploded on top of that steel disk. This produces a mushroom-like compression of the upper lead block, (which is assumed to be proportional to brisance), and a flattening of the lower block (which is assumed to be proportional to potential)

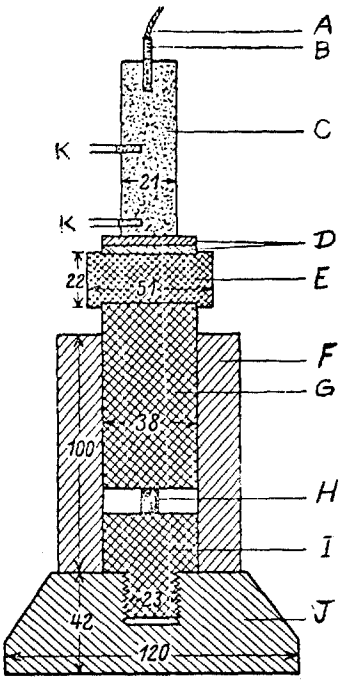
In the US modification of Hess' test, which has been used by Bureau of Mines under the name of *Compression Test with Small Lead Block*, one lead block is used. It is 38mm in diam & 64mm thick and is placed upon a rigid steel support imbedded in concrete. The block is covered with a disk of annealed steel, 38mm in diam & 6.4mm thick. On top of the disk is placed a 100g charge of expl to test, provided with a No 6 blasting cap. Detonation of the expl compresses the block appr proportionally to the brisance of expl (Ref 6, pp 107-08)

B. *Copper Cylinder Compression Test* **(CCCT)** known also as *Kast's Crusher Test* (Stauchprobe nach Kast in Ger). The apparatus used for this test, called "Brisance Meter of Kast" (Stauchmesser nach Kast in Ger), was developed in 1913 by Kast. It uses a small copper cylinder, H, for measuring the compression produced on expln of charge placed on top of the arrangement shown in Fig (which is a copy of of Fig 238, p 367 of Ref 10)

This method has been claimed to give more reproducible results than Hess' method (Ref 10, p 365) (See also Refs 2, 3, 5, 7, 8, 9, 11, 13, 15; and Vol 2, p B299-L of this Encycl)

Following are some copper compression values in mm as given by Kast (Ref 4, p 184): Blasting gelatin 4.8, Dinitrobenzene 2.9, Guncotton (dry) 3.0, Hexanitrodiphenylamine 4.1, Nitroglycerine 4.6, Picric acid 4.1, Tetryl 4.2, Trinitroanisole 3.5, Trinitroanisole 3.6, Trinitrobenzene 4.1, Trinitrocresol 3.5, and Trinitrotoluene 3.6

Copper cylinder compression test, CCCT, values, of various expls, expressed as percentages of TNT value, are listed in Vol 2, Table

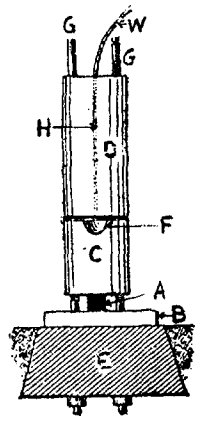


BRISANCE METER OF KAST

A – Fuse
B – Blasting cap
C – Explosive charge (at least 70 mm high)
D – Lead disks, each 2 mm thick
E – Nickel steel block, 20 mm thick & weighing 320g
F – Hollow steel or cast iron cylinder
G – Steel piston weighing 680g
H – Copper cylinder (crusher), 7 mm in diam & 10 mm thick, placed exactly in the center
I – Steel anvil
J – Steel or bronze base block
K – No explanation given in Refs

1, pp B266 to B295 of this Encycl

C. *Compression Test by Quinan's Method.* This test permits detn of not only brisance, but also of potential (strength). It is described in Ref 1, p 454; Ref 9, pp 192-93; Ref 11, pp 63-4 & Ref 11a, p 118. The method also is briefly discussed in Vol 1, p XXI of this Encycl. The following fig of apparatus will make it easier to understand the method



QUINAN APPARATUS

A – Solid lead cylinder. 25 mm in diam & 25 mm high
B – Steel plate, ca 25 mm thick
C – Solid steel cylinder 100 mm in diam & 128 mm high
D – Solid steel cylinder, 100 mm in diam & 254 mm high
E – Cast iron base block imbedded in cement
F – Cavity for sample (1-2g) and priming compn (0.25g)
G – Guiding posts provided on the upper parts with ratchets
H – Hole in the center of cylinder D thru which the electric wires, W, are conducted

The following values, expressed in percentages of NG, were detnd by Quinan method and are listed In Ref 11, p 84: NG 100, Blasting gelatin 106, Guhrdynamite (75%) 82, Guncotton (d 1.2) 83, MF 50 & Bk Pdr 28%

Refs: 1)Chalon(1911),451-52 2)H.Kast, SS **8**, 88-90 (1913) (Detn of Brisance by Kast's method) 3)Marshall **2** (1917), 497 4)H.Kast, SS **15**, 184 (1920) (Table of brisance values by Kast's method for various expls) 5)K.F.Meyer, SS **24**, 6-10 (1929) (Detn of brisance by Hess' & Kast's methods) 6)C.E.Munroe & J.E.Tiffany, "Physical Testing of Explosives", USBurMines Bull **364** (1931), pp 107-08(Compression test using lead blocks) 7)Sukharevskii & Pershakov (1932), 120-21 (Compression tests) 8)Marshall **3** (1932), 155 (Kast's compression test) 9)Vennin, Burlot & Lécorché (1932), 190-91 10)Stettbacher (1933), 365-67 11)Pepin Lehalleur (1935), 63-4 11a) Pérez Ara (1945), 118 (Quinan test) 12)Vivas, Feigenspan & Ladreda **4** (1945) - not found 13)Carpio **1** (1948), 64-7 (Hess' & Kast's tests) 14)Belgrano (1952), 39-43 (Hess' test and its modifications) 15)Giorgio (1964), 82-4 (Hess' & Kast's tests)

Compression (or Crusher) Tests for Determination of Pressures Developed by Explosives. See under CLOSED BOMB (OR VESSEL) AND INSTRUMENTS etc.

Compressive and Elastic Properties of Solids under Explosive Attack. Title of a chapter by M.A.Cook, "The Science of High Explosives", Reinhold, NY (1958), pp 202-25

Compressors are, in general, devices for compressing air, gases or solids (in the last case, they are also called presses). Compressors also serve for transferring gasses and vapors

Devices for compressing gases are used extensively in the chemical industry (see "High Pressure Techniques"), eg, in the manufacture of nitric acid by the ammonia oxidation process

Compressors for transferring gases and vapors may be divided into *vacuum pumps* or *exhausters* (when the gas is below acmospheric pressure) and into *fans, blowers, boosters* and *axial compressors* (when the gases are above atmospheric pressure)

Reciprocating compressors are the type most widely used in the chemical industry. They are available in industry from a fraction of a horsepower to 3000 hp and for pressures rrom 5 to 25000 psi

Other types are rotary compressors and turbocompressors

Ref: R.H.Perry et al, edit, "Chemical Engineers' Handbook", McGraw-Hill, NY, 4th Ed (1960), section 6.15

Comprimée, Poudre (Fr). Compressed propellant

CONAD. Abbr for Continental Air Defense Command.

CONARC. Abbr for Continental Army Command

Concrete in Ordnance Use. See under Cement and Concrete in Ordnance, Vol 2, p C125-R of this Encycl

Concrete Test. Under this name Marshall (Ref 2) lists the test described by Vennin & Chesneau (Ref 1). The test is similar to Trauzl Test (See Vol 1, p XXV of this Encycl) but instead of lead blocks 200mm in diameter and height, concrete blocks 500mm in diam & height are used. Charges of expls to test vary betw 5 & 10g. The results obtained are not as readily expressed in figures as with Trauzl Test (Ref 2)
Refs: 1)L.Vennin & G.Chesneau, "Les Poudres et Explosifs", C.Beranger, Paris (1914) 2)Marshall **2** (1917), 473

Concussion. A sharp or hard knock, blow, or collision. The shock of such a blow or a stunning, damaging, or shattering effect from a blow (Ref 4)

When a high expl detonates the effects may be divided into several phases (Ref 2): a)The phase in which the expanding gases cause the case to swell and fragment is called the *Fragment Effect*; b) The *Blast Effect* is the artificial gale set up by the rush of gases which form a compression wave in the surrounding medium. The accordian-like action of the air, being successively compressed & expanded until the wave dies down, is called *Concussion*; c)The high temps produced by all explosions have an *Incendiary Effect* on everything combustible at or near the point of explosion

See also Refs 1 & 3
Refs: 1)Meyer (1943), 393-94 2)All & En Expls (1946), 10-12 3)Clift & Fedoroff, Vol **4** (1946), 17 4)Merriam-Webster's (19619, 472

Concussion Fuze. A bomb fuze designed to function in air in response to the concussion produced by the explosion of a preceding bomb. Also called *airburst fuze* or *air pressure fuze*
Ref: OrdTechTerm (1962), 132

Concussion Grenade. A grenade that relies for its effect on the blast of its detonation rather than the fragmentation of its case, and is often designed to stun rather than kill (Ref 1)

The term is also applied to an offensive hand grenade having a nonmetallic container, designed to kill or injure by blast & concussion. It is distinguished especially from a fragmentation or defensive grenade. The offensive grenade is so called because the thrower, being out of range of the grenade's effects, can continue to advance as he throws, and does not have to take cover (Ref 2)
Refs: 1)Merriam-Webster's (1961), 472 2)OrdTechTerm (1962), 144

Concussion Primer. This term is applied to the primer of the so-called concussion element of an inertia-operated device in a fuze which functions when the projectile starts on its flight
Ref: Davis (1943), 454

Condensation Product of EDNA, Formaldehyde & Ammonia. On the basis of a British report, the condensation product of EDNA, formaldehyde & ammonia was nitrated to yield a compd (mp 167° dec 208° & nonhygroscopic) which was reported to be of interest as an explosive. The structure of the compd resembled that of RDX. It appeared, from its physical & expl props, to offer no advantage over Tetryl or RDX as an expl. The un-nitrated condensation product was indicated to be of interest for use in pyrotechnic compns
Ref: H.A.Aaronson, "Preparation and Properties of the Nitrated Condensation Product of Haleits, Formaldehyde and Ammonia", PATR **1600** (May 1946)

Condensed Electric Spark, Detonation of Explosives by. Basset & Basset (Ref) found that small pellets of PETN & RDX, pressed at 5000 kg/cm^2, are detonated by a spark from the discharge of a condenser at 2000-12000 volts, although a spark at the same voltage without a condenser causes only burning. By measuring the capacity of the condenser, it was determined that RDX requires more energy than PETN for detonation. "Deal pressed" MF & LA can also be detonated by the electrical discharge from a condenser
Ref: J. Basset & J. Basset, CR **231**, 759-61 (1950) & CA **45**, 1767-68 (1951)

Condensed Explosives. This term applies to both solid & liq expls (such as TNT, PA, Tetryl, RDX, NG, NGc & DEGDN) which have been reduced to a denser form by casting, compression of crystalline particles or of a liq to a smaller volume

The detonation of condensed expls was studied in Russia by Landau & Stanyukovich (Ref 1)

who showed that numerous so-far unsolvable discrepancies arise from the application of the equation of state which forms the basis of the modern theory of detonation of condensed expls. The authors assert that the density of the expl is sufficiently high as to make application of the gas law impracticable. They suggest comparing the detonation decomp products with a liq whose particles are in a state of oscillation; this conditions the course of expansion of the decomp products (Ref 1)

Merzhanov & Dubovitskii (Ref 4) formulated a general theory for the thermal explosion of condensed expls, which takes into consideration the removal of particles from the reaction volume. This theory makes it possible to calc all the basic characteristics of thermal explosion such as critical conditions, depth of preëxplosion decompn & induction period

"Detonation is Condensed Explosives" is the title of a book by J. Taylor (Ref 3) who discusses in detail the various aspects of the subject. See also studies & reports listed as Refs 2, 5 & 6

Refs: 1)L.D.Landau & K.P.Stanyukovich, DoklAkadN **46**, 396-98 (1945) & **47**, No 4, 273-76 (1945); CA **40**, 4523 & 4217 (1946) 2)G.Morris & H.Thomas, "On the Thermochemistry and Equation of State of the Explosion Products of Condensed Explosives", Research (London) **1**, 132-44 (2947-48) 3)Taylor (1952) 4)A.G. Merzhanov & F.I.Dubovitskii, DoklAkadN **124**, 362-65 (1959) & CA **55**, 5955 (1961) 5)L.Rudlin, "On the Origin of Shockwaves from Condensed Explosions in the Air. Part 2 Measurements of Airshock Pressures from 8-lb TNT Spheres of Various Densities at Ambient Pressures", Naval Ordnance Laboratory Technical Report **NOLTR 63-13** (Jan 1963) 6)Pvt E-2 M.A.Neiverth, "Soviet Research on Liquid Explosives in the Last Decade", US Army FSTC Report (March 1964)

Condensed Powders. See under Bulk and Condensed Powders, Vol **2**, p B322-R

Condensed Polymerization. A polymerigation in which atomic displacement occurs to some extent, as in aldol polymerization and benzoin formation

Ref: Hackh's (1944), p 672

Condenser Chronograph. (Kondensatorchronograph in Ger). An app for ballistic measurements invented in 1876 by R. Sabine and improved in 1900 by M. Radaković. See also CHRONOGRAPHS, p C304-R

Ref: Cranz **3** (1927), 101-18

Condensers. These are devices used for: a)concentrating matter or energy b)cooling vapors to liquids c)polymerization of org compds. Electrical condensers consist of insulated conductors which accumulate electricity. Optical condensers are instruments consisting of lenses or mirrors which condense light beams

Ref: Hackh's (1944), 220

Conductive and Convective Heat Transfer, Thermal Explosion by. There are three fundamental types of heat transfer: *conduction, convection & radiation.* All three types may occur at the same time, but it is advisable to consider the heat thransfer by each type in any particular case. *Conduction* is the transfer of heat from one part of a body to another part of the same body, or from one body to another in physical contact with it, without appreciable displacement of the particles of either body. *Convection* is the transfer of heat from one point to another within a fluid, gas or liquid, by the mixing of one portion of the fluid with another. In natural convection, the motion of the fluid is entirely the result of differences in density resulting from temp differences; in forced convection, the motion is produced by mechanical means. *Radiation* is the transfer of heat from one body to another, not in contact with it, by means of wave motion thru space (Ref 5)

Merzhanov et al (Ref 1) detd the critical conditions for the thermal detonation of Tetryl. The exptl values of the critical temp lie between the values calcd for purely conductive & purely convective heat transfer, indicating the complex nature of the transfer in this case. It was concluded that the effective heat transfer coefficient increases during the decomp process due to the stirring of liq phase by the gaseous products which are involved

A quasi-stationary reaction condition is assumed when accumulation of heat in the system is small relative to the heat conducted away. The sl shifting equilibrium between the heat evolved & the heat dissipated is considered to be composed of a number of stationary states. The theory of quasi-stationary reactions is discussed using an example of an autocatalytic re-

action of the 1st order (Ref 2)

Merzhanov et al (Ref 3) also made an investigation of the thermal explosion of Dinitrohydroxydiethylnitramine (mp 52.5°) and of Tetryl (mp 130°) in the liq phase under conditions where the reaction zone was free of temp distribution and the entire thermal gradient was at the walls of the container. The method & exptl setup were the same as previously used by the authors (Ref 1). The activation energy, thermal effect & critical temp were reported for both expls (Ref 6)

The critical conditions of thermal spontaneous ignition, for a body of arbitrary shape, when cooling is accomplished by both convection & conduction are analyzed by Grishin & Todes (Ref 6). Although exact nonlinear equations are reqd, it is possible to solve the approx linear homogeneous equations for the conventional mode of cooling. The authors report that the thermal relaxation time is, in many cases, more easily detd experimentally according to the model based on nonreacting substances with suitable geometric & thermal parameters (Ref 4)

Refs: 1)A.G.Merzhanov et al, DoklAkadN **128**, 1238-41 (1959) & CA **55**, 24013 (1961) 2)A.G. Merzhanov & F.I.Dubovitskii, ZhFizKhim **34**, 2235-44 (1960) & CA **55**, 18110 (1961) 3)A.G. Merzhanov et al, ZhFizKhim **35**, 2083-89 (1961) & CA **56**, 6230 (1962) 4)A.M.Grishin & O.M. Todes, DoklAkadN **151** (2), 365-68 (1963) & CA **59**, 12585 (1963) 5)R.H.Perry et al, Edit, "Chemical Engineers' Handbook, McGraw-Hill NY, 4th Ed (1963), Section 10-2 6)Pvt E-2 M. A.Neiverth, "Soviet Research on Liquid Explosives in the Last Decade", US Army FSTC Report (March 1964), p 8

Conductive Rubber. The history of conductive rubber dates back to 1882, when graphite was proposed as a conductor in rubber. Since that time considerable work has been done by various investigators in developing conducting rubber, both natural & synthetic. The electrical resistivity of such rubbers may be as low as 100 ohm-cm. A recent technique of incorporating the black (acetylene black) into a rubber solution or cement has been shown to produce rubbers of very high conductivity, having resistivities as low as 1 ohm-cm (Ref 2)

Conductive rubber finds applications in ordnance for making the soles of shoes, conductive floor mats & in making various buckets, paddles, containers & table tops where certain expls are handled. Conductive floor & conductive shoes are reqd for grounding all personnel at operations where expl(such as primers, initiators, detonators, ignitors, tracers & incendiary)mixts are exposed. Some materials sensitive to static sparks are LSt, La, MF, Tetrazene, Diazodinitrophenol, $KClO_3$-LSt mixts, igniter compns, Grade B Mg powd and BkPdr dust when exposed in layers. Dust of solid proplnts can also be ignited from the spark that can accumulate on a person, and conductive floors & shoes must be used when the dust is present. In addn, dust-air mixts of Amm Picrate, Tetryl, Tetrytol & dust of solid proplnts are sensitive to static elec discharges. Many flammable liquids & air mixts (ether, ethyl alcohol, ethyl acetate, acetone & gasoline) can also be ignited by static discharge from a person (Refs 1 & 3)

The US Navy also used conductive rubber blankets over fire control equipment to prevent fogging & freezing during cold weather. The elec current carried by the blanket produced enough heat to keep the equipment in operation (Ref 1)

Refs: 1)L.Gilman, "Plastics Developments", PA Tech Div Lecture (Jan 1947), p 6 2)Kirk & Othmer **3** (1949), 68 3)Anonymous, "AMC Safety Manual", Headquarters, US Army Material Command, Washington, DC (June 1964)(A reprint of AMCR 385-224, formerly ORD M7-224), pages 7-4

Conductivity, Electrical; Conductometry and Conductometric Titrations. Electrical conductivity is the quality or ability of a substance to transmit electrical energy. If it deals with the conductivity of an electrolyte in solution, it is then called *electrolytic conductivity. Conductometry* deals with analyses by measuring electrolytic conductivity, based on the fact that ionic substances in many solvents conduct electricity. *Conductometric titrations* are quantative analysis based on the fact that with the addn of the titrating agent to a soln being titrated, the *specific conductivity* (reciprocal of specific resistance in mhos) changes at a different rate before and after the end point (Comp with Potentiometric Analysis)

Refs: 1)Kirk & Othmer **4** (1949), 325-33 (Conductometry) 2)W.G.Berl, Edit, "Physical Methods

in Chemical Analysis", Academic Press, NY, Vol 2 (1951), 51-104 (Conductometry) 3)J.J. Lingane, "Electroanalytical Chemistry", Interscience, NY (1958), Chap IX (Conductometric Analysis) 4)A.Weissberger, Edit, "Physical Methods of Organic Chemistry", Vol1, Pt 4, Interscience, NY (1960), Chap 45 (Conductometry) 5)Vogel (1961), 969-87 (Conductometric titrations)

Conductivity, Thermal. This term is applied to the property of a substance to transmit heat. The *coefficient of thermal conductivity* also called *specific heat of conductivity* is the quantity of heat in gram calories transmitted per second thru a plate of the material one cm cube, when the temp difference between the two sides of the plate is one degree C (Ref 1)

The thermal conductivity values of some common explosives, in cal/sec/cm/°C, are as follows (Ref 2):

Ammononium Nitrate	3.4×10^{-4}
Cyclonite (RDX) (d 1.53)	6.98×10^{-4}
DBX (AN/RDX/TNT/Al-21/21/40/18) (d 1.75)	13.2×10^{-4}
Lead Azide	1.55×10^{-4}
Mercury Fulminate	1×10^{-4}
Minol 2 (AN/TNT/Al-40/40/20) (d 1.74)	16.5×10^{-4}
Picric Acid (d1.41)	6.24×10^{-4}
Tetryl (d 1.53)	6.83×10^{-4}
TNT (d 1.54)	5.6×10^{-4}
Torpex (RDX/TNT/Al-42/40/18) (d 1.82)	9.7×10^{-4}
Tritonal (TNT/Al-80/20) (d 1.73)	11×10^{-4}

Refs: 1)Hackh's (1944), 220 2)W.R.Tomlinson & O.E.Sheffield, "Properties of Explosives of Military Interest", AMC Pamphlet 706-177 (August 1963) (Reprint of ORDP 20-177 & PATR 1740) (Data from other sources are cited in Ref)

Conductometric Method of Analysis of Nitric-Sulfuric Acid Mixtures. See Y. Lacroix & M. Chaverou, MP **41**, 423-39 (1959)

Conductometry and Conductometric Titrations. See under Conductivity, Electrical

Cone-charge Effect. One of the Brit names for Shaped Charge Effect

Cône de charge. Fr for Warhead (of a torpedo)

Confined (or Cased) Charge. See under CHARGE in Vol 2, p C150-L of this Encycl. The unconfined chge is known as a *bare charge*

Confinement, Effect on the Detonation Waves in Solid Explosives.
Ref: L. Deffet & J. Boucart, "The Effect of Slight Confinement on the Detonation Waves in Solid Explosives", a paper reported in the 6th Symposium on Combustion, Reinhold, NY (1957) pp 642-48 (10 Refs)

Congreve, Sir William (1772-1828). British artillery General, inventor of incendiary rockets which were used with success in the battle of Boulogne (1806), in the battle of Copenhagen (1807), during the British-American War (1812), and up to about 1960. He also designed a new gun for the ship, Frigate
Refs: 1)Websters Biographical Dictionary (1959), 339 2)Rocket Encycl (1959), 101

Coniferol (Coniferyl Alcohol or 4-Oxy-3-methoxy cinnamic Alcohol), $HO.CH_2CH{:}CH.C_6H_3(OH)$-$O.CH_3$; mw 180. 20, O 26.64%; col prisms, mp 73-74°; v sol in eth; sol in alc & alkalies; difc sol in hot w, almost insol in cold w; prepn & other props are given in Beil (Ref 1). It is supposed that four moles of coniferol condense to form Lignin, a cellulose-like substance
Refs: 1)Beil **6**, 1131 & [1093] 2)Hackh's (1944), 221 3)Clift & Fedoroff **4** (1946), 17

Consistency of Commercial Explosives. Commercial expls of today are available in a free flowing form such as dry sand, representing one extreme, and in a form as cohesive as raw rubber, at the other extreme. There are also hard plastic expls, such as Blasting Gelatin and some Gelatin Dynamites. Either extreme condition of consistency requires the sacrifice of some important consideration. The quality of being free flowing reduces the rate of detonation if the expl is used as a loosly packed charge. Rubbery consistency increases the cost of manufg the expl. Of the coml expls available today, the greatest efficiency & economy are obtd in high Ammonium Nitrate content expls, which are somewhat cohesive, or in *Gelamites* which are cohesive but only slightly plastic

Refs: J.J. Berliner et al, "Explosives", Pamphlet **1592**, 840 Broadway, New York 3, NY (ca 1953)

Consistency of NC Test. Same as Viscosity Test of Nitrocellulose (See Vol 2 of this Encycl, p C122-R, test h)

Constants K_1 and K_2 of Muraour. In order to evaluate the *vivacity* [(dP/dt)/Pmax] of a colloidal propInt in which the surface of the grains remains constant during burning, Muraour proposed in 1931 to determine the value K_1 which is inversely proportional to the vivacity of a propInt

For the detn of the constant K_1, a charge of propInt is burned in a closed bomb (qv) at a loading density of 0.2g/cc and as the burning proceeds the pressure-time curve is recorded on the paper attached to the rotating drum of the pressure-time registering device. After reading the curve, a new curve is constructed on graph paper by plotting time (t) on the abscissa and Napierian logarithm of pressure (lnP) on the ordinate. This usually gives a straight line for pressures betw 500kg/cm^2 and for pressures ca 200kg/cm^2 below the maxium pressure, Pmax. In experiments conducted by Muraour et al, Pmax varied betw 2400 & 2500kg/cm^2. On dividing P max by the slope of the above straight line [d (lnP)/dt], (called "coefficient angulaire" in Fr), the constant K_1 is obtd. It is practically not influenced by variations in loss of heat thru the walls of the closed bomb and only very little influenced by changes in loading density, except for very low d's. Muraour also showed that K_1 is equal to the area under the pressure -time curve ($\int P dt$), where P = Pmax/2, assuming that burning proceeds under ideal conditions

On dividing K_1 by thickness (web size), in mm, of propInt grains manufd from the same cake (pâte or galette in Fr), the value K_2 is obtd. This value is found to be constant depending only on the composition of the propInt cake and is independent of shape or thickness of the grains

If the maximum pressure obtd on burning the propInt in a closed bomb is Pmax, the average pressure under which burning proceeds is equal to Pmax/2. It was found experimentally that for Pmax ca 2000kg/cm^2, the value $1000/K_2$ is a good approximation for the rate (expressed in mm/sec) at which the thickness of a propInt grain diminishes, when it burns simultaneously on two parallel surfaces. This is equal to twice the linear burning rate

The following table gives some K_1 & K_2 values for tubular, double-base, solventless propInt (the compn is not given), examined by Muraour et al:

Thickness of propInt grains, mm	Maximum pressure, Pm, kg/cm^2	K_1, (kg/cm^2) x sec	K_2, (kg/cm^2) x sec
1.36	2502	9.71	7.14
2.56	2454	17.6	6.90
3.83	2415	26.9	7.02
4.79	2416	31.0	6.90
5.68	2384	39.5	6.98

If the surface of the grains of a propInt does not remain constand during burning, the calcn of K_1 & K_2 is more complicated and is not given here

For more info on detn of coefficients K_1 & K_2 see refs listed below:
(This item was reviewed by Mr. L. Shulman of PicArsn)
Refs: 1)H.Muraous, "Poudres et Explosifs", Presses Universitaires de France, Paris (1947), 90-2 2)H.Muraour & G.Aunis, MAF **22**, 522-29 (1948) 3)Ibid, **23**, 859-66 (1949) 4)Ibid, **25**, 121-22 (1951) 5)H.Muraour & G. Aunis, MP **35**, 290-91 (1953)

Constants of Explosives and Propellants are the factors characterizing various expls. Important constants are brisance, detonation rate, explosion temperature, heat of explosion, power sensitivity to friction and impact. These properties are known also as Characteristics of Explosives and Propellants (See Vol 2, p C149 -L of this Encycl)

Constitution of Pure Explosives Compounds. The relationship between performance and constitution of pure organic expl compounds has been the subject of extensive studies by Lothrop & Handrick
Refs: 1)Anonymous, "Study of Pure Explosive Compounds", Part I. Correlation of Organic Structure with Explosive Properties from Existing Data (Jan 1947); Part II. Correlation of Thermal Qualities with Explosive Properties

(April 1947); Correlation of Composition of Mixtures with Performance (May 1950); Part IV. Calculation of Heat of Combustion of Organic Compounds from Structural Features and Calculation of Power of High Explosives (May 1952) (Contract Nos W-19-020-CRD-6436, DA-19-020-ORD-12, and DA-19-020-ORD-47) 2)W.C.Lothrop & G.R.Handrik, ChemRevs **44**, 419-45 (1949); Chim & Ind (Paris) **67**, 253-57 (1952); and Explosivst **1954**, 121-24

Constrained Shell Analysis. As applied to the design of rotating bands for artillery projectiles, an analysis that recognizes the possibility of band failure at one of three areas: the band land surface, the band land -groove fillet, and the inner surface of the band. This analysis provides formulas for checking each of these points
Ref: OrdTechTerm (1962), 82

Construction of Buildings and Installations for Explosives & Propellant plants. Technical safety considerations and practical experience in the planning and constructing of explosives & propellant plants are aspects of the subject discussed by Banik (Ref 1), See also Ref 2
Refs: 1)E.Banik, Explosivst **1956**, 130-34 & 155-58 2)Anon, "AMC Safety Manual" (1964), Sect 5-1, 5-10, 5-19, 18-3 & 18-4

Consumable Cartridge Cases. See under Combustible and Consumable Cartridge Cases

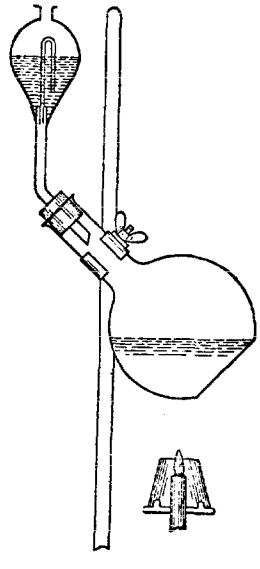
Contact Igniters. See under Igniters.

Containers for Explosives. Zimmer & Asaoka (Ref 1) have investigated various materials as candidates for fabricating containers that safely hold comparatively large loads of expl materials. The inspiration for this study came from earlier work at DuPont which led to the development of "tote barricades" to protect personnel, hand-carring sensitive material, from the effects of an accidental explosion. These barricades withstood the expln of 2 grams of NG. The new sample carrier, to be explosion-safe, was reqd to withstand a min load of 5g expl sample, and was to be fabricated from a material which would form harmless fragments upon detonation of the expl

In first developmental studies, containers were made with polyurethane. However, it was found (Ref 1) that medium density polyethylene is an ideal material to fabricate expl safety containers. Two different sizes of containers were developed by Zimmer & Asaoka: a 10-cm diameter container which can easily withstand the expln of 15g of NG; and a 32-cm diameter 500g of NG, if small detonators (DuPont S-67 squib) are used. The smaller container weighs 0.775 kg and is easily carried in one hand; the larger container weighs less than 22kg and is easily portable with carrying handles or in a cart

See also Ref 2
Refs: 1)M.F.Zimmer & L.K.Asaoka, Explosivst **1963**, 237-42 2)Anon, "AMC Safety Manual" (1964), Sect 13-12, 13-15, 13-16, 14-2, 20-4, 20-5 & 28-8

Contat-Göckel Valve. A Device which serves to prevent entrance of air from without into a closed flask. It consists of a small separatory funnel contg a soln of Na bicarbonate and provided inside with a thin syphon tube. The stem of the funnel is inserted in the stopper of the flask (Comp with Bunsen Valve described in Vol 2, p B341-R of this Encycl)
Ref: Treadwell & Hall, Vol **2** (1942), p 540



CONTAT–GÖCKEL VALVE

Continuous Digester for Cellulose. The equipment has been in use since 1949 by the Hercules Powder Co for producing chemical-grade cotton from linters. The apparatus was patented by Segl (Ref 1) and is more fully described in Ref 2 *Refs:* 1)W.E.Segl, USP 2673690 (1951) & Off Gaz **680**, March 30, 1954, p 1279-L 2)Anon, ChemEnrg **61**, May 1954, pp 116 & 118

Continuous Digester for Nitrocellulose, patented by Milliken of Hercules Powder Co. has been used for manuf of NC of low viscosity, such as required for lacquers

In this method, a suspension of purified NC & water is pumped into a steel pipe 4" (10.2 cm) in diam and 4000ft (1220 m) long, which is coiled into several elongated rectangular sections, arranged one above the other. The section near the entrance end of the pipe is heated by a steam jacket, insulated on the outside, and that near the exit end is cooled by a water jacket To prevent flashing of NC-water slurry in the coils, it is subjected to a back pressure by connecting to the exit end a standpipe 200ft (61m) high, supported by a steel tower. At the top of standpipe, the NC-water slurry overflows and then runs down another pipe to receiving tubs below. The rate of flow of the slurry and its temp are controlled and recorded so that any desired degree of viscosity reduction may be obtd. It requires ca 25mins to obtain reduction to ½sec-viscosity. Motor-driven centrifugal pumps connected in series furnish the pressure
Refs: 1)M.G. Milliken, IEC **22** 326-28 (1930) & CA **24**, 2597 (1930) 2)M.G.Milliken, USP 1911201 (1930) & CA **27**, 4078 (1933)

Continuous Methods for Manufacturing Explosives. The main feature of a *continuous* process is that the final expl product does not remain in the app for a prolonged period of time and the quantity accumulated at any period of time is much smaller than that in the *batch* process. The large quantity of expl material accumulated in a batch process, especially at the end of exothermic nitration, presents a serious safety hazard which is more or less absent in current continuous processes

The continuous method of nitration is particularly useful when the rate of nitration is rapid (such as in case of manufg NG, NGc, or PETN) and an overheating and expln might take place with the batch process. Nitration of cellulose, on the other hand, does not proceed very rapidly (unless mixed acid contains phosphoric or glacial acetic acid instead of sulfuric) and the batch process is usually satisfactory

Success of a continuous operation will depend on the possibility of easy phase separation (such as of NG from spent acid), but when such separation is difficult and requiring a long settling out period (such as RDX in presence of formaldehyde & nitric acid), the continuous method become difficult and dangerous

The smaller quantities of materials undergoing reactions in a continuous operation simplify the cooling and mixing problems, permitting nitrations to be carried out at higher temps than in batch processes. Thus higher outputs per unit time and unit volume of equipment are possible (Ref 21)

Continuous methods for manufg expls usually consist of the following operations: nitration, separation of nitrated product from spent acid, and purification of nitrated product. In some cases only nitration is continuous, while separation and purification are batch processes

Continuous nitration methods may be divided into the following groups: a)Nitration of a liquid material (such as glycerin, glycol, etc) with a nitrating acid in the liq state b)Nitration of a liq material with an acid in the vapor phase c)Nitration of a solid material (such as cellulose, starch, PE, etc) with an acid in the liq state d)Nitration of a solid material with an acid in the vapor state e)Nitration of a solid material with an acid in the vapor state, followed by nitration with an acid in the liq state f)Nitration in the vapor phase, both the substance to be nitrated and the nitrating acid
Historical. Although it is generally believed that continuous nitration is a more recent invention than the batch process, the first continuous nitrators appeared at about the same time (ca 1866) as the batch nitrators (Ref 1)

A comprehensive historical review on continuous methods of nitration is given in Refs 20, 38, 42, & 47. See also Refs 2, 3, 4, 5, 6, 7, 7a, & 31

Most continuous methods invented to 1927 were too complicated to operate and gave no better yields than ordinary batch processes. Their only advantage was increased safety, since only a very small quantity of finished product remained in the app at any one time

The first real successful continuous process of nitration was that developed in 1927 by *Schmid* of Austria (Refs 8 & 9). The app was designed for manufg NG, but it was also suitable for manufg other liquid expls, such as NGc, DEGDN, etc (See also Refs 10, 11, 13, 14 & 29). The first installation was at Gamsen-Brig, Switzerland. Schmid's

app was considerably improved by Ger engineer Meissner and the modified form became known as Schmid-Meissner's apparatus. Installations of Schmid & Schmid-Meissner's equipment contain not only a continuous nitrator, but also a continuous separator and several washing columns. After Schmid's accidental death in 1930, Meissner extented the application of continuous method to nitration of substances like benzene, toluene, etc. The "Firma Meissner", Köln, Bayenthal has been manufg Schmid-Meissner's equipment (Refs 14, 16, 22, 37, 39, 43, 44 & 48). One of the S-M units was installed in 1954 for the Argentine Navy's plant at Azul (south of Buenos Aires) (Ref 34a)

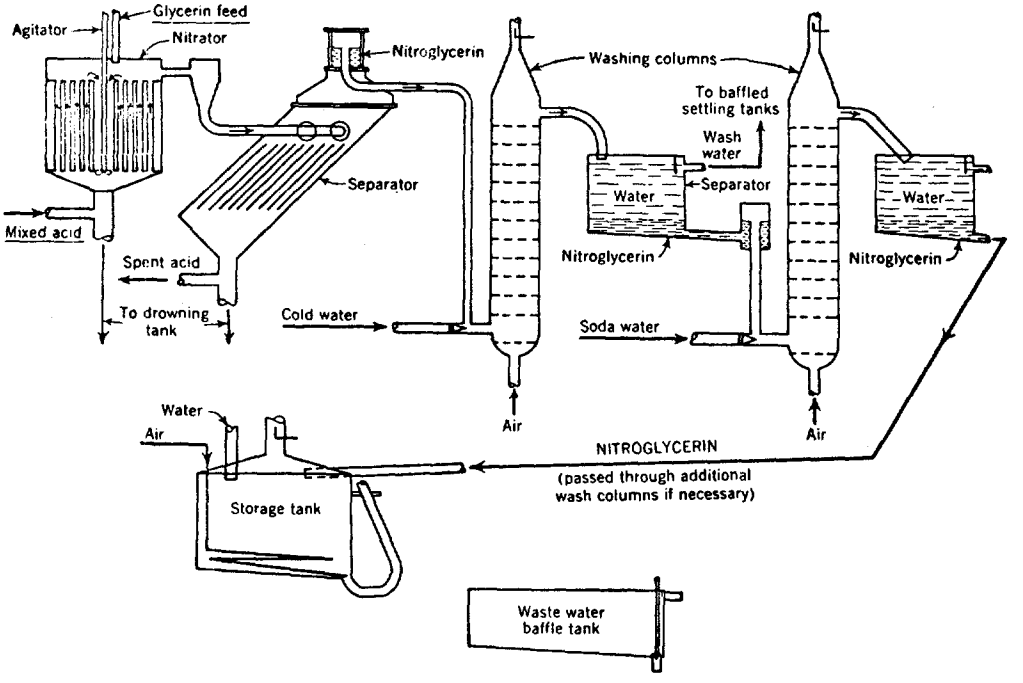
The next successful continuous process for manufg NG, was that developed in 1935 by *Biazzi* of Vevey, Switzerland (Refs 15 & 26) and the first Biazzi plant was built in 1936 in Italy. Since then many plants were built thruout the world (Refs 23, 27, 29, 30, 32, 42, 47, 48 & 49), including Canada & USA (Ref 31)

One of the latest successful continuous NG processes was that patented by *Nitroglycerin AB* in 1955 and installed at Gyttorp, Sweden (Ref 34). A similar type of process was developed a few years later by *Hercules Powder Co*, Wilmington, Delaware (cited in Explosivst **1963**, p 22-R)

Continuous methods have also been developed for manuf of expls other than NG. As examples may be cited nitration plants for: benzene (Refs 5 & 6a); aromatic hydrocarbons other than benz (Refs 7, 13 & 16); PE (Refs 17, 19, 21, 21b & 22); DEG (Refs 21, 22 & 28); various org compds (Ref 36); cellulose, starch, sugar, etc (Ref 21); methyl nitrate (Ref 23a), etc. A continuous method for manufg propellants is described in Ref 46. A continuous method for manuf of TNT by the method of Bofors was installed in 1955 at the Fábrica Naval de Explosivos, Azul, Argentina (Ref 34a)

Description of Some Continuous Methods for Manufacturing Explosives:

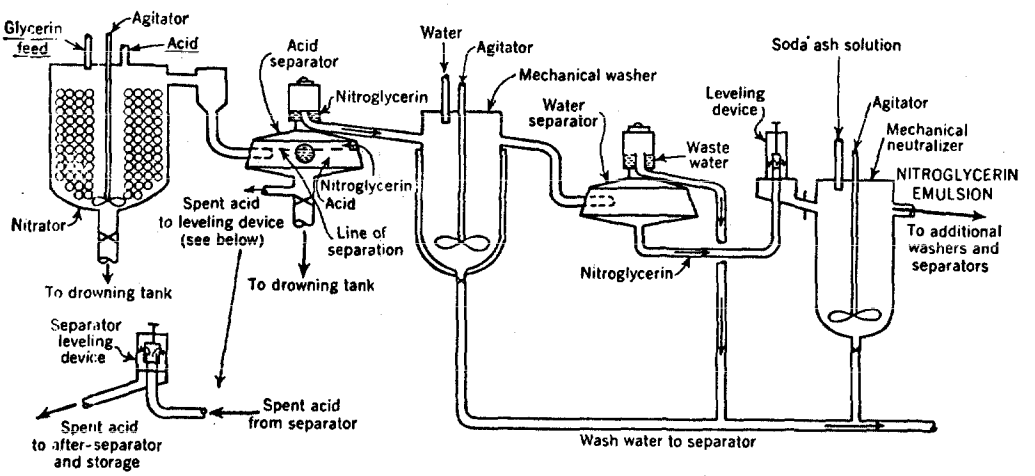
a) *Schmid-Meissner's Process.* It involves continuous nitration of glycerine (or other liquids, such as ethyleneglycol or diethyleneglycol), separation of the nitrated product from spent acid, and purification by neutralization and washing. The stainless steel nitrator of the process is equipped with a motor driven steel propeller, vertical cooling coils and an overflow tube to separator. A two-way valve, which connects to the mixed nitric-sulfuric acid line, or may be opened to the drowning tank, is fitted centrally to the bottom of the nitrator. Brine at ca -5° is circulated at controlled rate around the cooling tubes in the annular space outside the nitrator.



SCHMID-MEISSNER CONTINUOUS METHOD FOR MANUFACTURING NG

As a measured amt, per unit time, of mixed acid is fed in from the bottom, a calcd amt of glycerin is fed in from the top at a level below that of the overflow pipe. The flow of materials into the nitrator is by means of automatically controlled air pressure. The propeller mixes the two liquids and glycerin is nitratec by acid to NG. The temp in the nitrator is not allowed to exceed 18°. The emulsion of NG & spent acid flows off continuously thru the overflow to the separator, which is a stainless steel,inclined,rectangular tank provided with a number of vanes, equipped with a bottom draw-off valve for spent acid, a sight glass near the top and a steel neck with overflow pipe for the NG. Owing to the difference in density, the spent acid settles to the bottom of the separator and the nitrated product rises. The withdrawal of acid from the bottom is so regulated as to keep the separated nitrated product in the top of the separator at a predetermined level. The spent acid is discharged to the acid recovery plant and the NG flows by gravity to the base of the first of the washing columns. Each column consists of superimposed cylindrical glass sections (rings) separated by perforated stainless steel plates and rubber ring gaskets. As the nitrated product from the separator enters the bottom of the first washing column, it is mixed with cold water and is emulsified by means of air injected into the liquid. This also forces the material to rise to the top of the column, where it overflows into a tank which serves as an intermediate separator. Here the nitrated product rapidly settles to the bottom, from where it is conducted to the base of the second wash column, where it is mixed with hot dil aq soln of soda & ammonia and is emulsified with air. The emulsion flows from the top of the column to another intermediate separator, where the nitrated product settles to the bottom and is separated from the soda ash water, which is conducted to waste via settling tanks. Any NG held in the tanks is drawn off periodically and rewashed. The nitrated product is subsequently passed thru addnl wash columns and separators until the desired stability is attained. Finally the purified product is passed thru a flannel filter bag before beigh caught in a lead tank for storage and use (Refs 14, 22, 29, 37, 39, 43, 44, 45, 48 & 49)

b) *Biazzi's Process.* Its operation is similar to that of Schmid-Meissner's, but it is considered to be safer because of the smaller amt of NG in the system at a given time. The nitrator is a small cylindrical stainless-steel vessel provided with a bank of helical cooling coils. A high-speed shrouded impeller-type agitator forces the nitrating acid up thru the bank of coils and down thru the cylindrical space in the center of the coils. The fresh mixed acid and the material to be nitrated stored in large scale tanks or bins enter, in controlled ratio, at the top of the nitrator, striking the liquid at such a point in the vortex that the feed is immediately thrust beneath the surface and carried down thru the central space formed by the coils. The mixture then spirals back, up and out, thru the banks of cooling coils a portion being carried off by the overflow pipe which leads to the separator. The flow of mixt past the coils is counter to the flow of cooling brine circulating thru the coils. Because of this arrangement, permitting rapid reaction thru rapid



BIAZZI CONTINUOUS METHOD FOR MANUFACTURING NG

heat absorption, the mixed acid can be stronger than that used in the Schmid-Meissner's process. The cooling coils, agitator, and cover of the nitrator are mounted as a unit separate from the cylindrical body, which can be lowered hydraulically for inspection

The Biazzi acid separator is a stainless-steel vessel of the shape shown in the Fig. The emulsified mixt of nitrated product and spent acid enters tangentially thru the side at about midway its height and imparts a slight centrifugal action to the upper layer of liquid in the separator. This action helps to break the emulsion and to prevent local overheating. The spent acid flows continuously from the bottom of separator thru a draw -off, controlled by an adjustable collar, which can be raised or lowered by means of a micro screw attachment. After leaving the first separator, the acid is passed thru an after-separator, from which some of the nitrated product is recovered. As the spent acid leaves the after-separator, it is usually diluted with water to dissolve traces of dissolved nitrated product

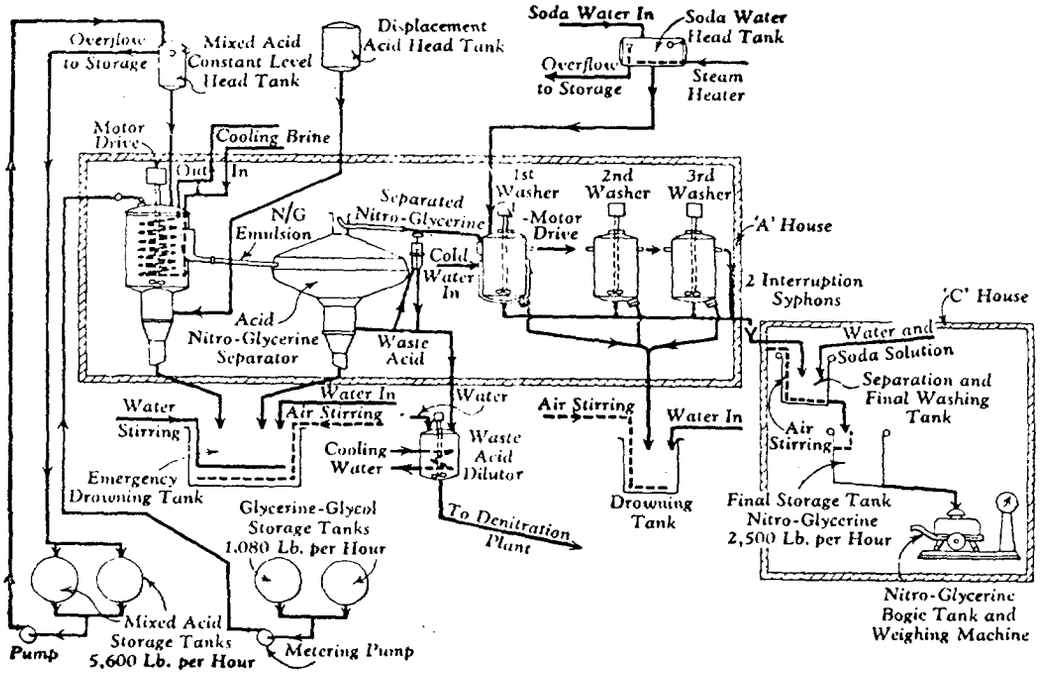
The separated nitrated product is continuously drawn off from the first separator into a stainless -steel wash tank equipped with a cylindrical baffle and an impeller-type agitator. Water is continuously added to the washer and the mixt overflows into a second separator. The nitrated product is drawn from the bottom of the separator and is conducted to the second washer, where some soda wash soln is added with agitation. If a high purity nitrated product is required, such as for NG used in proplnts, the emulsion from the second washer, together with some soda ash soln is conducted thru a battery of 3 or 4 washers, in which wash waters are running countercurrent to the movement of NG. From there the emulsion passes thru a series of separators arranged in cascade and is collected in a storage tank

In cases of overheating, the nitrator and the first separator can be emptied quickly into a drowning tank, which is generally filled with water, but can be filled with sulfuric acid (Refs 15, 27, 29, 31, 32, 39, 45, 47 & 49)

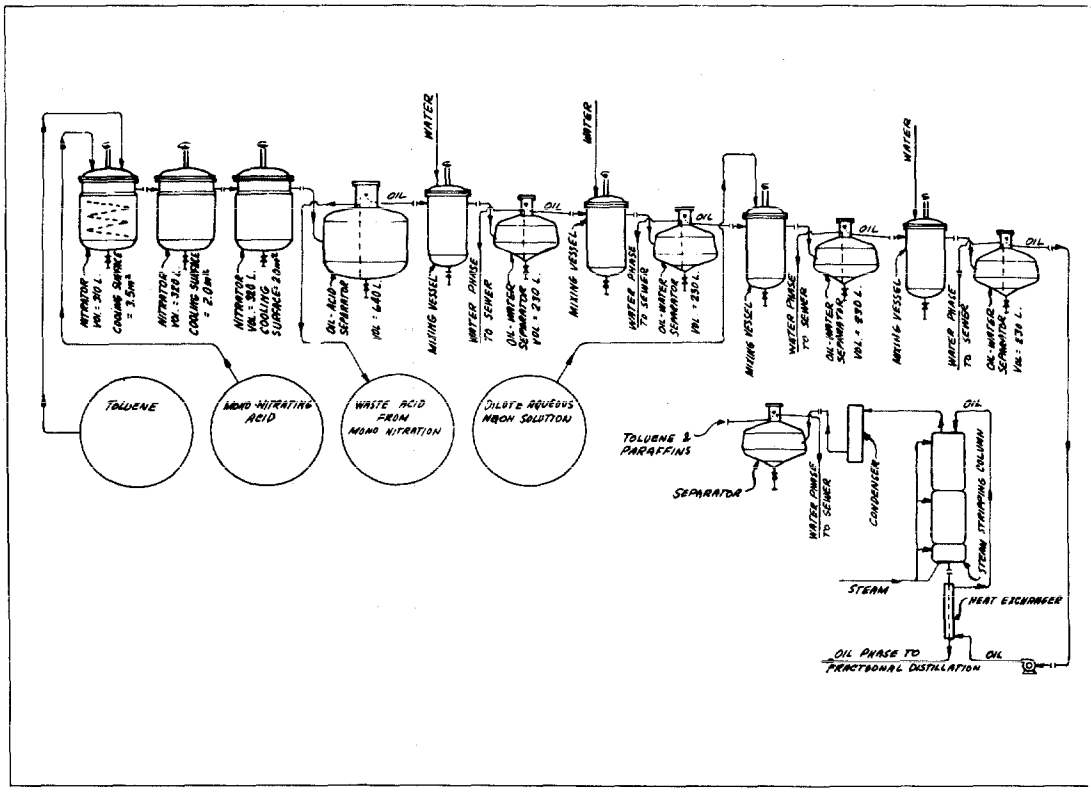
A slightly different arrangement of Biazzi's process is given by Berthmann (Ref 44, p 33)

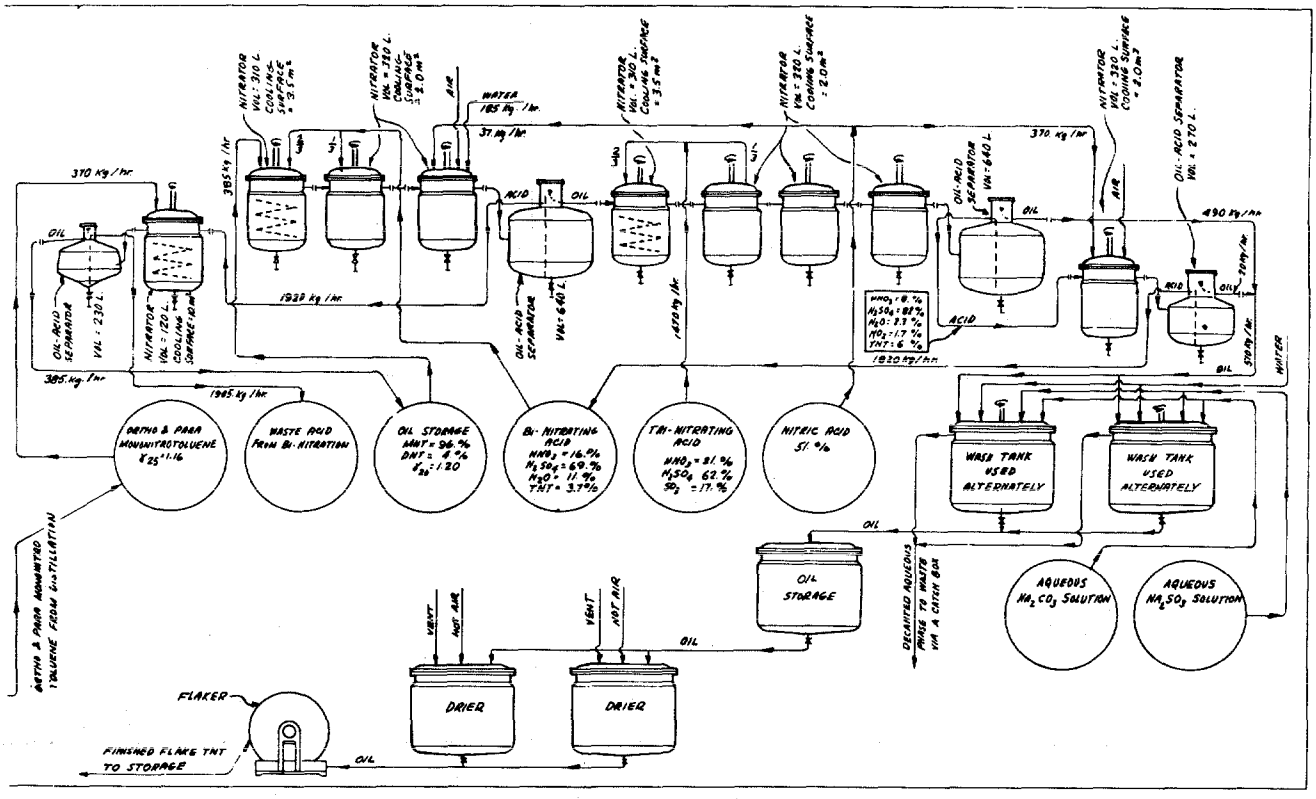
A flow sheet of Biazzi's process installed at the Canadian Industries Ltd Calgary plant is given by Pennie (Ref 48). It is reproduced here

The Biazzi's process can be used not only for the manuf of NG, NGc & DEGDN but also for manuf of aromatic and aliphatic nitrocompds. For mononitration, only one nitrator is required for each installation. This includes MNB, MNT, Methyl nitrate, etc. For higher degrees of nitration of



BIAZZI CONTINUOUS METHOD FOR MANUFACTURING NG
CANADIAN INSTALLATION





BIAZZI CONTINUOUS METHOD FOR MANUFACTURING TNT
BELGIAN INSTALLATION

aromatic compds, two or more nitrators are used in series. Such nitrations usually employ the spent acid from higher nitration (after fortification) as the mixed acid for the next lower nitration (Ref 29)

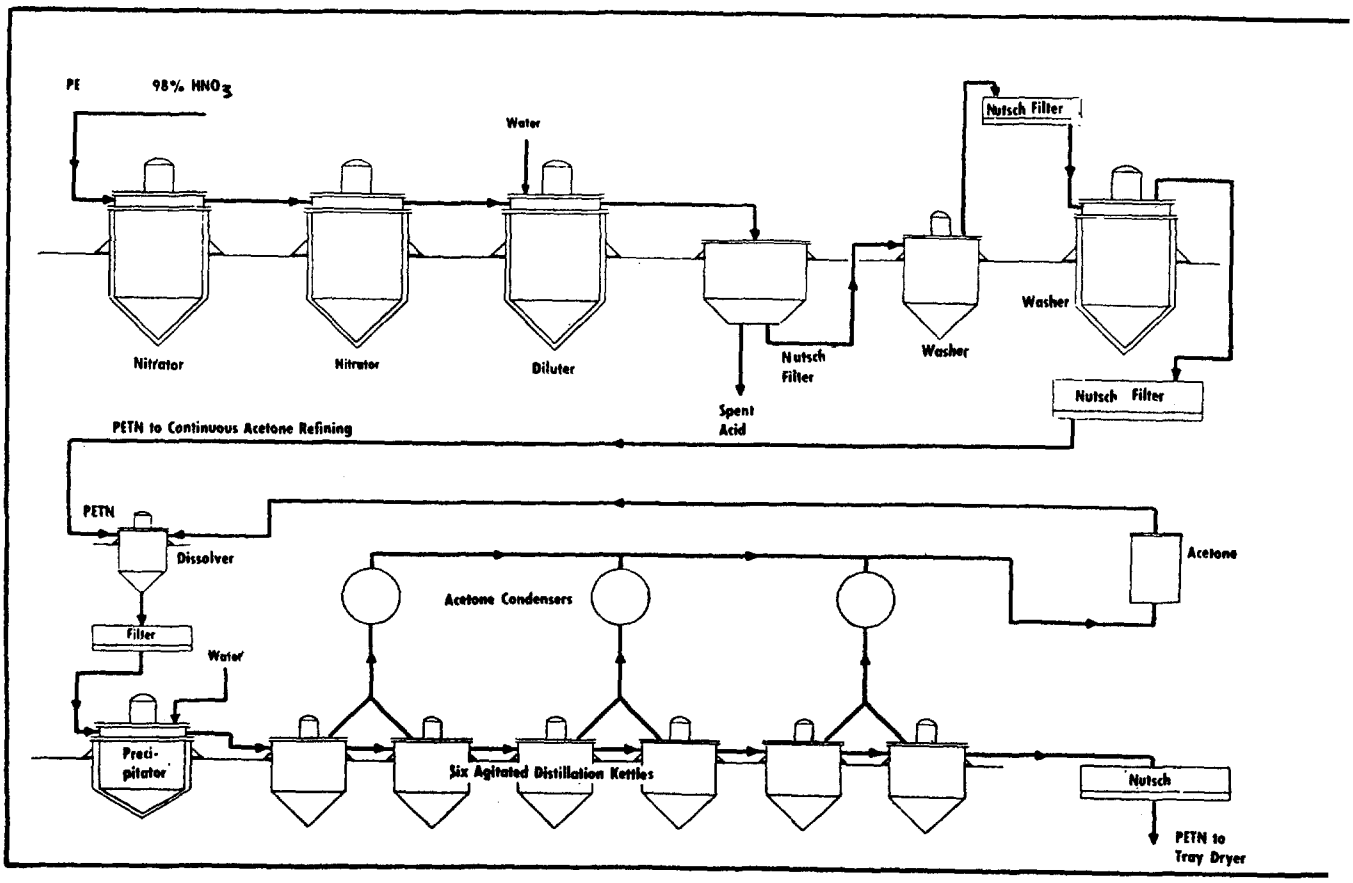
c)*Biazzi's Procdss for Manufacture of TNT* is represented in the Fig on p C505

Continuous Process for Manufacturing PETN. The following process, using stainless steel equipment, was developed during WWII at the Troisdorf Plant of Dynamit AG:

PE of 97-8% purity was fed continuously to the first nitrator from a hopper by means of a screw conveyer and, simultaneously, nitric acid, 98% strong, was introduced in quantity of 5-6 parts per part of PE, from a constant head tank thru a rotameter. The first nitrator was a cylindrical vessel provided with a stirrer, a jacket and coils carrying AN brine at -5° to - 11°. The temp in the nitrator was +15°

The reaction slurry overflowed to the second nitrator where the temp was lowered to +10°. The concn of acid dropped to 80%. The slurry then overflowed to the dilutor where water was added to reduce the concn of acid to 30%, while the temp was maintained at 15-20° by means of brine cooling and agitation. The dilution pptd the crysts of crude PETN and the slurry then overflowed to one of the two cylindrical nutsch filters thru a swing pipe that could be directed to either nutsch. Each of them was provided with a stainless steel filter cloth with a hole in the center which could be closed with a conical plug. With the hole closed, the slurry was run in, with suction, from the dilutor until a sufficient amt of crude PETN had accumulated. Then the swing pipe was diverted to the second nutsch. After drawing off as much of spent acid as possible, the plug was removed and the crysts were transferred thru the pipe, by a stream of water to the continuous washing system consisting of an agitated tank for washing with water, a nutsch to remove the acid water, a tank for neutralizing with soda ash soln and a final nutsch

The crude washed PETN was then transferred



GERMAN CONTINUOUS METHOD FOR MANUFACTURING PETN

to the continuous refining plant, which consisted of a dissolver (to dissolve PETN in acetone), a filter and a precipitator (where PETN was pptd by adding water to the acetonic soln). Then the slurry was conducted thru several distillation kettles to remove the bulk of acetone and the concd hot slurry cooled and filtered thru one of the nutsches. Montan wax, used a phlegmatizer, could be added to the last still (Ref 22)

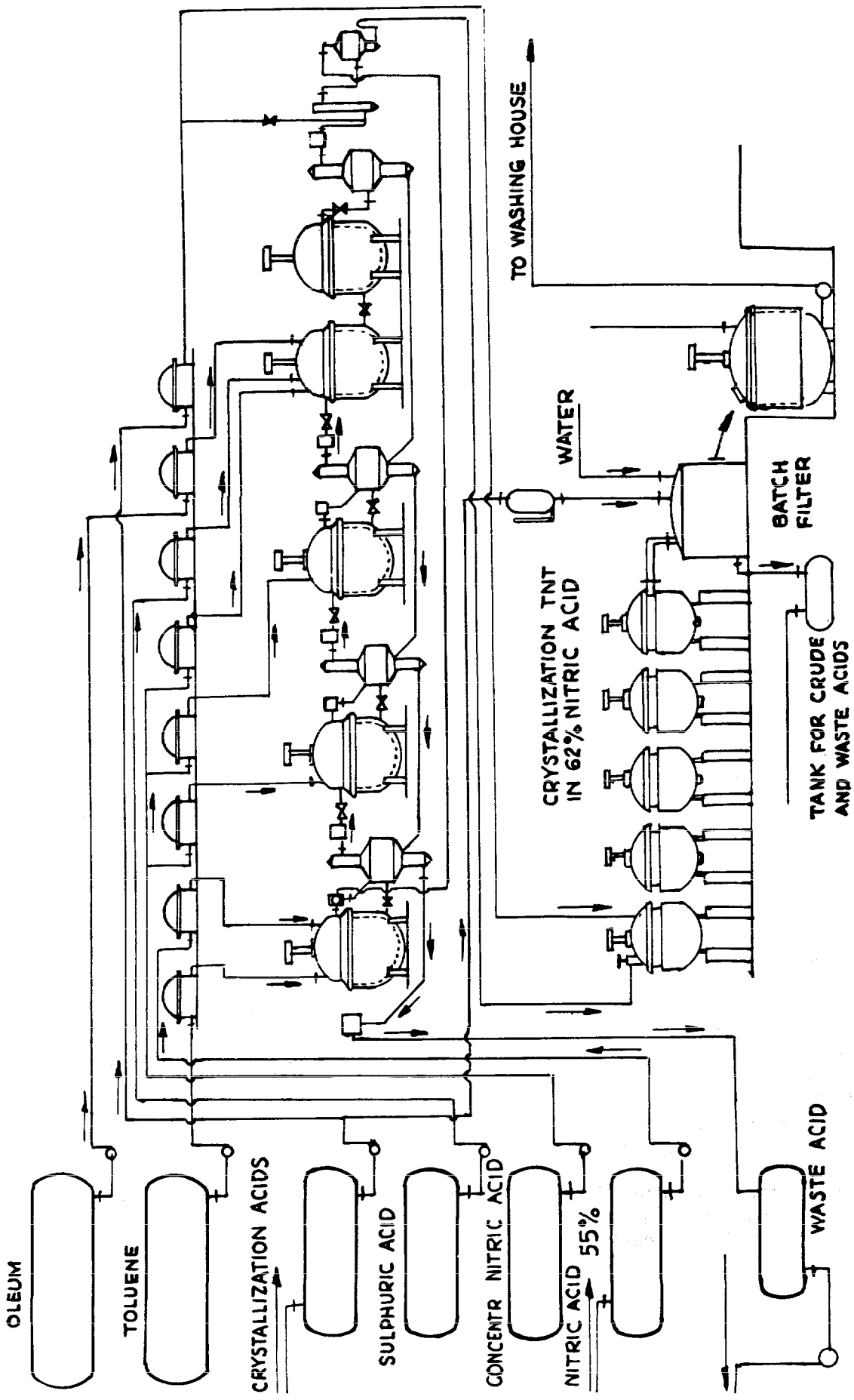
Continuous Process of Bofors Company for Manufacturing TNT is represented in Fig, p C508

Similar Bofors equipment was installed in 1954 -55 at the Naval Explosive s Plant at Azul, Argentina (Ref 34a)

Continuous Methods for Manufacturing Nitrocellulose. Several methods are known, of which the Dietzsch process developed during WWII in Germany is briefly described here under Combustible and Consumable Cartridge Cases. This method combines vapor-phase and liquid-phase nitration processes. The Japanese used during WWII a continuous liquid-phase method of nitration of paper pulp, which was in the form of a sheet

A continuous vapor-phase nitration of cellulosic material in the form of a sheet was developed in the USA by the Brown Paper Co, Berlin, New Hampshire. No description of this method is available

Refs: 1)J.Downie, BritP 1813 (1864); A.Nobel, USP 5717 (1866) (Accdg to Explosivst **1962**, 215 & 246-R these patents described two original methods of prepg NG, one of them, known as the "warm" method was a continuous process as far as nitration was concerned, while the other, known as the "cold" method, was a batch process. In the "warm" method, the cooled nitric-sulfuric acid was allowed to flow together with the glycerin into a lead funnel with perforations at its lower half. As the funnel was not cooled the temp of nitration was ca 45°, but was never allowed to rise above 65°. In the "cold" method, the glycerin was added in small amts, with hand stirring, to stoneware jugs, contg mixed acid and cooled in an ice-water bath. The temp of nitration was near 0° In both methods the NG was drained off, washed with warm w and then with a warm soln of Na carbonate until acid-free. It is interesting to note that the "warm" method, although abandoned in favor of the more economical and safer "cold" method, contained certain features that are to be found in the most advanced continuous processes in operation today) 2)A.E. Rudberg Leslie's Illustrated Newspaper, Jan 15, 1876 & O. Guttmann, "Manufacture of Explosives", Whittaker, London, Vol **2** (1895), p 95, cited in Explosivst **1962**, 246-R (One of the earliest processes for continuous nitration of glycerin) 3)Sir Hiram Maxim, BritP 5809 (1891) cited in Explosivst **1962**, 246-47 (Continuous nitration of glycerin by mixing it with nitric-sulfuric acid in a tubular vessel by means of an air-operated injector) 4)R.Evers, GerP 183183 (1907), cited in Explosivst **1962**, 261 (The title of this patent: "continuous production of nitroglycerin", is misleading, because it only deals with a spraying nozzle used for feeding glycerin -acid mixture into a batch nitrator provided with cooling coils) 5)Anon, SS **10**, 46-7 (1915) (App of Dr Kubierschky for continuous nitration of benzene) 6)Marshall **1** (1917), 220 (NG plant at Waltham Abbey, which seems to be semicontinuous) 6a)Colver (1918), 125-26 (App of Neumann designed in 1914 for continuous nitration of benzene); 126-28 (App of Dr ter Meer for continuous nitration of benzene); and 128-31 (App of Kubierschky for continuous nitration of benzene) 7)J.G.Baxter, BritP 125091 (1919) & Explosivst **1962**, 262-L, (A semicontinuous NG plant) 7a)W.Macnab, JSCI **41**, 354T-355T (1922) (Continuous processes of manufg expls developed during WWI include: manuf of PA by nitration of sulfonated phenol, developed by Brookes Chemicals Ltd, at Lightcliffe, England and the TNT plant devised at Oldbury, England by Holley & Mott) 8)A.Schmid, SS **22**, 169-73 & 201-06 (1927) (Description of the first really successful continuous NG plant for which Ger & US patents were issued) 9)A.Schmid, BritP 284700 (1928) (A two-chamber nitrator for use with Schmid's continuous process) 10)R.Neubner, SS **23**, 44-6 (1928) (Discussion on advantages of Schmid's process) 11)G. von Feilitzen SS **23**, 343-44 (1928) (Description of Schmid-Meissner's plant installed at Brig-Gamsen, Switzerland) 12)R. Evers, GerP 513396 (1930), cited in Explosivst **1962**, 262-L (Continuous nitration of glycerin, which relies on internal mixing of the streams of acid & glycerin which are impacted on a spherical surface) 13)Marshall **3** (1932), 43-4 (Continuous nitrator & separator of A.Schmid for manuf of NG); 83 (Continuous process for nitrating aromatic compds at Brookes Chemicals Ltd, England) 14)Stettbacher (1933), 158-62 (Continuous processes of A. Schmid & J. Meissner; schematic view of Schmid -Meissner installation) 15)M.Biazzi, ItalP 322990 (1935) (Continuous process for manuf of NG) (The US patent was filed in 1939 but was not granted until 1948. It is listed here as Ref 26) 16)Thorpe



BOFORS AB CONTINUOUS METHOD FOR MANUFACTURING TNT

4 (1940), 468-69 (Continuous process of MNT DNT & TNT manuf introduced during WWI at Oldbury, England. It was modified and improved in 1932 at Waltham Abbey), 496 (Continuous method of Schmid for manuf of NG) 17)W. deC. Crater, USP 2363834 (1944); CA **39**, 3471 (1945) & IEC **40**, 1628 (1948) (A continuous nitrator for manuf of PETN) 18) D.F.Othmer & H.L.Kleinhans Jr, IEC **36**, 447-51 (1944) (Continuous partial pressure nitrator using nitric acid alone) 19)A.A. Swanson, PB Rept **320** (1945) & O.W.Strickland et al, PB Rept **925** (1945) (Continuous methods for manuf of PETN, which were used in Germany during WWII) 20)N.Shreve, ChemMetEngrg **52**, 103-05 (May 1945) (History of continuous process development) 21)E.Berl, USP 2384415 (1945) & CA **40**, 206 (1946) (Continuous production and stabilization of NC, NS, DEGDN, PETN & Nitrosugar) 21a)E.Berl, ChemMetEngrg **52**, 202, 204 & 206 (May 1945) (The problem of batch versus continuous operation in the explosives industry is primarily concerned with improvements in safety of operations) 21b)A.A.Swanson et al, PB Rept **320** (1945) (Manuf of PETN in Germany) 21c) O.W.Stickland et al, PB Rept **925** (1945) (Continuous methods used in DAG plants) 21d)C.H.Brooks et al, PB Rept **39480** (1945) (Schmid-Meissner installation in Germany) 22)R.A.Cooley, ChemInd **59**, 645-49 & 759 (1946) (Continuous Schmid-Meissner process for NG & DEGN manufg, as used in Germany during WWII; Continuous method for manufg PETN) 23)R.Ashcroft et al, BIOS Final Report **833** or PB Rept **63877** (1946), Item 2, p A2/4 (Continuous Biazzi process installed in 1938-39 at Schlebusch, Germany and modified in 1940) 23a)H. & B. Walter, FIAT Final Rept **1035** or PB Rept **78271** (1947) (Continuous method for manufg Methyl Nitrate) 24)W. DeC. Crater, IEC **40**, 1628 (1948) (Bibliography on WWII German continuous methods of manuf expls) 25)Stettbacher (1948), 60 (Lists some continuous installations) 26)M.Biazzi, USP 2438244 (1948) & CA **42** 4008 (1948) (See Ref 15) 27)J.C.Smith, ChemInd **62**, 929-31 (1948) (Biazzi method) 28)A.Bresser, IndChemist **25**, No 289, 92-8 (1949) & CA **43**, 4013 (1949) (Recent developments in the use of continuous flow of materials and countercurrent washing of NG & DEGDN) 28a)W. deC. Crater, IEC **41**, 1889-91 (1949) & **42**, 1716 & 1718 (1950) (Review of literature on nitration, including continuous methods) 29)Kirk & Othmer **6** (1951), 29-30 & **9** (1952, 324-27(Continuous methods of Schmid, Schmid-Meissner& Biazzi) 30)Stettbacher, Pólvoras (1952), 81-3 (Continuous method of Biazzi) 31)W. deC. Crater, IEC **44**, 2039 (1952) (The 1st commercial continuous unit for manuf of NG in North America was put in opn in 1952 by the Canadian Industries Ltd, near Calgary, Alberta. Slightly later the 1st US continuous plant was installed by the DuPont Co near Martinsburg, W.Virginia. Both plants are equipped with Biazzi units. The same type of equipment was used by the US Navy at Indian Head, Maryland and Radford, Virginia) [See also Anon, ChemEngrg **60**, No 3, 130-34 (1953)] 32)H.J.Klassen H J.M. Humphreys, ChemEngrg Progr **49**, 641-6 (1953) (Biazzi's continuous plant) 33)F.Tabouis & J. Vaganay, MP **37**, 413-16 (1955) & Picatinny Arsenal Translation No 20, byG.R.Loehr (A laboratory app for continuous prepn of expls, such as RDX, Dinitrophenetol or Trinitrophentol) 34)Nitroglycerin AB, Guttorp, Sweden, BritP 735888 (1955) & CA **50**, 7855 (1956) (Continuous methods of manufg NG and other org nitrocompds) 34a)B.T.Fedoroff, Info obtd during visit to Azul Plant, Argentina, (1955) 35)Anon, ChemEngrg **63**, No 1, 199-200 (1956) (A continuous method of manufg nitroparaffins developed by Commercial Solvents Corp) 36)N.A.P.Nilsson, USP 2737522 (1956) & CA **50**, 6796 (1956) (An improved continuous method for production of liquid org nitrocompounds, such as NG, NGc, DEGDN, MNB, MNT's & Ethylnitrate) 37)DRIM(?), "30 Years of Continuous Nitroglycerin Process Schmid-Meissner," Explosivst **1957**, 65-7 (Description of process in Ger & Engl, including cuts & photographs) 38)Anon, IEC **50**, 42A-43A (1958) (Continuous methods for manufg expls) 39)Groggins (1958), 99-101, 111-13 & 746-48 (Continuous methods for manufg expls) 40)J Stuart II, USP 2840303 (1958) & OffGaz **731**, No 4, p 491 (Centrifugal separator used in conjunction with Hercules continuous manuf of NG) 41)F.S.Stow & C.D.McKinney, Australian P 222341 (1959), cited in Explosivst **1963**, p 24, Ref 65 (Description of Hercules Co continuous process for manufg NG) 42)A.Stettbacher, Explosivst **1959**, 67-71, 187-93 & 206-10 (His-

torical review and description of various continuous methods of manufg expls) 43)Girdler Construction Division Chemetron Corp, "J Meissner Continuous Process for the Chemical and Explosives Industries," Louisville 1, Kentucky, May 1, 1959 (*A pamphlet, 9pp*) (The Girdler firm is the sole N.American agent for chemical processes owned by the firm of Josef Meissner, Köln, Germany. It can construct plants for continuous nitration of glycerin & aromatic hydrocompounds and for manuf of hexamethylenetetramine, a starting material for the manuf of RDX) 44)A.Berthmann, "Explosivstoffe", C.Hanser Verlag, München (1960), 30-3 (Continuous methods of manufg expls) (Part of Winnaker-Küchler, "Chemische Technologie", Vol **4**) 45)Ullmann **12** (1960) 774-75, 778 & 796-97 (Continuous methods of manufg expls) 46)H.Freyer, DBP 1082175, Kl 78c, Gr 20, abstracted in Explosivst **1962**, 82 (Continuous manuf of proplnts) 47)M.Biazzi, "25 Années Biazzi", Vevey, Switzerland (1962) (An illustrated pamphlet on Biazzi's method) 48)A.M.Pennie, "Nitroglycerine – A Century of Manufacture", Explosivst **1962**, 242-43, 261-66 & **1963**, 21-4 (A review of continuous processes of manufg NG) 49)C.Giorgio, "Tecnica degli Esplosivi", DelBianco, Udine (1964); 104 (Continuous manuf of NG by Schmid-Meissner's method); 105 (Continuous manuf of NG by Biazzi's method); 106-08 (Continuous manuf of NG by Nitroglycerin AB, Gyttorp, Sweden) 50) W.H.Rinkenbach, "Explosives," in Kirk & Othmer, Vol **8** (1966), p 595 (Improved Biazzi Process)

Continuous Mixing of Castable Propellants at Aerojet-General Corp. Discussed in conf Progress Rept by A.S.Cohen, Contract NOrd 18171. It was presented at the Integration Committee on Propellants and Explosives. Operations Sub-committee. 3rd Meeting held at NOTS, 15-16 March, 1960

Control System. A coordinated group of components designed to exert a directing influence on other components. The system for properly maneuvring a missile in response to guidance intelligence furnished by the guidance system (See also Automation; Automatic Control; Automatic Process Control in Vol 1, p A512-L of Encycl)
Refs: 1)DictGuidedMissiles (1959) 2)OrdTech Term (1962), 84

Conversion of War-Time Propellants to Peace-Time Products. The subj of an article by Dr Kolsen published in Chem Ztg **79**, 68 (1955) and abstracted in Explosivstoffe **1956**, 43

Convolute Wrapping. This is a method of inhibiting the burning of rocket proplnt grains by continuous multilayer wrapping of the grains with thin sheets of inhibitor plastic. Adhesion between layers is generally accomplished by moistening with a volatile solvent. The wrapping may of spiral form or other geometric patterns
Ref: R.P.Baumann; private communication (Sept 1964)

Cook-off. The premature deflagration or detonation of ammunition caused by the absorption of heat from its environment. Usually it consists of the accidental and spontaneous discharge of, or expln in, a weapon, caused by an overheated chamber or barrel igniting a fuze, proplnt charge or bursting charge
Ref: OrdTechTerm (1962), 84

Cook-off Temperature and Its Determination are briefly described under AMMONIUM NITRATE BLASTING EXPLOSIVES in Vol 1, p A354, Note a. The test was developed by Spencer Chemical Co, Kansas City and is described in the pamphlet entitled "Ammonium Nitrate Explosives for Underwater Applications" June 18, 1960. Some values obtd by this test are given in Table on p A354, Vol 1 of this Encyclopedia

Cool Burning Propellants. See Cool (or Cooled) Propellants

Cooled Explosives. See Cool (or Cooled) Explosives

Cooled Propellants. See Cool (Cooled) Propellants

Cooled SC Cordite. See under CORDITES

Cool (or Cooled) Explosives. This name is given to expls, such as Ammonium Nitrate, Guanidine Nitrate & Nitroguanidine, which explode or burn with the production of gases

appreciably cooler than gases from the expln or combustion of other expls, such as TNT, RDX, PETN & Propellants. The incorporation of Nitroguanidine into Propellants produces cool & flashless compns. There are also cool Primary Explosives, such as Nitrosoguanidine, which detonate without producing a flame

Ref: Davis (1943), pp 324, 387 & 391

Cooling Agents or Coolers. These substances are added to expls or proplnts to reduce the temp of expln or combustion. The following types of substances can be used as cooling agents:

a) Inert Substances, such as Kieselguhr, agrillaceous earth, powdered chalk or marbre, etc. Their cooling action is due to absorbtion of part of the heat developed on expln or combustion

b) Water or Hydrated Salts. Small amts of water or of salts contg large amts of water of hydration [such as $CuSO_2.5H_2O$; $MgSO_4.7H_2O$; $Na_2SO_4.10H_2O$; $Na_2B_4O_7.10H_2O$; $K_2Al_2(SO_4)_4.24H_2O$; $(NH_4)_2Al_2(SO_4)_4.24H_2O$; etc], when incorporated in expl mixts, absorb heat thru evapn of water, thus lowering the temp of gases produced on expln (Refs 1,2,3&4)

Hydrated sodium carbonate, $Na_2CO_3.10H_2O$, is not suitable for use because it attacks both AN and NG, usual ingredients of dynamites (Ref 3, p 353)

c) Volatile Salts. Small amts of salts such as NaCl, KCl, NH_4Cl, $CaCl_2$, $(NH_4)_2SO_4$, K_2SO_4, $NaHCO_3$, $Na_2C_2O_4$, $K_2C_2O_4$, $(NH_4)_2C_2O_4$, K hydrogen tartrate, Al stearate, etc, absorb thru volatilization or molecular dissociation, part of the heat produced on expln or combustion, thus cooling the gases

d) Carbonaceous Materials, such as charcoal, coal, coke, cellulose, sawdust, flour, triacetin, etc, being non-explosive, retard the rate of combustion, thus not allowing the temp of gases to become too high. In addition, their products of combustion change the compn of the expln gases by increasing the total volume and the amt of CO_2 & H_2O

e) Cool (or Mild) Explosives, such as NGu, GuN, AN, mono- and dinitroaromatic compds, explode or deflagrate with the production of gases that are appreciably cooler than those produced on expln of HE's, such as NG, NGc, DEGDN, TNT, PETN, RDX, etc

A lowering of expln or combustion temps is required in the following cases:

a) Explosives to be used in gaseous and/or dusty coal mines. If these expls do not produce a "cool flame", they might ignite firedamp or dust thus causing a coal mine explosion. These "cool flame" expls are known as "permissible". See under COMMERCIAL (or INDUSTRIAL) EXPLOSIVES, Section II. Coal Mining Explosives, Permissible, p C 444-R

b) Smokeless propellants, especially those intended for use in large caliber cannons. If the combustion temp is not sufficiently lowered, a "muzzle flash" is produced. This is undesirable because it might reveal to the enemy the location of the weapon, especially at night. In this case, the cooling agents act as "flash reducers"

c) Pyrotechnic mixtures, especially those used to produce "colored smokes". They should burn at low temps to prevent thermal decompn of the organic dyes which are usually incorporated in such mixts. The use of cooling agents, such as $NaHCO_3$ & $(NH_4)_2C_2O_4$ not only slows down the rate of burning, but also makes it more even and prevents spattering (Ref 4)

Refs: 1)C.G.Storm, US BurMines Bull **96** (1916), 8-9, & 13 2)Naoúm, NG (1928), 382-420 3)Davis (1943), 322-24 & 353 4) W. Kiefer & J. Resko, JChemEduc **22**, 385-86(1945) 5) Taylor & Gay (1958), 30 (Cooling agents) 6) A. Berthmann & G. Kuhn, USP 2829036(1958) & CA **52**, 10581-82(1958) (Cooling agents for permissible expls)

Cool (or Cooled) Propellants. This term is applied to artillery proplnts having an isochoric flame temp ranging approx from 2000 to 2700° K at 1000 psi and a heat of expln with an approx range from 700-800 cal/gm. The formulation of a cool proplnt may be that of a single-base, double-base, or triple-base propellant

Cool proplnts were initially developed by Gen U. Gallwitz in Germany beginning in 1934. These proplnts were intended to be less erosive than NG proplnts and at the same time possess the high ballistic potential reqd for muzzle velocities of the order of 3300 ft/sec. The Ger proplnts contg DEGN or TEGN are called "G Pulver"(Gallwitz Propellant) or "K Pulver" (name used in the Ger Air Forces) (Ref 3). Cool proplnts have now been adopted and further developed in the USA, Canada

and Gt Britain

The principal constituents of a cool proplnt are: NC plus 8-15% of a cooling agent (qv); NC plus an org ester other than NG, such as TEGN or DEGN; or NC plus NG & Nitroguanidine. A typical formulation of the latter type is represented by NC(13.15% N) 20, NG19, NGu 54.7, *centralite* 6 & *cryolite* 0.3%. See also Refs 1,2 & 5

The Recherches Chimiques (Ref4) patented smokeless proplnts prepd in the usual manner and made cool-burning by surface-coating the dried grains with 4-24% of the nitrates of polyalcohols, such as DEGN, TEGN, 2-hydroxymethyl-2-methyl-1,3-propanedial or diethanolamine nitrate

Cool proplnts are used when it is desirable to reduce gun erosion and flash in high cyclic rate weapons

The increasing use of solid-fuel gas generators has caused the demand for a family of proplnts having flame temps below 2000°F. These proplnts are low-energy, low-burning-rate compns used to power various elec, servo & mechanical control systems. Many solid proplnts have been developed in the past few years as gas generators. They can be classified as homogeneous double-base, composite double-base, and composite AN proplnts. The Hercules Powder Co. (Ref 6) has issued a bulletin describing two promising proplnts, designated PHM & PKN (compn not reported), which they have developed as gas generators

(This section was written in collaboration with Mr. E.F. Reese of PicArsn)

Refs: 1)C.S.Davis, "Develop a Cool Burning Powder to Give 2000 ft sec Velocity in the Cal .30 Machine Gun", PATR **758** (1936) 2)J.P.Picard et al, PATR **2193** (1955)(Conf) 3) PATR **2510** (1958), pp Ger 70(G Pulver) & Ger 81 (Gudolpulver) 4) Recherches Chimiques SA, Belg P566271 (1958) & CA **53** 10766 (1959) 5) Conf "Propellant Manual", SPIA/M2 (1962) 6) Hercules Powder Co, Expls & Chem Dept, Tech Data Bulletin **XP-1**, "Preliminary Data on New Cooled Propellants" (1963)

Cooppal & Cie or SA de la Poudrerie Royale de Wetteren, Belgium. One of the oldest propellant & expl manufg plants in Europe. It was founded in 1778

Ref: Daniel (1902), 155

Cooppalite & Nitrocooppalite. Cooppalite is one of Belgium's current "explosifs SGP" (Sécurité-Grisou-Poussière) permitted for use in coal mines. It consists of NaCl 42, AN 21.5, NG/NGc 35 & NC 1.5%. This coml mining expl, as well as the Nitrocooppalites, is manufd by SA de la Pondrerie Royale de Wetteren Cooppal & Cie, Belgium

Nitrocooppalite V. One of the current industrial "explosifs SGP" consisting of AN 60, NG10, woodflour 7 & NaCl 23%. The sheath consists of 140g $NaHCO_3$ per 100g of expl. The expl mixture is similar to the Belg compn called *Flammivore*

Nitrocoopalite VIII One of the current Belgian "explosifs de sécurité renforcée" (Type 4) consisting of $NaNO_3$ 55.50, NG 10.11, NH_4Cl 34.30 & NC 0.09

See Vol 2, p B29 for a list of current Belgian Industrial Explosives

Refs: 1) Private communication from Dr. L, Deffet, Directeur du Centre de Recherches Scientifiques et Techniques pour l'Industrie des Produits Explosifs, Bruxelles, Belgium (10 March 1954) 2)Belgian Journal, Explosifs, Special No **4** (Oct-Dec 1958)

Cooppal Powder. One of the earliest smokeless sporting proplnts. It was origninally invented in 1889 in Belgium and wasmanufdthere and also in England. Daniel (Ref 1) reports an early analysis showing: NC 71.25, $Ba(NO_3)_2$ 23.65, resinous substances 3.45 & moisture 1.65%. Marshall (Ref 2) gives analyses published in 1917 as follows: **No 1 (1892) Fibrous 42-grain Bulk** - NC 73.5, metallic nitrates 21.3, shellac 3.2 & moisture 2.0% and **No 2 (1900) Gelatinized 30-grain Dense** - NC 91.2, metallic nitrates 2.0 nitrohydrocarbons 5.5 & moisture 1.3%

Cooppal Powder replaced in 1900 a Brit proplnt called **CL**(qv)

Refs: 1) Daniel (1902), 155 2) Marshall, Dict (1920), 24

Cooperative Build-Up of Detonation. The cooperative theory of deton by "mass flow" was presented in a report of the Brit Armament Res Dept, 26 Nov 1943. The essential feature of this theory is that it provides a mechanism of critical change-over from local thermal decompn, controlled by thermal activation, to hydrodynamic

activation in a deton wave. It is postulated that deton can occur by cooperative decompn of adjoining crystals, as well as by simultaneous activation of several atoms in the same crystal, as proposed by W.E. Garner & A.S. Gomm (1931) and by H. Muraour (1934). The details of this theory are given in the Ref
Ref: A.R.Ubbelohde, TrRoySoc **241A**, 201-04 (1948)

Coordinate or Dative Bond. See under Bond, Vol 2, p B241-R

Coordination Compounds (Complexes). See Ammine or Ammoniate and Coordination, Vol 1, pA275

Coordination Number. The valence number of the central atom of addition compds. It indicates the number of molecules or atoms held by that atom. Carbon & related elements have a max of 4, all other elements have generally a coordination number of 6. Thus $[PtX_6]R_2$ or $[CrX_6]R_2$, where R is usually a monovalent radical, while X is Br, Cl, CN, SCN, NO, NO_2 NH_3 or H_2O
Ref: Hackh's (1944), 224

Copolymerization. The production of high molecular wt compds by the addn of two or more different monomer units which alternate in the chain molecule. These compds frequently have useful props superior to those of polymers made from a single monomer. The technological importance of copolymers (such as synthetic rubbers, plastics & fibers) has led during the last decade to a large amount of both experimental & theoretical work on the principles of copolymerization
Refs: 1) F.R. Mayo & C.Walling, "Copolymerization", Chem Revs **46** 191-287(1950) 2) T. Alfrey, Jr, J.J. Bohrer & H. Mark, "Copolymerization" Vol **8** of High Polymers, Interscience NY (1952), 279pp 3) C.H. Bamford, W.G. Barb, A.D. Jenkins & P.F. Onyon, "The Kenetics of Vinyl Polymerization by Radical Mechanisms," Academic Press, NY(1958), 319pp 4) N.G. Gaylord & H. Mark, "Linear and Stereoregular Addition Polymers: Polymerization with Controlled Propagation," Interscience, NY (1959), 581pp 5) A.G. Organesyan, "Sintez Novykh Copolimerov" (Synthesis of New Copolymers), Gosudarst Tekh Izdatel, Erevan, Armyan (1960), 170pp

COPPER AND ITS COMPOUNDS

Copper, Cu (Cuivre in Fr; Kupfer in Ger; Rame in Ital; Cobre in Span; Med' in Rus), at wt 63, 54 yel-reddish ductile metal; d 8.933 at 20°; mp 1083°; bp 2582°; insol in w; sol in nitric acid and hot sulfuric acid. It is obtained from ores, mostly sulfide and oxide. Native Cu ore is rather scarce and one of the known large deposits is in the Upper Penninsula of Michigan. Metallurgy of ores varies with the type of ore. For example, for low-grade sulfide ores, the following operations are usually necessary: a) concentration b) roasting c) formation of Cu "matte" (40-50% Cu) d) reduction of matte to "blister" (96-98% Cu) and e) electrolytic refining to ca 99.9% Cu. Detailed description of Cu matallurgy is given in Refs 3,4,5,6,9,12 & 14

Metallic (not powdered or flaked) Cu and its alloys are widely used in industry, including ammunition and weapons. Pure Cu is used for electric wiring, switches, plumbing, electroplating, etc. Flaked Cu is used as insulation for liquid fuels (Ref 13, p299) and powdered Cu is used in primary and pyrotechnic mixts (See Addnl Refs B,F,G, H & I). It produces on burning a bluish-green coloration. Uses of Cu-Zn alloy *(brass)* are listed in Vol 2, pB261. The principal military use of brass was formerly for the manuf of cartridge cases (See Vol 2, pC78-R), but now other materials are used, such as steel, plastics and colloided smokeless propInts. Another Cu alloy, *bronze* (Cu 90 Sn 10%) was formerly used for casting gun barrels (Ref 13, p 167), but now steel is used for that purpose. The so-called *gilding metal* (Cu 90 & Zn 10%) has been used as a jacket for lead-alloy bullets (See Vol 2, p B327-R, under Bullets, Metal Jacketed)

Examples of uses of Cu powder in some reactions for the prepn of expls are given by Davis (Addnl Ref C, pp 134 & 158)

Action of expls and materials other than plastics on metals (including Cu) used in ammunition were studied at PicArsn by Warren, Eriksen and Sheffield (See Addnl Refs A, D & E)

US Army and Navy requirements for powdered Cu used in pyrotechnic compositions are listed in Specification JAN-C-768, entitled

Copper, Powdered (For Use in Ammunition). It is required that powdered Cu shall be produced from electrolytic material with a minimum Cu content of 99%. Granulation requirement is: minimum 99.5% shall pass thru No 200 US Standard Sieve (See Table 1, p A674 in Vol 1)

Accdg to Kirk & Othmer (Ref 8a), mixts of Cu pdr with organic chlorinated compds have been used as color intensifiers in some pyrotechnic compns

Refs: 1) Mellor **3** (1923), 1-116 2) Thorpe **3** (1939), 341-53 3) J. Newton, "Metallurgy of Copper," Wiley, NY(1942) 4) I.L. Bray, "Non-Ferrous Production Metallurgy", Wiley, NY (1947) 5) O.W. Ellis, "Copper and Copper Alloys", Amer SocMetals, Cleveland, Ohio (1948) 6) Kirk & Othmer **4** (1949), 391-467 7) G.D. Van Arsdale, "Hydrometallurgy of Base Metals", McGraw-Hill, NU(1953) 8) R.W. Ruddle, "The Physical Chemistry of Copper Smelting", Inst Mining & Metallurgy, London (1953) 8a) Kirk & Othmer **11** (1953), 324 9) A. Butts, "Copper. The Science and Technology of the Metal, Its Alloys and Compounds", Reinhold, NY (1954) (ACS Monograph No 122) 10) Gmelin Syst Nr **60** Teil A (1955) & Teil B(1958) 11) Sax (1957), 502 12)Ullmann **11** (1960), 119-231 13) Cond-Chem Dict (1961), 298-99 14) P. Queneau, "Extractive Metallurgy of Copper, Nickel and Cobalt", Interscience NY (1961), 33, 205, 225, 317 & 401 15) Anon, "Properties of Materials Used in Pyrotechnic Compositions" US Army Materiel Command Pamphlet, **AMCP-706-187,** Washington, DC (1963), 105-08.

Addnl Refs on Cu: A)K.S.Warren, PATR **1152** (1942) (Copper (or its alloys, such as brass) is attacked by LA only when moisture is present. The resulting cupric azide is very sensitive and dangerous to handle) (See also Vol 1, p A 532)) B)W.J. Wiswesser, PATR **1177** (1942) (An aluminum scrap contg ca 4.3% Cu was, shown to be practically as suitable as specification grade Al for use in some pyrotechnic flares) C) Davis (1943), 134 & 158 (Cu powder has been used in manuf of some expls. For example, TNB can be obtd by refluxing for 2 hrs picryl chloride with Cu pdr in hot aq alc; and HNDPhA can be obtd by boiling picryl chloride in nitrobenzene soln with Cu pdr for a short time) D) L.H. Eriksen, PATR's **1325** (1945), **1388** (1944) & **1451** (1944) [Storage, up to 2 years, of US military expls (such as TNT, RDX, Compns A & B, PETN, Pentolite, Tetrytol, Haleite and Ednatol) in contact with strips of Cu, brass or mild steel plated with Cu, at atmospheric temp or at 50°, had very little effect on metals, provided all the components were dry. Some tarnishing and corrosion of metal took place with some expls. More detailed info on this subject may be found in Addnl Ref E] E) O.E. Sheffield, PATR **1783** (1950), "Effects of Materials on the Properties of Explosives" (Action of many expls, proplnts and pyrotechnic compns on various substances, including Cu, its alloys brass & bronze and Cu-plated steel was investigated and tabulated. Results of investigations shown in this very extensive report are essentially the same as mentioned in Addnl Ref D) F) S. Kinoshita & T. Sakamaki, Japan P 2498 (1953) & CA **48,** 6700 (1954) [Electric detonator compns, such as: Cu (or Sb) powder 80, Ph mono-resorcinate 10 & $KClO_3$ 10%, with a binding agent] G) A.R. Lusardi & J. Wingler, PATR **2516** (1958) [Examples of some pyrotechnic compns analyzed at PicArsn: a) Mg(pdr)(coated) 12, Cu (pdr) 2, Ba nitrate 62, hexachlorobenzene 21 & linseed oil 3% b) Mg 48, Cu 2, Ba nitrate 16, K perchlorate 25, hexachlorobenzene 6 & asphaltum 3% (or paraffin oil 2 & dextrine 1%) c) Mg 46, Cu 8, Ba nitrate 36, Na oxalate 8, linseed oil 1% castor oil 1% d) Cu 21, Ba nitrate 68, Na oxalate 5, sulfur 4 & castor oil 2% e) Cu 15.75, Ba nitrate 51.0, K nitrate 18.50, Na oxalate 3.75, sulfur 5.60, charcoal 3.90 & castor oil 1.50%] H) Ellern (1961), 99-100 & 275 [Cu can be introduced in pyrotechnic compns as powdered metal or as its salts or complexes. Examples of compns using Cu metal are: a) Colored Flare (green)-Ba nitrate 59, hexachlorobenzene 21, Mg 16, Cu (dust) 2 & linseed (or castor) oil 2% b) Colored High Intensity Star (green) - Mg 15, hexachlorobenzene 15, Cu (pdr) 2, Ba nitrate 66 & oil 2% c) Tracer (green) - Mg 48, gilsonite 3, hexachlorobenzene 6, Cu(pdr) 2, Ba nitrate 16 & K perchlorate 25%) I) L. LoFiego & G.G. Butters, "Naval Pyrotechnic Formulas", NOLM (Naval Ordnance Laboratory Memorandum) **9292** (1947), Washington 25, DC (Naval Signal Cartridge MK3, Formula 2: Copper dust 1.9, Ba nitrate 67.2, hexachlorobenzene 14.7 Mg powder 14.7 & linseed oil 1.5%. Burns 5 sec with green light at altitude 250 ft)

Copper-64 (Cu^{64} and **Copper - 67** (Cu^{67})-Artificially obtained radioactive isotopes, emitting both gamma and beta rays. Half life of Cu^{64} is 12.8 hrs and of Cu^{67} 61±1 hrs. Their prepn & props are given in Refs 1 & 2 (See also Ref 3). DeMent (Ref 4, p 9) proposed to use "radioactive copper" (not specified which isotope), admixed with radioactive iron (Fe^{55}) and other ingredients, in smoke producing compns
Refs: 1) H.T. Easterday, PhysRev **91,** 653-54 (1953) (Prepn & props of Cu^{67}) 2) Gmelin, Syst Nr **60,** Teil A(1955), 726 & 736 (Prepn & props of Cu^{64}); 728 (Prepn & props of Cu^{67}) 3) Sax (1957), 502 (Toxicity & radiation hazard of Cu^{64}) 4) J. DeMent, USP 2995556 (1961) (Smoke producing compns)

Copper, Analytical Procedures. Detection and determinations of copper in various substances are described in Refs 1 & 2

Analytical procedure for Cu powder intended for use in US military pyrotechnic compns consists in detn of *total copper* (Ref 3). For this, transfer ca 2g of accurately weighed sample to a 200ml electrolytic beaker, add ca 20ml of 50% nitric acid soln and boil until the sample is dissolved and all nitrogen oxides have been driven off. Add ca 50ml of 50% sulfuric acid soln, dilute the liquid to ca 150ml and electrolyze using sandblasted platinum electrodes with the cathode tared. Use a current density of ca 3amp per $100cm^2$ of cathode surface. When the deposition of Cu is complete, remove the beaker and wash the cathode with distd w and then twice with alcohol. Burn off alc while moving the electrode back and forth, cool in a desiccator and weigh

For *granulation test,* attach a bottom pan to US Std No200 sieve, place 100.0g of the sample on the sieve, cover it and shake the ensemble for 5 mins either by hand or mechanically by means of a shaker geared to produce 300±15 gyrations per minute and 150±10 taps of the striker per minute. Transfer any material left on the sieve to a tared weighing dish and weigh accurately. The difference betw 100.0g and wt of the residue is the percentage passing thru the sieve
Refs: 1)Gmelin, Syst Nr **60,** TlA, Lfg 2(1955), 1285-1378 2) Vogel, InorgAnalysis (1961), 358-60, 496-500, 608-11 3) Std Methods ChemAnalysis, **1**(1962), 395-42 4) US Military Specification **MIL-C-768,** Copper, Powdered (For Use in Ammunition)

List of Copper Compounds

Copper (I) acetaldehyde Catalysts. See Vol 1, p A74-L

Copper Acetates.
Cuprous Acetate, $Cu(C_2H_3O_2)$; wh crysts, decompg rapidly in air; can be prepd by heating cupric acetate to ca 250° (Ref 1). Cuprous acetate was patented by DeMent (Ref 7) as an ingredient of the following smoke-producing compn: cuprous acetate 1.5, K iodide 0.6, K iodate 0.6, K bromate 0.7, K (or Na) chlorate 0.5 to 1.0 & sulfur (or charcoal) 0.5 to 0.8 part. Deflagration of this compn produced abundant violet-grey smoke-*Cupric Acetate,* $Cu(C_2H_3O_2)_2 \cdot H_2O$; grn-blue pdr decompg beginning at 240°; can be prepd by the action of AcOH on CuO or by other methods (Ref. 2). The cupric salt can serve as a basic material in the prepn of Cu(II) acetoarsenite, an ingredient of some pyrotechnic compns. The so-called Ruggieri's Russian Fire compn contained: Cu acetate 4, Cu sulfate 2 & Amm chloride 1 part (Ref3)
Refs: 1) Beil **2,** 109, (48) & {188} (Cuprous acetate) 2) Beil **2,** 110, (48) [115] & {188} (Cupric acetate) 3) Davis (1943), 60 4) Kirk & Othmer **4** (1949), 468 5) Ullmann **6** (1955), 793 6) Sax (1957), 502 7) J. DeMent, USP 2995526 (1961), p 10 8)Gmelin, Syst Nr **60,** TlB, Lfg 2(1961), 677-88

Copper (II) Acetoarsenite or Cupric Arsenate-acetate [called Paris Green or Schweinfurt Green and in German, Kupfer (II) - arsenitacetat or or Schweinfurter Grün], $Cu(C_2H_3O_2)_2 \cdot 3Cu(AsO_2)_2$; emerald grn pdr; very toxic. Can be prepd either from Cu(II) acetate & Cu(II) arsenite or from $CuSO_4$, AcOH & $NaAsO_2$ (Refs 1, 2, 3, 4 & 5). Was used as an insecticide and in some pyrotechnic compns giving blue light. Some compns are listed in Addnl Refs A, B, C & D
Refs: 1) Beil **2,** 110, (48) & [116] 2) Kirk & Othmer **7** (1951), 884 3) Ullmann **3** (1953), 846 (Schweinfurter Grün) 4) Sax (1957), 502 (Cu acetoarsenite) & 983 (Paris Green) 5) CondChemDict (1961), 299 6) Gmelin, Syst Nr **60,** TlB, Lfg 2 (1961), 963-65
Addnl Refs: A) G.J. Schladt, PATR **357** (1933) [One of the earlier blue light rifle signal compns developed at PicArsn contained K chlorate 60, Cu

acetoarsenite 20, calomel 13 & shellac 7%. Pellets compressed at 6000psi; burned at the rate of 2.8 in/min developing 180 CP/sq in; its ignition temp was 315° (5 sec) and impact test 13" with 2kg wt. An improved mixt, designated as **BL-1**, developed at PicArsn contained K perchlorate 65, Cu acetoarsenite 25 & Cu resinate 10%. It produced, on burning at the rate 2.7 in/min, a satisfactory blue light of CP 520/sq in; ignition temp 440° (5 sec) and impact sensitivity 20" with 2kg wt. The light was visible at greater range than 6000 yds, but in presence of moisture and dust in the atm the blue color tends to turn green. Incorporation of Mg(ca5%) increased CP but diluted the blue color] B) Davis (1943), pp 70 & 85 (Formulations of several pyrotechnic compns contg Paris green) C) Izzo (1950), 213 [Blue star compns: a) Paris green 20, K chlorate 50, Ba nitrate 17, shellac 10 & gum arabic (or dextrine) 3% b) Schweinfurt green 31, K chlorate 59, anthracene 6.5, lactose 2 & methylcellulose 1.5%, c) Schweinfurt green 15, K chlorate 80 & shellac 5% D) L. LoFiego & G.G. Butters, NOLM **9292** (1947)(Navy Blue Light, Mark 1, Formula 3: Paris green 32.6, Ba chlorate 39.8, Ba nitrate 19.4 & stearic acid 8.2%; chge 55g burns 30sec. Submarine Identification Flare (green): Paris green 14.0, Ba nitrate 66.0, Mg powder 15.0, asphaltum 3.0 & linseed oil 2.0%; burning time 55sec)

Copper Acetylides and Derivatives. See in Vol 1 of Encyclopedia the following items: *Cuprous Acetylide,* p A72-R; *Cuprous Acetylide Chloride,* p A74-L; *Cuprous Hydrogen Acetylide,* p A74-L; *Cupric Acetylide,* p A74-L and *Copper Acetylides Analytical Procedures,* pp A74 to A76 [See also Gmelin, Syst Nr**60,** TlB, Lfg 2(1961), pp 632-46 (Kupferacetylenide)]

Copper (I) Acetylide in Electric Primer Mixtures. Accdg to Ellern (1961), 67 & 272, cuprous acetylide has been used in some electric primer mixts, such as: Cu_2C_2(or Diazodinitrophenol or Tetra cene) 20, K chlorate 60, charcoal 15 & NS 5%. This mixt is suitable for ignition rather than for detonation

Copper (I), amide. $CuNH_2$, olive-grn unstable solid which explodes on heating. Can be prepd by treating soln of Cu nitrate in liq ammonia with K amide or by other methods

Refs: 1) F.F. Fitzgerald, JACS **29** 657(1907) 2) R. Juza,ZAnorgChem **231,** 127(1937) 3) Gmelin Syst Nr **60,** Teil B1 (1958), 155

Copper (II) ammine Chlorides. Several complexes, of general formula $CuCl_2 \cdot xNH_3 \cdot yH_2O$, are listed in Ref 2, Izzo (Ref 1, p 214) lists the following blue signal light compn contg one of the complexes: K perchlorate 73 the complex 18 stearine 6 & asphaltum 3%
Refs: 1) Izzo(1950), 214 2) Gmelin, Syst Nr **60,** TlB, Lfg 1(1958), pp 295-302

Copper (II) ammine Explosive Complexes. Following complexes are listed in Vol 1 of this Encyclopedia: *Copper (II) ammine Azides,* p A280 (See also Ref, p 149); *Copper (II) ammine Bromates* p A281(See also Ref, p 387); *Copper (II) ammine Chlorates,* p A281(See also Ref, p 334); *Copper (II) ammine Iodates, p A*281 (See also Ref, p 428; *Copper (II) ammine Nitrates,* pp A280 (See also item listed below); *Copper (II) ammine Perchlorates,* p A281(See also Ref, p 340)
Ref: Gmelin, Syst Nr **60,** TlB, Lfg 1(1958) 149, 334, 340, 387 & 428

Copper (II) ammine Nitrates. Several complexes of general formula, $Cu(NO_3)_2 \cdot xNH_3 \cdot yH_2O$, are listed in Ref 2, pp 179-82. Izzo (Ref 1, p 214) lists the following blue signal light compn contg one of the complexes: K chlorate 53, "nitrato di rame ammonicale" 26, charcoal 16 & sulfur 5%
Refs: 1) Izzo (1950), 214 2) Gmelin, Syst Nr **60**, TlB, Lfg 1 (1958), pp 179-82

Copper (II) ammine Sulfates [Not to be confused with Copper (II)-ammonium Sulfate] . Several complexes of general formula $CuSO_4 \cdot xNH_3 \cdot yH_2O$ are listed in Ref 2, pp 560-62. Izzo (Ref 1, p 214) lists the following blue light signal compn contg one of the complexes: K chlorate 54, "Solfato di rame ammonicale" 27.5 & charcoal 18%
Refs: 1) Izzo(1950), 214 2) Gmelin, Syst Nr **60** TlB, Lfg 1(1958) 3) Sax(1957), 502 (Lists the compd $CuSO_4.4NH_3.H_2O$ as "Copper ammonium sulfate"

Copper (II) ammonia Solution. See Cuprammonium Solution

Copper (II) ammonium Chloride [Not to be confused with Copper (II) ammine Chloride]. $CuCl_2$.-$2NH_4Cl.2H_2O$; pale blue crysts, losing all w of crystn ca 110-20°; decomp at higher temps. Can be obtained by crystn from an aq soln of Cu(II) chloride (1mole) and Amm Chloride (2 moles), or by other methods (Refs 1 & 3)

It was listed by Davis (Ref 2) as one of the ingredients of the following blue light compn: Cu-Amm chloride 2, K chlorate 8, calomel 6 & milk sugar 3 parts. Weingart (Ref 3, p 7) states that he obtained very good results when using this salt in blue light compns and he did not use calomel

Refs: 1) Mellor **3**(1923), 185-86 (called Dihydrated Ammonium Tetrachlorocuprate) 2) Davis (1943), 65 3) Weingart (1947), 7 4) CondChem-Dict (1961), 300

Copper (II) Ammonium Sulfates [Not to be confused with Copper (II) Sulfate Ammoniated]. The following compds are described by Mellor (Ref 1, p 255): a)*Ammonium Dicupric Sulfate,* $(NH_4)_2$-$SO_4.2CuSO_4$; grn prisms, d 2.85, decomp ca 350°. Can be prepd by melting $CuSO_4.5H_2O$ with 3 times its wt of $(NH_4)_2SO_4$ at ca 300° b) *Ammonium Cupric Sulfate, Hexahydrate,* $(NH_4)_2SO_4$.-$CuSO_4.6H_2O$; pale blue crysts, d ca 1.9, losing w at 130°to form a col *anhydrous* salt, d 2.348, which decomp at a higher temp

US Navy Blue Light, Mark 1, Formula 2 compn contained: Cu-Amm sulfate 19.0, Ba chlorate 53.0, CuO 14.0, red arsenic trisulfide 5 & shellac 9% (Ref 2)

Refs: 1) Mellor **3**(1923), 255 (Called Ammonium Disulfate Cuprate) 2) L. LoFiego & G. Butters, NOLM **9292**(1947) 3) Sax (1957), 502 (Lists the compd $CuSO_4.4NH_3.H_2O$ as "Copper Ammonium Sulfate") 4)Gmelin, Syst Nr**60,** TlB, Lfg 1(1958) not found

Copper Azides. The following salts are described in Vol 1 of this Encyclopedia: *Cupric Azide,* pp A532 to A533 (See also Ref, p 145); *Cuprous Azide,* pp A534 to A535 (See also Ref, p 142); *Cupric Azide, Basic,* p A533 (See also Ref, p 154); *Cupric Azide complexes* pp A533 to A534 (See also Copper Azide Explosive Complexes of Cirulis & Straumanis)

Ref: Gmelin, Syst Nr **60,** TlB, Lfg 1(1958)

Copper Azide Explosive Complexes of Cirulis & Straumanis. The following complexes are listed Gmelin (Ref 4). Their prepns are described in Refs 1, 2, or 3

Copper (II) allylamine Azide, $[Cu(C_3H_5.NH_2)_2](N_3)_2$; grn crysts, decomp with crackling noise at 179-80° (Ref 1, p 309 &Ref 4, p 151)

Copper (II) aniline Azide, $[Cu(C_6H_5.NH_2)](N_3)_2$; grn-brn ndls, expl at 169°; impact test value 10 cm with 1 kg wt (Ref 3, p 350 & Ref 4, p 151)

Copper (II)-o-anisidine Azide, $\{Cu[C_6H_4(OCH_3)NH_2]\}(N_3)_2$; dk-brn ndls, detonates violently at 125°(Ref 1, p 315 & Ref 4, p 151)

Copper (II) benzimidazole Azide, $[Cu(C_7H_6N_2)](N_3)_2$; brn solid, decomposes explosively at 183-85°(Ref 1, p 321 & Ref 4, p 153)

Copper (II) bis(ethylenediamine) Azide, $[Cu(en)_2](N_3)_2$; where (en) is designation of $(H_2N.CH_2.CH_2.NH_2)$; blue or violet crysts, mp 180°; expl on contact with block heated to 212-16°, easily sol in w; diffc sol in alc; insol in eth. Can be prepd by treating $Cu(N_3)_2$ with two moles of ethylenediamine or by other methods (Ref 2, p 339; Ref 4, p 152 & Ref 5)

Copper (II)bis(methylamine) Azide, $[Cu(CH_3.NH_2)](N_3)_2$; grn crysts, mp 126-28°, expl at 180-90°(Ref 3, p 345; Ref 4, p 151)

Copper (II)bis(pyridine) Azide, $[Cu(py)_2](N_3)_2$, where (py) is designation of C_5H_5N. See Vol 1 of Encyclopedia p A533 and also Ref 4, p 152

Copper(II)bornylamine Azide, $[Cu(C_{10}H_{17}.NH_2)_2](N_3)_2$; grn crysts, expl mildly at 207-08° (Ref 1, p 311 &Ref 4, p 152)

Copper(II)-iso-butylamine Azide $\{Cu[(CH_3)_2CH.CH_2.NH_2]_2\}(N_3)_2$; grn crysts, mp 139-40° expl at 198-200°(Ref 3, p 347 & Ref 4, p 151)

Copper-n-butylamine Azide, $[Cu(CH_3.CH_2.CH_2.CH_2NH_2)](N_3)_2$; grn lfts, mp 113-14°, decomp explosively at 196°(Ref 3, p 346 & Ref 4, p 151)

Copper(II)cinchonine Azide, $[Cu_2(C_{19}H_{22}N_2O)](N_3)_4$; dk-brn solid, decomp explosively ca 169° (Ref 1, p 322 & Ref 4, p 154)

Copper (II)collidine Azide, $\{Cu[C_5H_2(CH_3)_2N]_2\}(N_3)_2$; grn-brn amorphous pdr, expl at 198-200° (Ref 1, p 312 & Ref 4, p 153)

Copper(II)ethylamine Azide, $[Cu(C_2H_5.NH_2)_2](N_3)_2$; grn crysts; expl on contact with a hot plate, but not on slow heating; insensitive to

friction or impact (Ref 3, p 345 & Ref 4, p 151)
Copper(II)ethylenediamine Azides. See Copper (II)bis(ethylenediamine) Azide, and Copper (II)-mono(ethylenediamine) Azide
Copper(II)hexamethylenetetrammine Azide, $[Cu_2(C_6H_{12}N_4)](N_3)_4$; grn ndls, expl at 180-85° impact sensitivity 10cm with 1 kg wt (Ref 1, p 321 & Ref 4, p 153)
Copper(II)-2,4-lutidine Azide, $\{Cu[C_5H_3(CH_3)_2N]\}(N_3)_2$; brn amor ppt, expl at 208-09° with sharp report; can be detonated by impact (Ref 1, p 319 & Ref 4, p 153)
Copper(II)-2,6-lutidine Azide, $\{Cu[C_5H_3(CH_3)_2N]_2\}(N_3)_2$; dk grn crysts, expldg at 202-03° (Ref 1, p 311 & Ref 4, p 153)
Copper(II)monoethylenediamine Azide, $[Cu(en)](N_3)_2$; dk grn ndls, mp 175-77°, expl mildly at 210°; impact sensitivity 25cm with 1 kg wt (Ref 3, p 347; Ref 4, p 152 & Ref 5)
Copper(II)monopyridine Azide, $[Cu(py)](N_3)_2$; brn solid; expl props similar to those of copper (II)bis(pyridine) azide (Ref 3, p 350 & Ref 4, p 153)
Copper(II)-α-picoline Azide, $\{Cu[C_5H_4(CH_3)N]\}(N_3)_2$; dk brn or dk grn crysts, expl violently at 204-05° or on impact (Ref 1, p 318 & Ref 4, p 153)
Copper(II)-β-picoline Azide, $\{Cu[C_5H_4(CH_3)N]_2\}(N_3)_2$; one form exists as dk grn crysts, expl at 207-15° or on impact; another form, brn ppt, expl at 210-11°, or on impact (Ref 1, p 311 & Ref 4, p 153)
Copper (II) propylamine Azide $[Cu(C_3H_7.NH_2)_2](N_3)_2$; grn pdr, mp 109°, expl at 187° (Ref 3, p 346 & Ref 4, p 151)
Copper (II) pyridine Azide. See Copper (II) bis (pyridine) Azide and Copper (II)monopyridine Azide
Copper (II) quinidine Azide, $[Cu_2(C_{20}H_{24}N_2O_2)](N_3)_2$; dk brn ppt, expl at 152-54° (Ref 1, p 322 & Ref 4, p 154)
Copper(II)quinine Azide, $[Cu_2(C_{20}H_{24}N_2O_2)](N_3)_4.3H_2O$; grn-brn ppt, expl at 149-51° (Ref 1, p 322 & Ref 4, p 153)
Copper(II)quinoline Azide, $[Cu(C_9H_7N)](N_3)_2$; grn-brn ndls, expl at 207-08° or on impact (Ref 1, p 320 & Ref 4, p 153)
Copper(II)-iso-quinoline Azide, $[Cu(_9H_7N)_2](N_3)_2$; yel-grn ppt, expl at 197-200° (Ref 1, p 313 & Ref 4, p 153)
Copper(II)tolidine Azide, $[Cu(CH_3.C_6H_4.NH_2)](N_3)_2$; o-isomer, brn-grn crysts, decomp explosively ca 123°; m-isomer, brn-grn amor pdr, decomp explosively ca 157°; and p-isomer, brn-grn cryst, decomp explosively ca 135°. All three isomers are sensitive to impact (Ref 1, p 314 & Ref 4, p 151)
Copper(II)xylidine Azide $\{Cu[(CH_3)_2C_6H_3.NH_2]\}(N_3)_2$. Its 1, 3, 4 isomer, brn amor ppt, decomp explosively at 106-08° and 1, 4, 5 isomer, dk brn cryst ppt, expl at 130° (Ref 1, p 315 & Ref 4, p 151)
Refs: 1) A. Cirulis & M. Straumanis, J Prakt-Chem **160,** 307-28 (1941) 2) M. Straumanis & A. Cirulis, ZAnorgChem **251,** 335-40 (1943) 3) A. Cirulis & M. Straumanis, ZAnorgChem **251,** 341-54(1943) 4) Gmelin, Syst Nr **60,** Teil B, Lieferung 1 (1958) 5) Beil 4, III Ergänzung (1962), pp {485-861}

Copper Azidodithiocarbonate. See p A636-R in Vol 1 of Encyclopedia

Copper(I)-barium Fulminate. See under Copper Fulminate Explosive Complexes

Copper(II)bis(ethylenediamine) Azide. See under Copper Azide Explosive Complexes of Cirulis & Straumanis

Copper(II)bis(ethylenediamine) Bromate, $[Cu(en)_2]BrO_3.H_2O$; dk viol ndls; expl on heating. Method of prepn is in Ref 1
Refs: 1) J. Amiel, CR **200,** 672 (1935) 2) Gmelin, Syst Nr **60,** T1 B, Lfg 1 (1958) - not found 3) Beil **4,** Erg III (1962), {486}

Copper(II) bis(ethylenediamine) Chlorate, $[Cu(en)_2](ClO_3)_2$; red-viol crysts; expl on rapid heating to 168-73° or on impact. Can be prepd by warming $Cu(OH)_2$ (1 mole) with NH_4ClO_3 (2 moles) in 10% aq soln of ethylenediamine (2 moles), followed by crystn (Ref 1 & 3). Its *monohydrate,* prepd by Amiel (Refs 2 & 4), was recommended for use in primers
Refs: 1) W. Lange, Ber **59,** 2108(1926) 2) J. Amiel, CR **199,** 202(1934) 3) Gmelin, Syst Nr **60,** T1B, Lfg 1(1958), 335 4) Beil **4,** Erg III (1962), p {486}

Copper(II)bis(ethylenediamine) Dichromate, $[Cu(en_2](Cr_2O_7)$; brick-red crysts. Was prepd by by adding ethylenediamine (2 moles) to a soln contg $K_2Cr_2O_7$ (1 mole) and $CuSO_4$ (1 mole). Its

expl props were not detd
Refs: 1) Beil **4,** 233 2) N. Parravano & A. Pasta Gazz **37 II,** 256(1907) & JCS **92 I,** 962(1907)

Copper(II)bis(ethylenediamine) Iodate, $[Cu(en_2]-(IO_3)_2.2H_2O$; red-viol crysts, decomp explosively on heating. Its method of prepn is similar to that of the chlorate
Refs: 1) Beil **4,** [678] 2) W. Lange, Ber **59,** 2112(1926) 3) Gmelin, Syst Nr **60,** TlB, Lfg 1 (1958), 429-30

Copper (II)bis(ethylenediamine) Nitrate, $[Cu(en_2]-(NO_3)_2$; small, dark viol crysts melting with expl decompn. Was obtd by Amiel (Refs 1, 2 & 4) on heating aq soln of ethylenediamine with cupric nitrate. The dihydrate reported by some investigators was shown by Johnson & Bryant to be an anhydrous salt (Ref 3)
Ref: 1) Beil **4,** 233, (400), [6797] & {486} 2) J. Amiel, CR **199,** 202(1934) 3) C.H. Johnson & S.A. Bryant, JCS **1934,** 1784 4) Gmelin, Syst Nr **60,** TlB, Lfg 1 (1958), 183

Copper (II)bis(ethylenediamine) Nitrite, $[Cu(en)_2]-(NO_2)_2$; dk red crysts, mp 128° (decomp); can be prepd by treating $[Cu(en)_2]I_2$ with $AgNO_2$
Refs: 1) Beil **4,** [679] & {486} 2) G.T. Morgan & F.H. Burstall, JCS **1927,** 1264 3) Gmelin, Syst Nr **60,** TlB, Lfg 1(1958), 160

Copper (II)bis(ethylenediamine) Perchlorate, $[Cu(en)_2]\ (ClO_4)_2$; blue-viol prisms, expl violently on heating. Can be prepd by evapn under vacuum, over concd H_2SO_4, an aq soln of Cu sulfate (1 mole) mixed with ethylenediamine (2 moles) and Na perchlorate (2 moles). Its *monohydrate,* viol-blue ndls, expl at 264° or on contact with concd H_2SO_4. A hydrate with with $0.5H_2O$ was also reported
Refs: 1) Beil **4,** [678] & {486} 2) J. Amiel, CR **199,** 202 (1934) 3) P. Pfeiffer & H. Glazer, JPraktChem **151,** 137(1938) 3) Gmelin, Syst Nr **60,** TlB, Lfg 1(1958), 342

Copper(II)bis(ethylenediamine) Persulfate, $[Cu(en)_2]\ (S_2O_8)$; purple-red crysts, expl on heating, on impact, or on warming with concd H_2SO_4. Was prepd by adding an aq soln of $K_2S_2O_8$ to an aq soln of $CuSO_4$ (1 mole) contg ethylenediamine (2 moles)
Refs: 1) Beil **4,** [678] 2) G.T. Morgan & F. H. Burstall JCS **1927,** 1262 & 1265-6 3) Gmelin, Syst Nr **60,** TlB, Lfg1, 590-91

Copper Borates. See in Gmelin, Syst Nr **60,** TlB Lfg 2, 628-31. The metaborate is listed in Vol 2, p B247, under Borates

Copper(II) Bromate. See Cupric Bromate under Bromates in Vol 2 p B303-R and Gmelin, Syst Nr **60,** TlB, Lfg 1(1958), 387

Copper (II) Bromotetrazole. See under 5-Bromo-1, 2, 3, 4 - tetrazole in Vol 2, p B316-L and also conf PATR **2136**(1955) by R.J. Gaughran & J.V.R. Kaufman

Copper (I)-calcium Fulminate. See under Copper Fulminate Explosive Complexes

Copper (II) Carbonates. Only basic salts are known, of which $Cu_2(OH)_2CO_3$, mw 221.17, is a commercial product, known as *cupric carbonate* or *artificial malachite.* It is a dk grn toxic pdr, dec ca 200 °, d 3.7-4.0; insol in w; sol in acids. Can be prepd by adding Na_2CO_3 to a cold aq soln of $CuSO_4$ and filtering & washing the resulting ppt. They have been used in pigments, insecticides, fungicidies and pyrotechnics (Refs 1-4). Weingart (Ref 2a) does not recommend the use of Cu carbonates in pyrotechnics. Some pyrotechnic formulations are given in AddnlRefs A, B & C. US Military requirements for basic cupric carbonate for use as analytical reagent are listed in Ref 7
Refs: 1) Mellor **3** (1923), 267-80 2) Thorpe **3** (1939), 355-56 2a) Weingart (1947), 7 3) Kirk & Othmer **4** (1949), 468 4) Ullmann **11** (1960), 234 5) Gmelin, Syst Nr **60,** TlB, Lfg 2(1961), 650-53 6) CondChemDict (1961), 300 7) US Military Specification MIL-C-11163
Addnl Refs: A) G.J. Schladt, PATR **357**(1933) [Blue light pyrotechnic compns developed at PicArsn: a) Cu carbonate 30, K perchlorate 60 & asphaltum 10%; rate of burning 2.0 in/min and CP 180/sq in b) Cu carbonate 10, K perchlorate 60 & Cu resinate 10%; rate of burning 1.8 in/min and CP 73/sq in] B) Izzo (1950), 215 (violet light compn for stars: Cu carbonate 10, K chlorate 58.5, Sr chlorate 14.5, shellac 7 & sulfur 10%) C) Ellern(1961), 99 [Compns for blue flare candles contg Cu carbonate, K chlorate, calomel and shellac (serving as binder and color inten-

sifier) possess much lower luminosity than corresponding compns for red, orange, yellow or green flare candles]

Copper (II) Chlorate. See Cupric Chlorate under Chlorates in Vol 2, p C188 and also Gmelin, Syst Nr **60,** TlB, Lfg 1(1958), 332-33

Copper Chloride See under Chlorides in this Vol

Copper (II)chloride Azide. See under Azides in Vol 1, p A533-R

Copper Chloride Basic. See Copper Oxychloride

Copper (II) Chlorite. See Cupric Chlorite under Chlorites in this Vol

Copper (II) Chlorotetrazole. See under Chlorotetrazole and Derivatives in this Vol

Copper (II) Chromate. See under Chromates in this Vol

Copper Chromite. It is mentioned under Chromites in this Vol

Copper Compounds for Smoke Production. DeMent (Ref) proposed to use several copper-ion contg mixts as smoke-producing pyrotechnic compns. These mixts are listed under Copper Chloride in this section on Chlorides and under Copper - 64 and Copper - 67, Copper Acetate, Copper Oxide & Copper Stearate in this section on COPPER AND ITS SALTS
Ref: J. DeMent, USP 2995526(1961), pp 9-10

Copper Cyanamide. See under Cyanamide and its Salts.

Copper (II) -dicopper(I) Fulminate. See under Copper Fulminate Explosive Complexes

Copper (II)-disodium Fulminate. See under Copper Fulminate Explosive Complexes

Copper(II)ethylenediamine Complexes. See under Copper(II)bis(ethylenediamine) -, Copper(II)mono (ethylenediamine) - and Copper (II)tris(ethylenediamine) Complexes

Copper Formates. See Beil **2,** 15,(13),[20] & {23} and Gmelin, Syst Nr **60,** TlB, Lfg 2(1961), 665-77

Copper(I) Fulminate or Cuprous Fulminate, $Cu_2(CNO)_2$, mw 211.18 N13.27%, lt grey ppt with greenish tinge; expl violently on heating to ca 205° or on impact; stable in the dry state but oxidizes in presence of moisture; insol in w; heat of expln 508cal/g; less sensitive to impact than Hg or Cd fulminates (Refs 3,4,5,6 & 9). Was prepd by Wöhler & Martin by shaking Ag fulminate with Cu amalgam in water, in hydrogen atm (Refs 1 & 2). Sax (Ref 8) discusses toxicity and fire & expln hazards of fulminates. Cu(I) fulminate was used, accdg to Bebie (Ref 7), in detonators for gaseous coal mines
Refs: Beil **1,** 722 & (376) 2) L. Wöhler & F. Martin, Ber **50,** 591-92(1917) 3) Ibid, ZAngewChem **30,** 36(1917) 4) Ibid, SS **12,** 2 & 18(1917) 5) Colver (1918), 518 6) Davis (1943), 411-12 7) Bebie (1943), 51 8) Sax (1957), 727 (Fulminates) 9) Giua, Trattato **6**(1959), 425 (Fulminato di rame)

Copper Fulminate Explosive Complexes. The following complexes are described in the literature:
Copper(I)-barium Fulminate, $Ba[Cu(CNO)_3]\cdot 4H_2O$; col prisms (from w); expl on heating in a flame and from strong impact. Was prepd by treating Ba fulminate with an excess of cuprous chloride in w. Ba fulminate may be obtained by shaking Hg fulminate with Ba amalgam under w. Its uses were not specified (Ref 1 & Ref 2, p 2757)
Copper(I)-calcium Fulminate, $Ca[Cu(CNO)_3]\cdot 3H_2O$; col crysts, expl violently on heating or on impact. Its method of prepn is similar to that for Ba salt. Ca amalgam was used as a starting material (Ref 1 & Ref 2, p 2756)
Copper(II)-dicopper(I) Fulminate, $Cu[Cu_2(CNO)_4]\cdot 2H_2O$; lt grn lfts, expl violently on heating or impact; diffc sol in w or alc; does not lose w of crystn even at 80°. Was prepd by treating (at 30-40°) a satd aq soln of $Na[Cu(CNO)_2]$ with concd soln of Cu(II) nitrate. It is a good initiating agent slightly inferior to LA & MF (Ref 1; Ref 2, p 2755 & & Ref 3)
Copper(I)-disodium Fulminate, $Na_2[Cu(CNO)_3]\cdot 3H_2O$; col ndls turning bluish under influence of light; expl on heating and can be initiated but more difficultly than copper(I)-sodium fulminate. Was prepd by treating concd Na fulminate soln

with cuprous chloride at RT. Water of crystn could not be removed in vacuum over P_2O_5 (Ref 1 & Ref 2, p 2756)
Copper(I)-sodium Fulminate, $Na[Cu(CNO_2)]$; col crysts (from hot w), expl more violently than disodium salt on heating or impact. Was prepd by treating dil Na fulminate soln with cuprous chloride at 80°(Ref 1 & Ref 2, pp 2754-55)
Copper(I)-strontium Fulminate, $Sr[Cu(CNO)_4]$; $2H_2O$; col microscopic crysts, expl violently on heating or on impact. Was prepd by warming Sr fulminate in w with cuprous chloride. Sr fulminate was obtd by shaking Sr amalgam with Hg fulminate in w (Ref 1 & Ref 2, p 2755)
Refs: 1) Beil **1,** [777] 2) L. Wöhler & A. Berthmann, Ber **62,** 2754-57(1929) & CA **24,** 1348(1930) 3) Gmelin, Syst Nr **60,** TlB, Lfg 2(1961), 867

Copper(II)hydrazine Chlorate, $Cu(ClO_3)_2 \cdot 2N_2H_4$; col ppt, expl by itself without being touched. Was prepd by treating very cold Cu(II) chlorate alc soln with hydrazine hydrate
Refs: 1) W. Friederich & P. Vervoorst, SS **21,** 84 & 143(1926) 2) Gmelin, Syst Nr **60,** TlB, Lfg 1(1958), 335

Copper(II)-hydrazine Nitrate, $Cu(NO_3)_2 \cdot 2N_2H_4$; blue-grn pdr, expl on heating with crackling noise and evoln of reddish light. Can be prepd by treating alc soln of cupric nitrate with hydrazine hydrate
Refs: 1) K.A. Hofmann & E.C. Marburg, Ann **305** 221(1899) 2) Gmelin, Syst Nr **60,** TlB, Lfg 1(1958) 182

Copper Hydroxides. See under Copper Oxides and Hydroxides

Copper Hydroxychloride. See Copper Oxychloride

Copper(II) Iodate, $Cu(IO_3)_2$, wh to lt blue crysts, decomp rapidly on heating. Can be prepd by dehydrating its *monohydrate* (lt blue or blue-grn crysts), which can be prepd by treating a cupric salt soln with Na iodate
Refs: 1) J. Martinez-Cros & L. LeBoucher, Anales RealSoc EspañFisQuím(Madrid) **33,** 237 (1935) 2) Gmelin Syst Nr **60,** TlB, Lfg 1(1958), 426-27

Coppermethyl or Methylcopper, $CuCH_3$, yel unstable solid, expl by itself on drying and on exposure to the air. Was prepd by treating $Mg(CH_3)_2$ (or $LiCH_3$) with cupric iodide as described in the Ref. Only small quantity should be prepd at a time.
Ref: H. Gilman et al, JOC **17,** 1631-33(1953) & CA **48,** 1248(1954)

Copper (II) mono(ethylenediamine) Azide. See under Copper Azide Explosive Complexes of Circulis & Straumanis

Copper(II)mono(ethylenediamine) - tetraaquo Perchlorate, $[Cu(en)(OH)_4](ClO_4)_2$; dk bluish-violet nds, expl violently on heating. Was prepd by evapg an aq soln of cupric perchlorate and ethylenediamine in molecular proportions
Refs: 1) Beil **4,** [678] 2) G.T. Morgan & F.H. Burstall, JCS **1926,** 2026-27

Copper Naphthenate (formula indefinite), gr-blue solid, sol in gasoline & oils. Can be prepd by adding a soln of Cu(II) sulfate to soln of Na naphthenate, which is the most common salt of naphthenic acid (Ref 1). See also under Cobalt Naphthenate and Ref 3

Cu naphthenate manufd by the Nuodex Products Co, Elizabeth, NJ, under the name of "Fungicide B" was tested at PicArsn from the point of view of suitability for fungicide treatment of wood to be used for dunnage, crating and packing boxes of expls, such as TNT, Tetryl, Comp B, Comp C-4, 50/50 Pentolite & 60% Dynamite. It was shown that presence of naphthenate in wood caused no seriously adverse effect upon the stability of the expls tested (Ref 2)
Refs: 1) CondChemDict (1961), 302, 777-78 & 1047 2) S. Axelrod, PATR **1828** (1951) 3) Sax (1957), 504-05 [The formula for copper naphthenate is given as $Cu(C_6H_5COO)_2$ and mw as 221.9 (?)]

Copper Nitrates:
Cuprous Nitrate $CuNO_3$, wh pdr, not expl (Ref 6, p 162). Accdg to Ref 4, the existance of cuprous salt is questionable
Cuprous Nitrate $Cu(NO_3)_2$, bluish-wh crysts, decomp ca 160° with formation of basic nitrate. It was reported that cupric nitrate, when brought in contact with paper, caused its combustion and on contact with tin foil sparking was produced (Ref 9, p 56)

Copper Nitrate Trihydrate, $Cu(NO_3)_2.3H_2O$, mw 241.63, blue delq crysts, mp 114.5°; can be prepd by treating Cu or CuO with nitric acid, followed by evapn & crystn. It is an oxidizing material less dangerous than the anhydrous salt. There are also hydrates with 6, 9, & $2.5H_2O$ (Refs 1, 2, 4, 6, 7 & 8)

Cupric nitrates have been used as insecticides, in nitrating some org silicon compds, in electroplating solns, as catalysts, in analytical chemistry, and in textiles (Refs 4, 7 & 8). Accdg to Davis (Ref 3), Cu nitrate produces an emerald-green flame when used as an ingredient of some pyrotechnic compns

Refs: 1) Mellor **3** (1923), 280-87 2) Thorpe **3,** (1939), 358 3) Davis (1943), 60 4) Kirk & Othmer 4(1949), 471 5) Sax (1957), 505 6) Gmelin, Syst Nr **60,** TlB, Lfg 1 (1958), 162-69 7) Ullmann **11** (1960), 242-43 8) CondChemDict(1961), 302 9) Ellern(1961), 56

Copper(I) Nitride or Cuprous Nitride, Cu_3N, dk olive-grn or blk pdr, decomp explosively at ca 470° with evoln of light. Can be prepd by treating Cu or its salts, such as CuF_2, with gaseous NH_3 at high temps or by other methods

Refs: 1) F.F Fitzgerald, JACS **29**,659(1907) 2) R Juza & H. Hahn, ZAnorg Chem **244,** 134(1940) 3) Sax(1957), 505 4)Gmelin, Syst Nr **60,** TlB, Lfg 1(1958), 139-42

Copper Nitrites:

Cuprous Nitrite exists only in the form of double salts (Ref 3, p 157)

Cupric Nitrite, $Cu(NO_2)_2$ mw 155.59, olive-grn unstable ndls; mp decomp with rapid evoln of gas at 170-180°. Can be prepd from its *basic salt,* $Cu(NO_2)_2.3Cu(OH)_3$ (Ref 1; Ref 3, pp 157 & 161 & Ref 4). The complex *Copper (II) Tetrammine nitrite,* $[Cu(NH_3)_4](NO_2)_2$, olive-grn ppt, expl from impact or friction (Ref 3, p 159)

Refs: 1) Kirk & Othmer **4**(1949), 471-72 2) Sax (1957) - not found 3) Gmelin, Syst Nr **60,** TlB, Lfg 1(1958), 157-62 4) CondChemDict (1961), 302 (Basic copper nitrate)

Copper Nitroaminoguanidine. See p A212-R in Vol 1 of Encyclopedia

Copper Oxalates:

Cuprous Oxalate, $Cu_2C_2O_4.2H_2O$, fine brn ppt; heated alone in a crucible it changes to gray, then to greenish-gray giving off traces of w and then decomposes suddenly, almost explosively. Was prepd by Daniels (Refs 1 & 3) by adding cuprous sulfite, little by little, into a hot soln of an excess of oxalic acid

Cupric Oxalate, $Cu(C_2O_4).1/2\ H_2O$, mw 160.57, blue-wh ppt insol in w or alc. Can be prepd by adding to a boiling soln of a soluble oxalate (or oxalic acid) a soln of a Cu salt (Refs 2, 4, 6 & 7)

Davis (Ref 5) lists the following violet light pyrotechnic compn contg cupric oxalate: Cu oxalate 5, K chlorate 12, Sr oxalate 9 & shellac 3 parts

Refs: 1) Beil **2,** (224) 2) Beil **2,** 512 [486] & {1553} 3) L.S. Daniels, JACS **37,** 1169-70 (1915) 4) K.P. Chatterjee & N.R. Dhar, JPhysChem **28,** 1019(1924) & CA **19,** 203(1925) 5) Davis (1943), 85 6) Sax(1957), 505 7) Lange (1961), 246-47 8) CondChemDict (1961) - not found

Copper Oxides and Hydroxides:

Cuprous Oxide or Red Copper Oxide (Cuprite), Cu_2O, mw 143.08, red crysts, mp 1235°, decomp ca 1800°, d ca 6.0; insol in w; sol in acids and in Amm chloride & hydroxide. Can be prepd by the addn of bases to cuprous chloride or by the action of glucose on cupric hydroxide (Refs 1-8)

US Military requirements for cuprous oxide intended for use as a chemical reagent are listed in Specification **MIL-C-11163**

Cuprous oxide has been used in the prepn of cuprous salts, in ceramics, fungicides, pyrotechnics, electroplating and in antifouling paints. These paints were used extensively by the US Navy during WWII (Refs 3, 6, 7 & 9 and Addnl Refs E & F)

Cupric Oxide or Black Copper Oxide, CuO, mw 79.54, blk cubic crysts (Paramelaconite, d 6.40) or blk triclinic crysts (Tenorite, d 6.45), mp 1026° (decomp); insol in w; sol in acids, KCN & Amm chloride. Can be prepd by the ignition of cupric carbonate or nitrate (Refs 1-8)

US Military requirements for cupric oxide for use in pyrotechnic compns are listed in Specification **MIL-C-13600A**

Cupric oxide has been used in analytical chemistry (such as for detn of C & H in organic compds, by combustion), in storage batteries, electroplating, in catalysts, pyrotechnics,

primer & igniter compns, gasless delay compns, and in proplnts as burning rate modifiers (Refs 3, 6 & 7 and Addnl Refs A, B, C, D & G). See also Addnl Ref H

Accdg to Ellern (AddnlRef E, p 98), copper & its compds, notably CuO, are valuable adjuncts and intensifiers in the production of green light, but under certain conditions, CuO, in the presence of calomel, yields a beautiful blue color

Copper Peroxide, CuO_2. The existence of this explosive compd in pure anhyd state has not been proved, but the following information is reported in the literature: J. Thennard prepd in 1818 the peroxide as an olive grn ppt by treating alkaline cupric salt soln with an excess of H_2O_2. The compd was unstable; it evolved oxygen on standing. A similar, but purer, compd was prepd by G. Krüss in 1884 and then by L. Moser in 1907, by shaking $Cu(OH)_2$ with an excess of H_2O_2 at 0°, following by filtration and washing the ppt with w, alc & ether. The dried ppt was brn in color and could be exploded by touching it with a heated glass rod, or with a drop of of concd H_2SO_4 (Ref 5, p 133-37)

Copper Hydroxides:

Cuprous Hydroxide [Called Kupfer (I) - aquoxid in German], CuOH, mw 80.55, yel solid, mp - loses $0.5H_2O$ at 360°, d 3.4; insol in w, sol in acids (Ref 8, pp 248-49). Accdg to Gmelin (Ref 8, pp 97-8), no compds exactly corresponding to the above formula were known until P. Klason prepd CuOH in 1924 by treating purest CuI with aq NaOH soln. Methods of prepn of hydrated cuprous hydroxides, $CuOH.xH_2O$ are given in Ref 1, p 127

Cupric Hydroxide [Called Kupfer (II) - hydroxyd in German], $Cu(OH)_2$, mw 97.59, blue gelatinous substance, mp - loses H_2O forming CuO, d 3.368 insol in hot w, decomp by hot w; sol in acids & KCN. Can be prepd by interaction of a Cu(II) salt soln with an alkali (Ref 1, pp 142-45; Ref 3, p 474; Ref 4, p 504; Ref 5, pp 98-117; Ref 6, p 241; Ref 7, pp 301-02 & Ref 8, pp 246-47)

Cupric hydroxide has been used in prepn of Cu salts, in manuf of cuprammonium rayon, as a mordant, as a pigment, and as a starting material for prepn of Schweitzer's Reagent, first prepd in 1856 by a Ger chemist M.E. Schweitzer (1818 - 1860). This reagent consists of an ammoniacal soln of cupric hydroxide, and it can be prepd by adding KOH to a soln of $CuSO_4$ and NH_4Cl, followed by filtering and dissolving the ppt in 20% NH_4OH (3g of moist ppt per 1000ml of solvent)

This reagent dissolves cotton, silk and linen and is used in analytical chemistry (Ref 1, p 152 & Ref 5, pp 118-32). See also Hackh's (1944) 760

Refs: 1) Mellor **3** (1923), 116-51 (Cu oxides & hydroxides) 2) Thorpe **3** (1939), 353-55 3) Kirk & Othmer **4** (1949), 472-3 (Cuprous oxide); 473 (Cupric oxide); 474 (Cupric hydroxide) 4) Sax (1957), 505(Cupric); 519 (Cuprous); 504 (Cupric hydroxide) 5) Gmelin, Syst Nr **60**, TlB, Lfg 1 (1958), 35-65 (Cuprous); 65-97 (Cupric); 98-117 (Cupric hydroxide) 6) Ullmann **11**(1960), 243 (Cuprous); 244 (Cupric;) 241(Hydroxide) 7) Cond-ChemDict(1961), 303 (Oxides); 301-02 (Cu hydroxide) 8) Lange (1961), 246-49 9) Anon, " Properties of Materials Used in Pyrotechnic Compositions" **AMCP 706-187(1963),** 109-11

AddnlRefs: A) Davis (1943), 67 (Purple light pyro compn: CuO 6, K perchlorate 9, Sr nitrate 7, sulfur 7 & calomel 3 parts) B) L. Lo Fiego & G.G. Butters, NOLM **9292**(1947), p 2(US Navy Blue Light, Mark 1, Formulas 1 & 2 compns are listed in this section under Copper Ammonium Sulfate and Copper chloride) C) Izzo(1950), 214 [Blue light, slow burning pyro compn: CuO 15.5, K chlorate 46.0, K nitrate 15.5 & sulfur (flower) 23.0%] D) Shidlovskii (1954), 260 [Gasless delay compn: CuO 60 & Zn (pdr) 40% (Swed P 13 0717 of 1951)] E) Ellern (1961) 137, 139 & 282 (Cuprous & cupric oxides were recommended for use in "first fire" formulations, such as Cu_2O (or CuO) 33.33, PbO_2 33.33 & silicon 33.33%); 231 [Addn of 2-3% of Cu_2O (or CuO) to AN prevents its caking]; 275-76 [Green light tracer compn of high intensity: Ba nitrate 45, K perchlorate 16, Mg (pdr) 26, hexachlorobenzene 7, CuO 2 gilsonite 2 & oil 2%] F) J. DeMent, USP 2995 526(1961), p 9 [Smoke producing mixts: a) Cu_2O 20, $KClO_3$ 20, sulfur 20, KBr 20 & $KBrO_3$ 20%; it burned rapidly producing a pale violet-gray smoke; b) Cu_2O 9.0, $KClO_3$ 2.0 to 2.5, KIO_3 1.0 KI 1.0 part; it burned rapidly with a blue flame, releasing a deep violet smoke] G) US Army Specification No **50-55-9,** Signal Drift, M25 [Its components are "igniter composition": CuO, Si & Pb_3O_4 and "pellet composition": ZnO, Mg(pdr), MnO_2, red P & linseed oil] H) A.J. Clear, PATR **1597** (1946), "Effects of Certain Oxides of Iron and Copper on the Properties of TNT" [British findings that pres-

ence of CuO in TNT causes decreases in its ignition temp (to as low as 90°) but increases in its rate of decompn were not entirely confirmed. Mixts of TNT with 10% CuO or ferric oxide did not ignite when stored at 90° for 150 hrs and there was no apparent reaction under these conditions. The mixt of TNT with 10% CuO was less stable, when heated to 243°, than straight TNT. It does not seem that the presence of CuO increases the sensitivity of TNT to impact & friction, but the presence of ferric oxide does increase the sensitivity to impact]

Copper Oxides, Analytical Procedures. *Cupric Oxide* intended for use in US Military pyrotechnic compns is required to have moisture not over 0.2, CuO not less than 95.0 and acid insolubles not over 0.5%. The granulation shall be such that not less than 95% passes thru a No 200 US Std Sieve

a) *Moisture* is detd by heating a 5g sample for 1 hr at 100 to 150° in a tared dish, followed by cooling and weighing

b) *CuO Content* is detd by heating a 1g sample in 50ml of 10% sulfuric acid to boiling, continuing to boil for 10 mins, cooling, filtering thru No 40 Whatman paper and catching the filtrate in a 200-ml tall form beaker. After washing the filter paper with three 25-ml portions of w, and catching the rinsings in the above tall beaker, the liquid is acidifed with 3 ml of 15N nitric acid (freshly boiled to remove N oxides). The filter paper is saved for detn of acid insolubles and the liquid in beaker is electrolyzed at RT with a current of 0.3 amp using a tared platinum gauze cathode and rotating platinum anode. After discoloration of blue liquid,1g of urea is added and electrolysis is continued for addnl 15 mins. To test the completeness of reaction, ca 20 ml of w is added and electrolysis is continued for another 15 mins. If no Cu deposit is observed on upper part of cathode, the beaker is removed and, while the current is still on, the cathode is washed with distilled w and with acetone. Then the cathode is disconnected, dried, and weighed

$$\% \text{ CuO} = (125.18A)/B$$, where:

A = Wt of Cu deposited; B = Wt of sample corrected for moisture

c) *Acid Insoluble Material* is detd by transferring the filter paper with pptd material of previous opn to a tared crucible, igniting the paper to ashes by means of a flame, cooling and weighing the crucible

$$\% \text{ Insolubles} = (100A)/B$$, where;

A = Increase in wt of crucible; B = Wt of original sample (ca 1g)

d) *Granulation* is detd by placing a 100g sample on a No 200 US Std Sieve (to which a bottom pan is attached) and shaking for 5 mins by means of a mechanical shaker geared to produce 300 ± 15 gyrations and 150 ± 10 taps of the striker per min. The wt of material remaining on the sieve, deducted from 100, gives % passed thru the sieve (Ref 1)

Cuprous Oxide intended for use as a chemical reagent shall be carmine red in color and contain not less than 90% Cu_2O. Its maximum allowable impurities are: water solubles 0.500, chlorides (as Cl) 0.500, sulfates (as SO_4) 0.05 and iron (as Fe) 0.10%

a) *Cu_2O Content* is detd by heating a mixt of 0.2 g sample, 40 ml of 0.1 N K permanganate & 50 ml of 10% sulfuric acid to boiling and and titrating excess of permanganate soln with 0.1 N oxalic acid soln

$$\% Cu_2O = [100 \times (40 - A) \times B]/C$$, where:

A = Vol of 0.1N oxalic acid used; B = 0.007156 and C = Wt of sample

b) *Water Solubles* are detd by shaking for 5 mins a 1g sample with 25ml distd w, filtering and evaporating the filtrate in a tared dish. After igniting gently the residue, the dish is cooled and weighed

c) *Chlorides* are detd by dissolving 1g sample in 70% nitric acid, diluting with w and adding an excess of 2% aq $AgNO_3$ soln. After filtering thru a tared Gooch the ensemble is dried and weighed

d) *Sulfates* are detd by shaking a 1g sample for 5 mins in 20ml distd w, filtering and adding to filtrate 4 drops of concd HCl and 3ml of 12% $BaCl_2$ soln. If any turbidity appears within 1 min, the sample does not pass the test

e) *Iron* is detd by dissolving 0.5g sample in 3 ml concd HCl, diluting to 50ml with w and adding an excess of NH_4OH. After heating to boiling and filtering, the ppt is washed, dried, ignited and weighed

$$\% \text{ Fe} = (100 \times A \times B)/C$$, where

A = Wt of residue; B = 0.6992 and C = Wt of sample

Copper Oxychlorides (Called also Basic Copper Chlorides). Compds of general formula, $xCuCl_2 . CuO . zH_2O$. Mellor (Ref 1) lists 18 compds,

some of which are propably mixts. Gmelin (Ref 5) also lists several oxychlorides, among them: $CuCl_2.CuO.H_2O$; $CuCl_2.2CuO.2H_2O$; and $CuCl_2.3CuO.H_2O$. Sax (Ref 4) lists $CuCl_2.2CuO.4H_2O$ as emerald grn to greenish-blk pdr, and gives *Brunswick Green,* as an alternate name. The same compd is listed in Lange (Ref 7). Its mw is 365.60 and it loses $3H_2O$ at 140°. Kirk & Othmer (Ref 3) lists anhyd salt $CuCl_2.3CuO$, brn pdr and its tetrahydrate $CuCl_2.3CuO.4H_2O$, grn pdr which is used in pigments and fungicides. CondChemDict (Ref 6) lists $CuCl_2.3CuO.3½ H_2O$ and states that the compn is variable

Davis (Ref 2), lists the following pyro compns contg "copper oxychloride" : a) Pilot blue light compn: K chlorate 46, Cu oxychloride 32, sulfur 28, calomel 3 & shellac 48(?) parts b) Blue Bengal light compn: K chlorate 5, Cu oxychloride 2 & shellac 1 part c) Lilac light compn: K chlorate 17, Sr carbonate 9, Cu oxychloride 2, Pb chloride 1, sulfur 7 & dextrin 1 part d)Lilac mauve light: K chlorate 17, Sr carbonate 9, Cu oxychloride 4, Pb chloride 1, sulfur 7 & dextrin 1 part

Refs: 1) Mellor **3**(1923), 178-79 2) Davis (1943), 67, 70 & 84 3) Kirk & Othmer **4**(1949), 470 4) Sax (1957), 505 5)Gmelin, Syst Nr **60,** TlB, Lfg 1(1958), 321-23 6) CondChemDict(1961), 303 7) Lange (1961), 248-49

Copper Perchlorates:

Cuprous Perchlorate, $CuClO_4$, wh pdr. Was prepd by dropping a spiral of bright copper wire into an ethereal soln of chlorine tetraoxide in a test tube and removing the white coating from the wire. Its expl props were not investigated (Refs 2 & 5)

Cupric Perchlorates. No anhyd salt was prepd, but its hepta-, hexa-, tetra- and di-hydrates are known (Ref 5). The most common seems to be *hexa-hydrate,* $Cu(ClO_4).6H_2O$. It was first prepd in 1831 by Sérullas by heating CuO with perchloric acid. Portillo & Alberola prepd it in 1930 (Ref 3) by treating Cu carbonate with 30% perchloric acid. The ppt, washed with alc & ether and dried in vacuum over P_2O_5, was lt blue in color, mp 82° and decomposed at 120° (See also Refs 4 & 5)

According to Ellern (Ref 6), a match can be produced which uses Cu (or Sr) perchlorate dissolved in triethanolamine and combined with cellulose acetate - with the regular matchheads affixed

Refs: 1) Mellor **3,** (1923) - not found 2) M. Gomberg, JACS **45,** 413(1923) 3) R. Portillo & L. Alberola, AnalesRealSocEspañFisQuím (Madrid) **28,** 1117-24 (1930) 4) Sax(1957), 505 5) Gmelin Syst Nr **60,** TlB, Lfg 1(1958), 337-38 6) Ellern (1961), 101

Copper Phthalocyanine, $(C_6H_4C_2N)_4N_4Cu$; solid d 1.59. A blue pigment deriv of org pigments having as a structural unit four isoindole groups, $(C_6H_4)C_2N$, linked by four N atoms so as to form a conjugated chain. It is extremely stable to light, acids, alkalies and heat. Can be prepd by reaction of phthalic anhydride, urea & cuprous chloride at ca 200° or by reaction of o-chlorocyanobenzene with cuprous cyanide & Cu pdr. Has been used in enamels, linoleum, plastics, rubber goods, etc. (Refs 2 & 3).The uses of phthalocyanines in proplnts are discussed in Ref 4

Refs: 1)Beil - not found under Phthalcyanin or under $C_{32}H_{16}N_8Cu$ 2) Hackh's (1944), 654 & 674 3) CondChemDict (1961), 303 & 892-93 4) B.J. Konrot, PATR **2040** (1954)(Conf) (Not used as a source of info)

Copper Picramates. See Vol 1, p A242-L under Amino-phenols and Derivatives

Copper Picrate will be described as Phenol, Trinitro, Copper Salt

Copper (II) Resinate or Cupric Resinate, $Cu(C_{20}H_{29}O_2)_2$; mw 666.43, grn pdr; insol in w, sol in ether & oils. Can be prepd by heating Cu sulfate with rosin oil, followed by filtering and drying the ppt. Has been used as a preservative paint, particularly for ship's bottoms, and as an insecticide (Refs 3 & 4). Its uses in pyrotechnics are discussed in Addnl Refs A, B & C

Refs: 1) Beil - not found 2) Hackh's(1944), 242 (not found) 3) Sax(1957), 506 4) CondChemDict (1961), 304

Addnl Refs: A) G.J. Schladt, PATR **357** (1933) [Blue light pyrotechnic compns developed at PicArsn: a) K perchlorate 65, Pb phosphate 25 & Cu resinate 10% b) K perchlorate 65, Cu chromate 25 & Cu resinate 10%] (See also under Copper Acetoarsenite) B) G.J. Schladt, PATR **634**(1935) (Cupric resinate as substitute for shellac, a strategic material, in pyrotechnic compns) C) B. Sukornick et al, PATR **2142** (1955) (Conf) (NOT used as a source of info)

Copper Resorcinate, Trinitro or Copper Styphnate will be described as Resorcinol, Trinitro, Copper Salt

Copper Rhodanides. See Copper Thiocyanates

Copper (II) Salicynate or Cupric Salicylate, $Cu(C_7H_5O_3)_2.4H_2O$; mw 409.8, blue-grn ndls, mp decomp ca 110° forming brn *basic salt.* Was first prepd, in 1855 by Piria, by treating Cu sulfate with Ba salicylate (Refs 1 & 4). Pickering (Ref 2) prepd it by treating a soln of K salicylate with Cu sulfate. Uses of cupric salicylate and of the basic salt in US proplnts are discussed in conf Ref 5. Toxicity is discussed in Ref 3
Refs: 1) Beil **10,** 59, (25) & [33] 2) S.A. Pickering, JCS **101,** 180(1912) 3) Sax(1957), 517 4) Gmelin, Syst Nr **60,** TlB, Lfg 2(1961), 800 5) Propellant Manual **SPIA/M2** (1962) (Conf), Unit Nos 390, 391, 550, 552 & 560

Copper (I) - sodium Fulminate. See under Copper (I) Fulminate Explosive.Complexes

Copper (II) Stearate or Cupric Stearate, $Cu(C_{18}H_{35}O_2)_2$; mw 630.50, pale bulu-grn pdr (from xylene), mp 125°. Can be prepd by double decompn of an alkali stearate and cupric acetate in aq-alc soln (Refs 1, 2, 3 & 4). DeMent (Ref 5) patented its use in a smoke producing pyrotechnic compn consisting of Cu stearate 5,0, K chlorate & "halogen donor", such as fluoride, chloride, etc.
Refs: 1) Beil **2,** 379, (172), [350] & {1007} 2) W.F. Whitmore & M. Lauro, IEC **22,** 647-48(1930) 3) Sax (1957), 507 4) CondChemDict (1961), 304 5) J. DeMent, USP 2995526(1961), p 9 6) Gmelin Syst Nr **60**, TlB, Lfg 2 (1961) - not found

Copper (I) - strontium Fulminate. See under Copper (I) Fulminate Explosive Complexes

Copper (II) Sulfate Ammoniated; Copper (II) ammino Sulfate or Cupric Ammonia Sulfate, $CuSO_4$-$4NH_3.H_2O$; mw 245.8, dk blue cryst pdr. This compd,known since 1693 (Ref 3), can be prepd by dissolving cupric sulfate in Amm hydroxide and precipating with alc. Used in manuf of Cu arsenate, insecticides & treating fiber products
Refs: 1) Mellor **3**(1923), 253 (Monohydrated cupric tetrammino sulfate 2) Sax (1957), 502 (Called Copper ammonium sulfate) 3) Gmelin, Syst Nr **58**, TlB, Lfg 1, p 563 [Kupfer (II)-Amminosulfat] 4) CondChemDict (1961), 405

Copper (II) Sulfate, Ammonium. See Copper (II) - ammonium Sulfate

Copper Sulfates:
Cuprous Sulfate, Cu_2SO_4, greyish-wh pdr, stable in dry air but decomp by moisture. Can be prepd by heating powdered Cu_2O with methyl sulfate at 160° until methyl ether ceases to be evolved (Ref 1, p 232; Ref 5, 487-89)
Cupric Sulfate Anhydrous, $CuSO_4$, mw 159.61; grn-white crysts, mp-decomp > 600°, d 3.606 at 15°. Can be prepd by heating Cu with coned sulfuric acid, in a closed vessel or by other methods. Used as a dehydrating agent (Ref 1, pp 235-36; Ref 5, 491)
Cupric Sulfate Pentahydrate, Blue Stone or Blue Vitriol, $CuSO_4.5H_2O$; mw 249.69, blue crysts, mp - loses $4H_2O$ at 110° and the fifth H_2O at > 250°; d 2.286 at 15.6/4°; sol in w; sl sol in alc; dissolves slowly in glycerin. Can be prepd by dissolving Cu oxide (hydroxide or carbonate) in hot dil sulfuric acid and evaporating the liquid for crystn.It is the most important Cu salt. It is used in several industries (such as textile, leather, steel, petroleum, ore flotation, synthetic rubber), analytical chemistry, electric batteries, insecticides, etc (Ref 1, p 235; Ref 2, p 357; Ref 3, p 474; Ref 4, p 507; Ref 5, p 508; Refs 6, 7 & 8). Its uses as an ingredient of industrial expls are discussed in Addnl Refs A, B, C & D, and as an ingredient of pyrotechnic compsn in Addnl Refs E, F & G

Powdered $CuSO_4.5H_2O$ loses part of the w of crystn when kept in a desiccator over P_2O_5 forming the trihydrate, $CuSO_4.3H_2O$ and, if kept in a vacuum over P_2O_5, the *monohydrate,* $CuSO_4.H_2O$ (Refs 1, 2, 3, 5 & 6)
Cupric Sulfates, Basic. Several salts of general formula $CuSO_4.xCuO.yH_2O$ are listed in Ref 1, p 261 and in Ref 5, pp 579-89. Kirk & Othmer (Ref 3, p 477-78), describe the compd $CuSO_4.7CuO.12H_2O$, mw 932.81,grn amorphous pdr which turns blue on drying; mp - loses six H_2O at 149°and the remainder at 260°. Can be prepd by treating an aq soln of Cu sulfate with Ca hydroxide. Used as a fungicide (Ref 3). Its uses in pyrotechnic compsn are discussed by Davis (AddnlRef D, pp 70 & 84)
Refs: 1) Mellor **3**(1923), 231-67 2) Thorpe **3**

(1939), 356-58 3) Kirk & Othmer **4**(1949), 474-78 4) Sax (1957), 507 5) Gmelin Syst Nr **60,** TlB, Lfg 1(1958), 487-526 & 579-89 6) Ullmann **11**(1960), 246-55 7) CondChemDict (1961), 304-05 8) Lange (1961), 248-49
Addnl Refs: A) R.L. Hill & A.J. Strane, USP 1299942(1919) & CA **13,** 1930(1919) (Anhyd cupric sulfate in small amts to prevent undue "setting" or hardening of AN expls in storage. Eg: AN 26, Na nitrate 35, NG 21, woodmeal 14, $CuSO_4$ 3 & Chalk 1%) B) Ibid, USP 1307495 (1919) & CA **13,** 2281 (1919) (Use of anhyd Cu sulfate as in previous item. Eg: AN 88, TNT 10 & $CuSO_4$ 2%) C) Ibid, CanadP 193310(1919) & CA **14,** 124-25 (1920) (Addn of 0.5 to 10% of cupric sulfate, from which H_2O of crystn has been wholly or partially removed, to AN expls prevents their hardening unless frozen) D) Davis (1943), 60 (Ruggieri's Russian Fire - see under Copper Acetate); 70 (Lilac light Bengal fire: Basic Cu sulfate 6, K chlorate 26, Sr sulfate 10, Pb nitrate 5, sulfur 4, shellac 1 & stearine 1 part); 70(Violet light Bengal fire: Basic Cu sulfate 1,K chlorate 25, Sr sulfate 20 & sulfur 20 parts); 84 (Violet light star: Basic Cu sulfate 8, K chlorate 56, Sr sulfate 16, Pb chloride 3, poplar charcoal dust 3, sulfur 20 & dextrin 3 parts); 84 (Violet light star: Basic Cu sulfate 12, K chlorate 24, Pb chloride 2, sulfur 8 & dextrin 1 part); 353 (Use of Cu sulfate pentahydrate as a cooling agent in permissible expls, because it gives off water on heating, thus cooling the gases of expln); 384 (Use of Cu sulfate in test for Nitroguanidine) E) Weingart (1947), 7 (Use of cupric sulfate in pyrotechnic compns contg chlorates is not recommended) F) Izzo (1950), 214 (Blue light pyrotechnic compn: K chlorate 58, cupric sulfate 17, calomel 11, shellac 11 and dextrin & gum arabic 3%) G) Ellern (1961), 99 (Considers blue vitriol as an ineffective addn to pyrotechnic mixts) H) US Federal Specification **O-C-828A** (Copper Sulfate Pentahydrate, Technical)

Copper Sulfides:
Cuprous Sulfide, Cu_2S, mw 159.20, blk solid, mp ca 1100°; occurs as the mineral *chalcocite* (copper glance). Can be prepd by heating CuS in a stream of hydrogen or by other methods (Refs 1, 2, 4 & 7). Used in protective paint for vessels (Ref 9, p 321
Cupric Sulfide, CuS, mw 95.63 blk solid mp 1100°, decomp ca 2200°, d 4.6; insol in w, sol in nitric acid; occurs as the mineral *covellite.* Can be prepd by passing hydrogen sulfide into a soln of cupric salt. This gives pptd CuS. Accdg to Weingart (Ref 3), only fused CuS is suitable for use in pyrotechnic compns, producing blue and purple lights in presence of calomel. Fused CuS is prepd by heating Cu sheets with calcd amt of powdered sulfur in a closed clay crucible. Details of procedure are given in Ref 3, p 8. Izzo (Ref 5) gives a purple light compn contg fused, and then pulverized CuS. Other uses of CuS are given in Refs 4, 8 & 11. They are prepn of antifouling paints, of semiconductors and of aniline black
Refs: 1) Mellor **3** (1923), 210 & 220 2) Thorpe **3** (1939), 355 3) Weingart (1947), 7-8 4) Kirk & Othmer **4** (1949), 477-78 5) Izzo (1950), 215 [Purple light pyrotechnic compn: CuS (fused and then pulverized) 20, K perchlorate 30, Sr nitrate 23, calomel 10 & sulfur 17%) 6) Sax (1957), 507 & 509 7) Gmelin, Syst Nr **60,** TlB, Lfg 1(1958), 432 & 470 8) Ullmann **11**(1960), 255-56 9) CondChemDict (1961), 305 & 321

Copper Sulfocyanates. See Copper Thiocyanates

Copper (II) Tetraquoethylenediamine Perchlorate. See Copper (II) mono(ethylenedianine) - Tetraquo Perchlorate

Copper Tetrazolate will be described under Tetrazole and Derivatives

Copper Thiocyanates, Copper Sulfocyanates or Copper Rhodanides (Kupfer Rhodanide in Ger): *Cuprous Thiocyanate,* CuSCN, mw 121.65, white or yellowish pdr, mp 130° with decompn; stable in light; d 2.85; nearly insol in w; sol in NH_4OH or KSCN. When ignited in air, it burns with a blue flame. Was first prepd in 1854 by Rivot. Can be prepd by adding aq NH_4SCN to a soln of $CuSO_4$ slightly acidified with SO_2 gas (Ref 1). Prepn of very pure salt is described by Demmerle (Ref 5). It has been used in galvanotechnique (Ref 9), in antifouling paints (Ref 10) and as an ingredient of non-fulminate primer compns, in which it functions as a combustible and as an antacid. By decomposing into CuO it also provides a solid vehicle for the transfer of heat. Typical primer compns contg CuSCN are listed in Refs 3 & 11. CuSCN was formerly used in some US Army

primers and requirements and tests for that material are covered by US Army Specification **No 50-11-22A**

Cupric Thiocyanate, $Cu(SCN)_2$, mw 179.73; dk brn or blk pdr, rather unstable; mp - decomp. Can be prepd by treating freshly pptd $Cu(OH)_2$ with thyocyanic acid or by other method. In presence of ammonia, it forms ammoniacates (ammines), which are more stable than $Cu(SCN)_2$ (Refs 1, 2 & 9). No info at our disposal about its uses

Refs: 1) Beil **3,** 153, (67), [114]& {264}2) T.L. Davis & A.V. Logan, JACS **58** 2155 (1936) [Prepn of pure $Cu(SCN)_2$] 3) Davis (1943), 456-57 [Primer compositions: a) K chlorate 67, CuSCN 15, sulfur 16 & charcoal 2% b) K chlorate 60, Sb trisulfide 30, CuSCN 3 & sulfur 7%] 4) Kirk & Othmer **4** (1949), 741 5) R.L. Demmerle, IEC **42,** 2(1950) (Prepn of very pure CuSCN) 6) Sax (1957), 508 (Cuprous thiocyanate) 9) Ullmann **11** (1960), 244-46 10) CondChemDict (1961), 321 (Cuprous thiocyanate) 11)Ellern (1961), 67 [Electric primer mixt: Cu(or Pb) thiocyanate 45 & K chlorate 55%]; 272-73 (Older type percussion primer mixt: K chlorate 41.5, Sb trisulfide 9.5 CuSCN 4.7 & ground glass 44.3%) 12) Gmelin Syst Nr **60,** TlB, Lfg 2(1961), 868 & 874

Copper (II) - 2,4,6-trinitrobenzoate, $Cu(C_7H_2N_3O_8)_2.2H_2O$; blue crysts, mp-loses $2H_2O$ becoming *anhydrous;* wh, hygr pdr explg on heating or on impact. Can be prepd by treating as aq soln of 2,4,6-Trinitrobenzoic Acid with freshly precipitated $Cu(OH)_2$ or by other methods

Refs: 1) Beil **9,** (168) & [285] 2) F. Ephraim & E. Rosenberg, Ber **51,** 659 (1918) 3) C. Krauz & O. Turek, SS **20,** 54 (1925) & Chim & Ind (Paris), Spec Number **1926,** 528C 4) Gmelin, Syst Nr **60,,** TlB, Lfg 2(1961), 814

Copper (II) - 2,4,6-trinitrophenolate. See under Phenol, Trinitro

Copper (II)tris(ethylenediamine) Bromate. $[Cu(en)_3](BrO_3)_2.H_2O$; blue prisms, mp - expl on heating. Can be prepd by warming 75% ethylenediamine soln with $Cu(BrO_3)_2$, similarly to prepn of chlorate

Refs: 1) Beil **4,** {485} 2) J. Amiel, CR **200** 672 (1935) 3) Gmelin, Syst Nr **60** , TlB, Lfg 1 (1958), 387 (Not found)

Copper (II) tris(ethylenediamine) Chlorate, $[Cu(en)_3](ClO_3)_2.H_2O$ blue delq prisms, mp - expl 178-85°; detonates violently on impact or on contact with concd sulfuric acid. Was obtd by warming 75% aq soln of ethylenediamine (3 moles) with $Cu(ClO_3)_2.6H_2O$ (1 mole), followed by crystn above 20°

Refs: 1) Beil **4,** {485} 2) J. Amiel, CR **199,** 202(1934) 3) Gmelin, Syst Nr **60,** TlB, Lfg 1 (1958), 335

Copper (II)tris(ethylenediamine) Nitrate, $[Cu(en)_3](NO_3)_2.H_2O$; blue delq crysts, mp-expl mildly ca 292°; when left in a desiccator under reduced pressure it transforms to $[Cu(en)_2](NO_3)_2$. Was prepd by heating a 75% aq soln of ethylenediamine (3 moles) with $Cu(NO_3)_2.6H_2O$ (1 mole) followed by crystn

Refs: 1) Beil **4,** {485} 2) J. Amiel, CR **199,** 202 (1934) 3) Gmelin, Syst Nr **60,** TlB, Lfg 1 (1958), 183

Copper (II)tris(ethylenediamine) Perchlorate, $[Cu(en)_3](ClO_4)_2.H_2O$; small dk blue crysts, mp-expl ca 290°, detonates on impact. Was prepd by treating a 75% aq soln of ethylenediamine (3 moles) with $Cu(ClO_4)_2.6H_2O$ (1 mole), followed by crystn. Only a small quantity can be prepd at a time because the reaction might proceed with explosive violence

Refs: 1) Beil **4,** {485} 2) J. Amiel, CR **199,** 201(1934) & **200,** 672(1935) 3) Gmelin, Syst Nr **60,** TlB, Lfg 1(1958), 342

Copper Crusher Gages. See under Closed Bomb (Vessel) Technique

Copper Cylinder Compression Test, known as *Brisance Meter Test of Kast* (Stauchprobe nach Kast in Ger; Essai de brisance à l'appareil de Kast in Fr). It is briefly described in Vol 2, p 299-L under Brisance Test Methods and also in this Vol under Compression (or Crusher) Tests for Determination of Brisance

Copper Index or Copper Number of Cellulose. See Copper Value of Cellulose

Copper Value (Copper Index or Copper Number) **of Cellulose.** It represents the amt of Cu reduced from the cupric to cuprous state in alkaline soln by 100g of cellulose (such as cotton). In case of cotton, it gives an indication as to whether any appreciable changes have taken place during purification procedures; in case of woodpulp, it indicates the degree of purity. Accdg to Dorée (Ref 4 pp 26 & 32-3), normal cotton cellulose has copper values below 0.2 while sulfite pulp has value>2. The alpha-cellulose obtd from sulfite pulp had a value of 0.8

The procedure originated by E.G. Schwalbe in 1907 is described in Ref 2, p 691 & Ref 4, p 27. The method was modified by Knecht & Thompson, by Braidy & Hägglund. These methods are described in Ref 4, pp 29-36

Marshall (Ref 3) stated that Braidy's modification gives more consistent results than the original Schwalbe's method

Procedure: Place in a flask 95 ml of the soln (prepd by dissolving in 1 liter of distd w 350 g of $Na_2CO_3 . 10H_2O$ & 50g of $NaHCO_3$) and add to it (from a burette) 5 ml of 10% sol of $CuSO_4 . 5H_2O$. Bring the mixt to a boil and pour it immediately over 2.5 g of air dry cellulose, placed in a 125 ml Erlen flask. After eliminating (by means of a glass rod) the air bubbles from cellulose, close the flask (by means of a glass bulb) and immerse it up to the neck in a water bath, the top of which is covered to prevent cooling by draughts. After heating for exactly 3 hrs, filter the mixt under suction, and wash the cellulose (impregnated with Cu_2O) on the filter, first with dil soda soln and then with hot w, until free from sol Cu salts. Remove Cu_2O by treating the cellulose with 15ml and then 10ml of the soln (prepd by dissolving 100g of ferric alum in mixt of 100g concd H_2SO_4 & 140 ml w and bringing the vol with w to 1 liter), and wash the cellulose with 2N-sulfuric acid. Titrate the combined filtrates and washings with N/25 K permanganate soln until discoloration. One ml of permanganate soln is equivalent to 2.5 mg of reduced Cu

Coppering is metal fouling accumulated in the bore of a weapon due to repeated firing. The metal (mostly Cu or its alloys) is deposited from the rotating bands or jackets of the projectiles

Ref: OrdTechTerm(1962), 84

Corajo, Ivory Nut or Vegetable Ivory. A hard white substance obtd from the tagud nut which is the fruit of the negrito palm (Phytelephas Macrocarpa), or from some other palms known in South America. It consists of cellulosic bodies & other carbohydrates, including mannan. Vegetable ivory has been used in pulverized form as a combustible ingredient, in place of woodmeal, in some blasting expls. For example, F. Olsen patented in 1924 a blasting expl contg 3-10% vegetable ivory plus other ingredients (See Ref 32, p B204-R in Vol 2 of this Encycl)

Refs: 1) Marshall **3** (1932), 230 2) Hackh's (1944), 226, 656 & 828 3) Merriam-Webster's (1961), 510, 1204 & 2537

Corbette-type Magazine. This is a beehive or dome type magazine having the front & rear, as well as the sides, covered with earth as an extra precaution in the event of an explosion. The magazine is provided with ventilators and is protected against lightning by an efficient lightning protection system. Reinforced concrete is used as the material of construction for the magazine. This is the preferred type of magazine for the storage of all kinds of explosives and all items of ammunition. Solid propInts & pyrotechnics are also preferably stored in this magazine because of better temp control, but they may be stored also in above ground type magazines

Refs: 1) Anon, TM9-1903, TO11A-1-37 (1956), 29 2) Anon, AMC Safety Manual AMCR 385-224 (1964), 17-26 & 18-1

Cord, Detonating; Cordeau, Cordeau Fuse or Detonating Fuse (Called Cordeau détonant in Fr; Detoniruyoushchii shnur in Russ; "Bonocord", trade name in Sweden; and Detonierende Zündschnur or Knallzündschnur in Germany & Switzerland). This is essentially a small round core of expl that is covered with a protective sheath consisting of various combinations of textiles, waterproofing material, wire & plastic (Ref. 19). The function of the sheath is to minimize damage from abrasion & other types of physical abuse, and to minimize the chance of water

penetration. The purpose of Detoning Cord is not to transmit flame to an expl, as Safety Fuse does, but to initiate charges of HE Explosion of the core of Detonating Cord is accomplished usually by a Blasting Cap (See Vol 2, p B185-L). Many different types & sizes of Detonating Cord are manufd for special purposes, but the kinds commonly used in commercial blasting operations are primarily described here (Refs 10 & 17)

An early French Detonating Cord (called Cordeau détonant, modèle 1902) consisted of a tin tube filled with a PA core. The external diam of tube was 5.4 mm and the vel of deton of the cord was 6690-6745 m/sec. About 1907 L. Lheure of France introduced commercial Cordeau. It consisted of a lead tube filled with TNT or Tetryl. Its vel of deton was 5110 m/sec. This type of Cordeau was introduced in US by 1913 by the Ensign-Bickford Co, Simsbury, Conn (Refs 1, 3,5,7a,10 & 14). See also *Cordeau Bickford* under Bickford Fuse or Miner's Safety Fuse, Vol 2, p B112

Snelling & Koch (Ref 2) patented a Cordeau in which molten TNT was poured into a lead tube. After the tube cooled, it was subjected over its entire circumference to the action of rapidly moving hammers, whereby its length was increased and its diam decreased. By this method the TNT was reduced to a finer state of division and consequently became more sensitive to initiation. Such a Cordeau is reported to be smoother & stronger than when the Pb tube is drawn or rolled

British Detonating Cord is described in Ref 18. This Pamphlet, which describes some British Detonating Cords, was not available in our Library

A Hexanitromannite Cordeau, proposed by Sébert & Fritsch to the Poudrerie à Sévran-Livry, developed a vel of deton of 7000 m/sec, but was considered too expensive & too sensitive for coml use (Refs 3 & 5). More recently the SA D'Explosifs et de Produits Chimiques manufd a Cordeau consisting of flat hollow Pb bands filled with Tetryl. It is claimed that flat Cordeau is easier to introduce into a bore-hole than round Cordeau. Other models of Fr Detonating Cords employing a PETN core & a fabric cover are called: *Cortex*, OD 4.8mm, gms of expl/m 8 & vel of deton 5990-6410 m/sec and *Éclair*, OD 5.5 mm, gms of expl/m 11 & vel of deton 6355-6745 m/sec. *Primacord* (American) had OD 5.2 mm, grns of expl/m 10 & vel of deton 6290-6450 m/sec and *Cordtex* (British), OD 5.0 mm, gms of expl/m 10 & vel of deton 6715-6825 m/sec (Refs 10 & 13). See also Refs 8a, 11, 12, 15, 16 & 17a

Majrich (Ref 6) reported the manuf & props of four German coml Detonating Fuses: a) TNT core in a Pb case; vel of deton 4600-5500 m/sec b) PA in a tin case; vel of deton 6600-6700 m/sec c) MF deflegmatized with paraffin in a yarn wrapping; vel of deton 5250 m/sec and d) PETN in a cellophane wrapping, coated with guta-percha; vel of deton 6300-7300 m/sec. The Number 3 Cord is initiated by the weakest of detonators (No 1), while the others require a No 8 detonator. Other advantages of the number 3 Cord are that it is not affected by exposure to temps of -180 to 60 °C nor by immersion in water. Majrich (Ref 6) also tested a Cord contg Triazidotrinitrobenzene (See Vol 2, p B43-R) desensitized with paraffin. It has a vel of deton of 7780-7830 m/sec, and burns slowly (15.5 cm/sec) when merely ingited

The compns of some Russian Detonating Cords are as follows (Ref 7): 1) MF 75-80% & paraffin 25-20% 2) MF 28, Tetryl 71.5 & gelatin (binder & phlegmatizer) 0.5%. These cords have a deton vel of 5000 m/sec at diam of 5.5 mm. They are covered with three layers of fabric 3) MF 73 & Tetryl 27%, with gelatin 1.5% added This compn is impregnated on two layers of cotton thread. The fuse does not req a blasting cap 4) RDX core, having a vel of deton of 7600 m/sec 5) RDX & Tetryl core, having a vel of deton of 6500 m/sec 6) Grained PETN core enclosed in a cotton cloth tube and 7) PETN 90-97 & Pb nitrate 10-3%, to which 0.1-0.2% red lead oxide (Pb_3O_4) is added

Swedish Detonating Cord, called *Bonocord*, is described in Vol 2, p B218 under Bofors Detonating Cord or Bonocord

A general description of Swiss Detonating Fuses is given by Stettbacher (Refs 4 & 9). The Société Suisse des Explosifs, Gamsen-Brigue (Ref 8) patented a Detonating Fuse composed of a core of PETN or RDX in a gel of CC & a liq expl such as NG, NGc or Tetranitroisobutylglycerin. This compn is coated on several textile threads and covered with a fabric coating instead of lead. The deton vel was reported in private communication from the late Dr. Stettbacher to be 7000 m/sec

(See also under Detonating Fuse)

Refs: 1) Van Gelder & Schlater (1927), 728 2) W.O. Snelling & C.B. Koch, USP 1741380 (1929) & CA **24**, 1221 (1930) 3) Vennin, Burlot & Lécorché (1932), 570-72 4) Stettbacher (1933), 358-59 5) Pepin Lehalleur (1935), 371-73 6) A. Majrich, Chem Ztg **60**, 333-35 (1936) & CA **30**, 4672 (1936) 7) B.B. Rimkevitch, Chem Ztr **1942 II**, 2760 & CA **38**, 2821 (1944) 7a) Davis (1943), 11 & 164 8) SSEGB, Swiss P 230077 (1944) & CA **43** 2438 (1949) 8a) All & En-Expls (1946), 135 9) Stettbacher (1948), 107 10) M. Dutour, MAF **24**, 577-84 (1950) & CA **45**, 8772 (1951) Uniformity of velocities of detonation of detonating fuses) 11) A. Le Roux, MP **32**, 99-105 (1950)(Influence of conditions of manuf on the props of pliable PETN Detonating Cord) 12) M. Ortigues & G. Ziebelen, MP **32**, 107-16 (1950) (Quality of PETN intended for the manuf of Detonating Cord) 13) L. Le Roux, MP **32**, 201-04 (1950) (Characteristics of Cordex, Primacord, Cortex & Éclair Detonating Cord) 14) Berliner, Expls (1953), 37 15) J. Tranchant, MP **37**, 121-26 (1955) (Recrystallization of PETN for use in Detonating Cord) 16) DAG, Brit P 742300 (1955) (Improvements relating to a firedamp-proof Detonating Cord) 17) Blasters' Hdb (1958) 110 17a) McAdam & Westwater. (1958), 17, 113, 141 & 157 18) ICI, "Plastic Igniter Cord" Pamphlet, 2nd edit (1961) 19) Ord Tech Term (1962), 84

Corde à feu or Mèche à canon (Fr.) A device used in Europe before the introduction of fulminate primers for igniting BkPdr chges. It consisted of flax or hemp fibers, impregnated with concd solns of substances(such as Pb acetate or K dichromate) which would impart, after drying, increased combustibility to the fibers
Refs: Daniel (1902), 156

Cordeau. One of the American names for Detonating Cord, derived from the French term "Cordeau détonant." See Cord, Detonating

Cordeau Bickford. See under Bickford Fuse or Miner's Safety Fuse, Vol 2, p B112 of this Encyl.

Cordeau Combustible. An expl device, patented in 1878 by A. Quentin, consisted of a long paper tube impregnated with rubber and filled with a paste composed of *pulvérin* (finely powdered Bk Pdr), NG & glycerin
Ref: Daniel (1902), 156

Cordeau d'amorce (Fr.). Quickmatch

Cordeau détonant. French term for Detonating Cord or Detonating Fuse. See Cord, Detonating

Cordeau Fuse. See Cord, Detonating

Cordeau Maissin. Maissin, in 1888, patented in France the use of granulated NC as a core of flexible detonating cord (cordeau détonant souple, in Fr),which was prepd in the same manner as the safety fuse of Bickford (See Vol 2, p B112-L of this Encycl). This cord was examined by CSE (Commission des Substances Explosives) and found to be satisfactory from the point of view of performance (Ref 2) (See also Refs 3 & 5)

As manuf of this cordeau was a rather difficult opn, attempts were made to prep it by the method of filling with NC a lead tube of 12 mm ID and then drawing out the tube to 4 mm ID. The resulting cordeau was not satisfactory because fibrous material, like NC, is very difficult to distribute uniformly thruout the tube and to obtn high density of loading

It was decided then to prep the cordeau employing nitrohydrocellulose, which can be obtd in powdery form. Prepn of this material, first obtd in 1875 by Girard, was described by Vieille (Ref1). As lead is easily attacked by PA and some other expls, it was replaced by tin. The tin cordeau listed in Ref 4, had ID of tube 4mm, density of nitrohydrocellulose core 1.47 and detonation rate 4000 m/sec. See also Cord, Detonating
Refs: 1) P. Vieille, MP **2**, 21-35 (1884-1889) 2) CSE, MP **2**, 609 (Étude 45) 3) Daniel (1902), 417 4) Vennin Burlot & Lécorché (1932), 570-71 5) Pepin Lehalleur (1935), 372

Cordeau, Primacord. See under Cord, Detonating

CORDITE. This name is derived from the cord-

like form of a double-base propellant which is, strictly speaking, a modification of early *Ballistite* (See Vol 1, p B8). Cordite differs from Ballistite in that it is made from NC of High N content (Guncotton, insoluble in eth-alc but sol in acetone), and mineral jelly, incorporated with NG (Refs 4,5,7,8,11,12, 13,13a,16 & 19)

Historical. **Cordite Mark 1** (CSP_1 - Cordite Smokeless Powder) is a British proplnt developed in 1888 in Abel's laboratory by Dr. W. Kellner who later succeded Abel as British War Dept chemist. The proplnt was patented in 1889 for the British Government by Abel & Dewar (Ref 1). The original compn consisted of Guncotton (NC of 13.1% N) 37, NG 58 & vaseline, called by the British MJ (mineral jelly) 5%. Its prepn is described further in this section. The mineral jelly was added with the idea that it would lubricate the gun barrel and reduce its erosion. Actually MJ did not lubricate the barrel because it was consumed during combustion of the proplnt. However, the presence of MJ proved to be advantageous in other ways. It did lower the temp of combustion of the proplnt, in comparison with that of the original Ballistite (qv),and thereby reduced some of the erosion of the gun barrel; and it did improve the stability of the proplnt. But the British found that the use of Cordite MK 1, during the South African War (1899-1902), caused such excessive erosion in their guns, that it became necessary to modify its compn in order to further reduce the combustion temp (Refs 4,5, 19,26,30 & 33). See Table on p C 534

Cordite MD or MC (Cordite Modified) is the name given to the modified compn which consisted of NC (13.1%N) 65, NG 30 & MJ 5%. This proplnt was obtd by increasing the NC content & reducing the NG of Cordite Mark 1. It was first manufd in 1901 (Ref 4, p 54) by a process essentially the same as that used for the original Cordite, except that more acetone was needed to incorporate the NC (Refs 2,5,16, 21,26 & 33). See Table on p C 534

Cordite RDB (Cordite Research Department B).Until WWI Cordite MD was used almost exclusively as a std proplnt, although its manuf reqd the use of dry Guncotton which is very dangerous to handle in the dry state. Also it reqd the use of acetone, the supply of which was inadequate during WWI. It became necessary, therefore, to develop Cordite compns ballistically equivalent to Cordite MD, but nor requiring a critical material. This was accomplished by substituting NC of 12.2% N, and using eth-alc as a solv, in place of the dry Guncotton & acetone. The compns developed during WWI as Cordite RDB were: a)NC(12,2%N) 52, NG 42 & MJ 6% (Ref 9) b) NC (12.2%) N 45.5, NG 48.5 & MJ 6% (Ref 8 & 12). See also Ref 6. Cordite RDB was employed by the British Land Services during WWI and used until 1934. See Table on p C534

Cordite SC (Cordite Solventless, Carbamite) The use of a solventless Cordite was suggested at a conference at Watham Abbey in 1914. The composition proposed consisted essentially of NG & soluble NC. To facilitate gelatinization, which was effected by heating & rolling without the use of a vol solv, urethane derivs & benzylphenylether (qv) were added. These compds were found to exert also a stabilizing action. To avoid the risk associated with extrusion thru dies, the proplnt was at first made in flaked form. The gelatinizer finally adopted was benzylphenyl-urethane (Ref 30)

In 1919 a manufg process was begun for a compn known as *Ardeer Cordite*, consisting of NC (12.2% N) 50, NG 42 & benzylphenylurethane 8%. It was prepd in cord form by an extrusion process. In gun firing tests, Ardeer Cordite gave satisfactory results, but in stability tests it did not attain the reqd standards (Ref 30). By means of various stability tests, it was finally established that one of the most efficient compds for use as a stabilizer & gelatinizer in a solventless proplnt was symdiethyldiphenylurea. This compd is called by the British Carbamite and is known in US as Ethyl Centralite (See Vol 2, p C127-L). The compn adopted by the British Navy consisted of NC (12.2% N) 49.5, NG 41.5 & Carbamite 9%

Its manuf at the Royal Cordite Factory began in 1923. See Table on p C 534

Later Developments. The Cooled SC Cordite represents an exptl compn which was developed with a view to reducing erosion. Large scale firing tests were conducted in a number of guns, but due to a very limited reduction of erosion & the larger bulk of chge reqd as a result of its low energy value (See table), the compn was not adopted as a Service proplnt

Flashless Cordites. Prior to WWI much work had been done in many countries in the attempt to eliminate the bright flashes which occur at

the muzzle of a gun. The main directions in which investigations proceeded were: a) inclusion of flash inhibitors in proplnt compns and b) modification of compns so as to give a lower content of the flammable gases CO & H_2 in the combustion products. It has been found as a result of numerous investigations that salts of alkali metals provide the most active agents for inhibiting flash. In tests conducted at Woolwich, it was found that the well-known inhibitors of ignition, anti-knock agents & fire extinguishing materials (such as CCl_4, ethylene dibromide & Pb tetraethyl) were not effective in suppressing muzzle flash. However, Na & K croylite (Na_3AlF_6 & K_3AlF_6), possibly due to their volatilizing temps, were found to be the most efficient compds so far investigated for use in proplnts (Ref 30). But it is possible by the use of these additives to effect flashlessness in guns of small caliber, and no reduction of flash has been achieved by this method alone in guns larger than the British 18 pounder(4.5", diam)

With a view to suppressing muzzle flash by devising proplnt compns in which the proportion of flammable gases in the products of decompn was lowered, or alternately to lower the calorific value without increasing CO & H_2 contents, investigations were begun in 1921. Examination was made of a large number of high-nitrogen compds which were at the same time exothermic so as to decompose with min heat evolution. Aminotetrazole (See Vol 1, p A257-R) which contains 82.5% N has considerable merit but was unacceptable because of its hygroscopicity and absence of O in the molecule. Derivs of cyanamide & guanidine (such as dicyandiamidine nitrate, nitrosoguanidine, guanidine nitrate & nitroguanidine) were concluded to be the most promising compds. Proplnt compns with nitroguanidine have now been adopted by the British Services to replace earlier types (Ref 30)

Flashless Cordite, Characteristics. Several compns have been introduced in which the proportions of the ingredients are varied to meet differences in the characteristics of different guns in various aspects

The main shapes of such proplnts are cord, slotted tube & grains with a 7-hole perforation. Other forms which have been employed for special purposes are flake, ribbon, scroll & disc. The cord form is the simplest type to manuf & to control dimensionally, but has the disadvantage that as burning progresses the surface area decreases and thus the rate of gas evolution for a given pressure falls. With a tube or slotted tube form, where burning proceeds on both inner & outer surfaces, the surface area remains practically constant. With the so-called multi-tube or 7-hole tube, the total surface actually increases, as burning proceeds, up to about 30%. As a consequence of this surface relation it follows that a given vel of projectile can be obtd at a somewhat lower pressure with a slotted tube than with a cord and still lower with a multi-tube form. A further advantage of the grain multi-tube type is that the chge can be loaded mechanically into metal cartridge cases by pouring in a given wt or vol of proplnt

Apart from the risk of spontaneous ignition by chemical decompn a further danger with earlier proplnts was the ease with which they could be ignited by contact with heated metal such as shell fragments. A feature of British Cordites is that decompn on heating occurs without producing a flame even when in contact with metal heated to incandescence and when the remaining chge is removed from the heat source, the burning ceases after a short interval. This safety aspect has been regarded as an important consideration in the adoption of Flashless Cordite by the British Navy (Ref 30)

See also Vol 2, p C33-L, "Double-Base Cannon Propellants"

Preparation of NC for Cordite. See separate item at the end of this section

Manufacture of Cordite. **Mark 1 & MD or MC.** In the manuf of these Cordites gelatinization of Guncotton is brought about by NG, together with acetone, amounting to 20% for Mk1 and 35-38% acet-water (98/2) for MD. In addn to its action as a gelatinizer, acetone served to diminish the sensitiveness of Cordite paste during mixing & extrusion. In both compns the mixt of NC, NG & acet was worked in a mixer to give a dough or paste which was then extruded in a screw-or hydraulic-press thru a die-plate, thus forming cords from which the solv was removed. During the mixing operation, 5% of mineral jelly was added (Ref 3)

RDB Cordite. Because of the serious shortage of acetone during WWI, this modified Cordite was made from NC of 12.2% N, which enabled eth-alc to be used as the solv. A small reduction in calorific value was brought about at the same

CORDITES

British Double - Base Propellants

Designation	Composition [a] NC	% N	NG	MJ	Carb	Calorific Value (H_2O gas) Cal/gm	Total Gas cc/gn	Temp of Chamber Gases (Calcd) [b] °C	Gun Life Factor MD = 1
Mark 1	37	13.1	58	5	-	1125	888	3403	0.33
Md (or MC)	65	13.1	30	5	-	940	940	2939	1
RDB	52	12.2	42	6	-	911	960	2855	1.8
SC	49.5	12.2	41.5	-	9	890	957	2817	0.97
Cooled SC	50	12.2	36	-	14	750	-	-	1.2
Flashless			Various			670-805	-	2130-2525	3.7 - 8.0

[a] MJ - Mineral Jelly (vaseline)
Carb - Carbamite (Centralite) (or sym-diethyldiphenyl urea)

[b] The calcd chamber - gas temps are on the basis of no heat loss and prior to their adiabatic expansion in the gun barrel. At the time of shot ejection at the muzzle the temps have fallen, on the av, to about 0.6 of these values. After the further expansion of the gases to atm press outside the muzzle, the *mean temps* are estimated to ca 230° for Cordite SC, and to fall within the range 30-150° for flashless Cordite

time. The weighed NC (in which water had been replaced by alc) & NG were mixed in a kneading machine for 15 minutes with the calcd amt of alc added. The reqd amt of MJ was added and mixing continued for another 15 min. Finally the calcd amt of ether was added. A total of 60 p of solv contg 1.25 p of ether & 1 p alc by vol was used per 100 parts of Cordite. Blending was conducted until the mixture was homogeneous. The dough was extruded, cut to the desired size & dried (Ref 13a & 30)

Solventless Cordite. By dispensing with the use of volatile solvents and replacing them with a nonvolatile gelatinizing compd which remained in the final Cordite, considerable improvements resulted. Gelatinization was accomplished by heating & rolling NG, NC (12.2% N) & benzylphenylurethane. In later formulations, urethane was replaced by Carbamite (sym-diethyldiphenylurea), called in US Centralite I. This compn was prepd in cord form by an extrusion process

Cordite N. During WWII the British were using in some of their guns a composition called Cordite N. It consisted of NC (13.1% N) 19, NG 18.5, NGu 54.7, Carbamite 7.5, cryolite (K_3AlF_6) 0.3 & chalk 0.15 parts. Cordite N is about equal in force to US Pyro & NH proplnts and its estimated flame temp is 2430°K vs 2580°K for the US proplnts. However, two disadvantages of Cordite N discouraged its adaption by the US: a) the health & explosion hazards of NG volatization and b) the low mechanical strength of its multiperforated grains, especially at low temps (Refs 20, 24 & 32)

Samples of Cordite N with 1.5% K_2SO_4 added were made at the Nobel Works of Defense Industries, Limited for the Navy 6 inch/47 Gun. In tests at the Naval Proving Ground, this proplnt gave significantly less flash and was superior to the best FNH + 4% K_2SO_4. Cordite N later was proved flashless in major caliber cannon. However, the US Navy never used Cordite N in its regular operations, but this research with a NGu formula led to the development of a proplnt containing a plasticizer less volatile than NG. The flashless proplnt developed out of this research is called ALBANITE (See in Vol 1, p A119-L)

Cordite JP 76. This is a solventless proplnt consisting of NC(12.2% N) 49.6, NG 41.4 & Carbamite 9.0%. It was prepd in the form of grains 1.7" in diam, 1.0" long & diam of perforation 0.6" (Refs 23 & 35). Its decompn during accelerated aging was investigated chromato-

graphically by Schroeder et al (Ref 35)
Cordite N/M. This proplnt consists of NC (Gd C, Type 1, US Spec JAN-N-244) 19.0, NG 18.7, Carbamite 7.3, NGu 55.0% & KSO_4 1.00% added. It is listed in Ref 43
Cordite WM. It consists of NC (Grade C, Type 1) 65.0, NG 29.5, Carbamite 2.0 & MJ 3.5 with Ca carbonate 0.6% added. This compn is listed in Ref 43. See Ref 45 for its burning characteristics

The Cordite proplnts listed in Ref 48 are as follows: *Cordite CSC,* Unit No 280; *Cordite HSC,* Unit No 281; *Cordite SC,* Unit No 279; *Cordite SU/K,* Unit No 354; and *Cordite RS,* Unit No 282
Cordite Casting Powder No 1. This is a British proplnt described in Conf Ref 42, which is not used here as a source of info
Foreign Cordites. This infor concerns proplnts used during WWII:
Canada. Cordite was made at Transcona, Manitoba. Woodpulp was treated with mixed acids to form crude NC. After boiling for several days, the nitrated fibers were beaten in a papermaker's beater, having a large roll on the outside of which were metal knives. The knives chopped the fibers shorter and also loosened any impurities adhering to them. After washing to remove all foreign matter, the NC was centrifuged. A hydraulic press removed more moisture. While still in the press, alcohol was forced thru the compressed NC, replacing w. The resulting cake was broken in a kneader and the NG - acetone mixt was added. Chemical stabilizers were added to the mass while still in the kneader. The crumbly dough was extruded in the form of long cords which were cut to the desired length and dried. Since each batch of Cordite exhibited sl different characteristics, the practice was to blend a number of batches together. The blended Cordite was packed in wooden boxes, having a waterproof liner, and shipped to either magazine storage or the ammo loading plant (Ref 18)
Italian Cordite or Polvere C_2. It consisted of NC (mixt of Collodion Cotton & Guncotton, 50% sol in eth - alc) 71, NG 23, vaseline 5 & Na bicarbonate 1%. This compn was introduced about 1910 and prepd similar to British Cordite. Sodium Bicarbonate was added to prevent back-flash in firing the Cordite. The proplnt was prepd in the form of rods & tubes (Refs 14, 33 & 41)
Russian Cordite. It consisted of NC (12.2% N) 56.5, NG 28, DNT 11 & Et centralite 4.5% with 0.08% candelilla wax added. Its props were: heat of expln at const vol, w liq, 880 cal/g and adiabatic flame temp with reaction at const pressure 3750°F. This proplnt was considered to be cooler & slower burning than JPN proplnt (Ref 37)
Uses. Cordite is a standard British ("Service") propellant which has been used for over 60 yrs. It was supplied in great quanty to Japan during the Russo-Japanese War of 1904-05. The US also used Cordites during WWI, but soon abandoned their use, because of the excessive corrosion of gun barrels, in favor of single-base propellants. Cordite has also been used in exptl mixts developed as highly brisant expls (Ref 15)
Refs: 1) F. Abel & J. Dewar, BritP 5614 (1889) & JSCI **11,** 709 (1892); and BritP 11664 (1889) & JSCI **9,** 765 (1890) 2) Anon, SS **3,** 251-52 (1908) (MD Cordite) 3) Anon, SS **7,** 135-36 (1911) [Manf of Cordite; A brief translation from "Treatise on Service Explosives," London (1907)] 4) Marshal **1** (1917), 304 & 334 5) Barnett (1919), 78 6) W. Macnab, JSCI **41,** 356T (1922) 7) Brunswig, Props (1926), 54 8) Pascal (1930), 158 & 165-67 9) Marshall **3** (1932), 89 10) Vennin, Burlot & Lécorché (1932), 629 11) Stettbacher (1933), 196 12) Pepin Lehalleur (1935), 313-16 13) Hayes (1938), 13 & 69 13a) Thorpe **3,** 361-64 (1939) 14) Piantanida (1940), 179 15) J.D. Hopper, PATR **1041** (1940) (Expl mixt consisting of Cordite/ TNT-49/51; d of cast 1.58 vs 1.63 for 30/70 Pentolite; crushed 45g sand vs 44 for Pentolite & 38 for TNT) 16) Cond Chem Dict (1942), 288 17) Alvaro-Alberto, AnaisAcadBrasilCienc **14,** No 4, 341 (1942) & **15,** No 1, 40-41 (1943) & CA **40,** 2629-30 (1946) (Props of Cordite **1,** Cordite MD and a discussion of erosion of guns) 18) Anon, ChemMetEngrg **50,** 130-33 (1943) 19) Davis (1943), 295 20) W.H. Rinkenbach, PATR **1336** (1943) (Thermochemical & physical tests of Cordite N, manufd in Canada, in comparison with other proplnts, especially those contg NGu) 21) W.H. Rinkenbach, PATR **1359** (1943), p 2 & Table III (Stability of Cordite & double-base proplnts) 22) Pérez Ara (1945), 414 & 443 23) L. Pauling, OSRD **5967** (1945), 10 (Cordite JP 76) 24) Anon, Summary Technical Report of Division 8, NDRC Vol 1 (1946), 107 (Cordite N) 25) J. Corner, ProcPhysSoc (London) **58,** 737-58 (1946) & CA **41,** 1513-14 (1947) (Detailed study

of the products of expln of Cordite) 26) Mangini, Esplosivi (1947), 235 27) D.D. Eley et al, TrFarad Soc **43,** 559-91(1947) & CA **42,** 2772 (1948) (Visco-elastic behavior of Cordite) 28) J. S. Weiner & M.L. Thomson, BritJIndMed **4,** 205-15 (1947) & CA **42,** 2771 (1948) (Cordite does not seem to be toxic when handled at RT, but it is definitely harmful in a room at 86°. This is attributed to presence in atm of NG, nitrous gases & traces of CO. The main channel of absorption of Cordite is apparently thru the lungs) 29) C.A. Parker, JSCI **67,** 434-36 (1948) & CA **43,** 3617 (1949) (Mechanism of aging of Cordite, stabilized by Et centralite, was investigated by chromatographic separation) 30) J. Pring, "Modern Propellants Employed in British Ordnance", Paper read at a Meeting of the Chemical Engineering Group, 56 Victoria St, London, SW1 (31 May 1948) 32) W.A. Noyes Jr. Edit, "Science in WWII", Little Brown & Co, Boston (1948), 129-30 33)Giua, Dizionario, vol **2** (1949), 155 34) R. C. Brian & C.A. McDowell, TrFaradSoc **45,** 212-13 (1949) & CA **43,** 5188 (1949) (Ignition of Cordite by hot gases) 35) W.A. Schroeder et al, IEC **42,** 544 (1950) (Cordite JP 76) 36) J.D. Huffington, Nature **165,** 840-41 (1950) & CA **46,** 4234 (1952) (Anomalous rates of buring of Cordite) 37) Wimpress, Ballistics (1950), 4 38) SACMS, Ballistics (1951), 4-5 39) Kirk & Othmer **6** (1951) 77 40) J.D. Huffington, TrFaradSoc **47,** 864-76 (1951) & CA **46,** 2297 (1952) (Burning characteristics & structure of Cordite) 41) Belgrano (1952) 113 42) P.W.J. More & R. Pape, ERDE Tech Mem 14/M/54 (Conf) 43) Armament Engrg (1954), 43 44) D.M. Clemmow & J.D. Huffington, TrFaradSoc **52,** 385-96 (1956) & CA **50,** 14229 (1956) (Critical thickness & surface temp at initiation & at explosion were calcd for Cordite) 45) H.K. Acharya & V.M. Khanna, Chem & Ind (London) **1958),** 557-58 & CA **53,** 718 (1959) [Burning characteristics of a Cordite prepd in cord form: NC (13.1% N) 65, NG 29.5, Carbamite 2 & MJ 3.5%] 46) Giua, Trattato **6** (1959), 358-62 47) C.A. Heath & R. Hirst, 8th Symp Combustion, Pasadena, Calif **1960,** 711-20 (Pub in 1962) & CA **57,** 7505-06 (1962) (High-pressure combustion of Cordite SU/K) 48)Propellant Manual SPIA/M2 (1962) (Conf)

Cordite, Manufacture of Nitrocellulose The earliest process used in England for manufg NC was that of Abel (ca 1865). This process may be considered as a modification of the *pot process* (Topfverfahren in Ger) invented in 1852 by Baron Von Lenk of Austria. In the von Lenk process the nitration was carried out in two stages The 1st stage was the dipping of the skeins of cotton yarn in mixed nitric-sulfuric acid contained in earthenware nitrating pans, and the 2nd stage the completion of the nitration by allowing the partially nitrated skeins to remain in contact with a weaker acid, in earthenware pots, for a prolonged time (Ref 2, p 180; Ref 3, p 169; Ref 4, p 130; & Ref 5, p 353)

The following slightly different description of the "pot method" is given in Ref 4a, p 12: The dry cellulose was dumped into earthenware or iron pots contg mixed nitric-sulfuric acid, where it was nitrated for 45 mins to 2 hrs. The resulting NC was separated from the spent acid which could be done by several different means, all based on exerting pressure on the material. After the bulk of spent acid was thus removed, the NC was drowned in w. In the later modification of this method, the centrifuge was used to remove the acid. In order to get a greater efficiency and reduce to a minimum the size of the nitrating room, 6 or 8 pots were attached to iron supports located on a circular turntable. After partly filling the 1st pot with acid and then immediately (by means of Al forks) drowning the dry cellulose, the pot was covered and the table turned to have the 2nd pot placed below the acid feed pipe. After loading this pot with acid and cellulose, the same operation was conducted with the 3rd pot, etc. After completion of nitration, the 1st pot was dumped into the centrifuge and, while the opn of separating spent acid from NC proceeded, the 1st pot was reloaded. Then the NC was removed from the centrifuge and drowned in w. The same opn was conducted with the 2nd pot, etc. This method of nitration was designed by Hyatt and is known as the *table method* (Ref 4a, p 12)

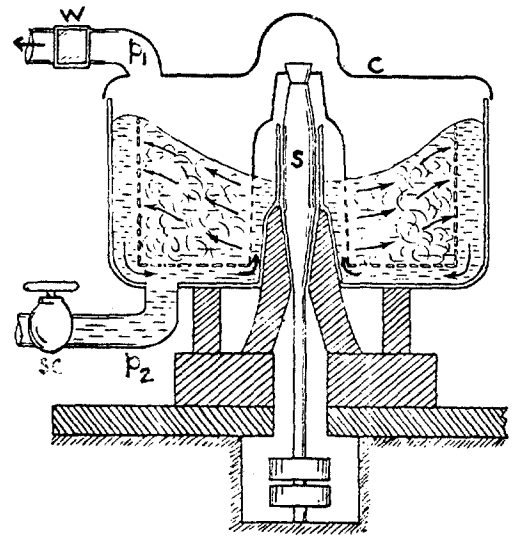
Following is the list of methods used for manufg Cordite - grade NC (Guncotton):

A. Abel's Process. In this process, cotton waste was used, instead of the skeins of cotton yarn employed by von Lenk. Batches of 1.25 lbs were dipped into 220 lbs. of mixed nitric-sulfuric acid contained in cast-iron dipping pans which were supported in an iron tank thru which cold w circulated to keep the temp below 70°F. The pans were provided, at the back, with gratings, on which to press out part of the acid from the charge. After remaining in the acid bath for 5

to 8 mins, the cotton was removed to the grating and a portion of the acid squeezed out by means of an iron lever having an iron plate attached to one end. The charge, weighing with adhering acid ca 15 lbs, was placed in an earthenware pot, covered with a light lid and set in a trough with circulating w. Nitration proceeded for 12 hrs, after which the contents of several pots were emptied into a centrifuge, where the excess acid was wrung out as much as possible. The NC was then removed and immersed quickly in a large volume of cold w, which was circulating thru the tank. After stirring for a while with a wooden paddle, the inflow of w was stopped and the tank drained. Then the tank was refilled with fresh w and the operation of washing repeated ca 6 times. The material was transferred to a centrifuge where the bulk of w was wrung out (Ref 2, p 180; Ref 3 p 169; Ref 4, p 45; Ref 5 pp 353-54). Stabilization by boiling in w is described in Ref 2, p 183. The yield on dry cotton waste was 163.7%

B. Direct Dipping Process, Also known as *Nobel's Method,* was an improvement over Abel's Process. The method was developed and used for many years at Nobel's Factory Ardeer, Scotland. Here the cotton waste was digested with 6 parts of mixed acid for 8 to 24 hrs at 20°, in water-cooled cast-iron pans, called "dippers", covered with Al lids. When the nitration was complete, the dippers were emptied into a centrifuge where the bulk of waste acid was wrung out. Then the NC was quickly removed by portions and thrown under the revolving paddles of the drowning tank, where it was quickly immersed in w. After a prescribed quantity of NC had been drowned, the acidic w in the tank was run off and the material was thrown on the draining table forming part of the drowning tank. The next step was washing of crude NC in a centrifuge with cold w from a hose, followed by transfer to the boiling house for stabilization (Ref 2, pp 180-81; Ref 3, 171-72; Ref 4, p 45; Ref 5, pp 354-55). Stabilization of NC by boiling in w is described in Ref 2, p 183. The yield on dry cotton waste was only 159.0%

Nitration by any of the above described methods produced good grades of NCs, but the consumption of acid was great and a quantity of cotton was wasted. Lots of toxic fumes escaped from the nitrators, on drowning. These fumes were injurious to workers. A great deal of labor was involved and charges of NC often fumed off while NC was still in contact with acid. In addn, both methods were time-consuming and required large quantities of w for washing the crude NC. These disadvantages led to the introduction ca 1905 of "centrifugal process", patented in Germany by Selwig-Lange and of the "displacement process", patented in England by F. & W. Thomson



CENTRIFUGE OF SELWIG-LANGE

C. Centrifugal Process or Selwig-Lange Method. It has been extensively used in continental European countries (such as Germany, France, Italy, Sweden, etc) and only to a limited extent in Great Britain. One of the older British centrifugal installations, for manuf of Cordite NC was at Dartford Works of Curtis & Harvey. The later model (See Fig), is known as the *nitrating centrifuge with acid circulation.* It consists of the outer casing with Al cover, c, provided with a pipe, P_1, through which the nitrogen oxide gases (formed during nitration of cellulose) are exhausted by an exhaust fan to an acid recovery tower. The color of these gases can be observed thru the thick glass window, w. The inner casing of the centrifuge consists of a perforated Al or iron basket which is attached to a vertical shaft, s, driven by a pulley. A pipe, P_2 provided with a stopcock, sc, is used to run in the mixed nitric-sulfuric acid and to draw off the waste acid. The procedure is briefly as follows: With the cover, c, removed and the inner casing rotating at 25 to 30 rpm, the mixed acid, cooled to 15°, is introduced thru sc and P_2 until about

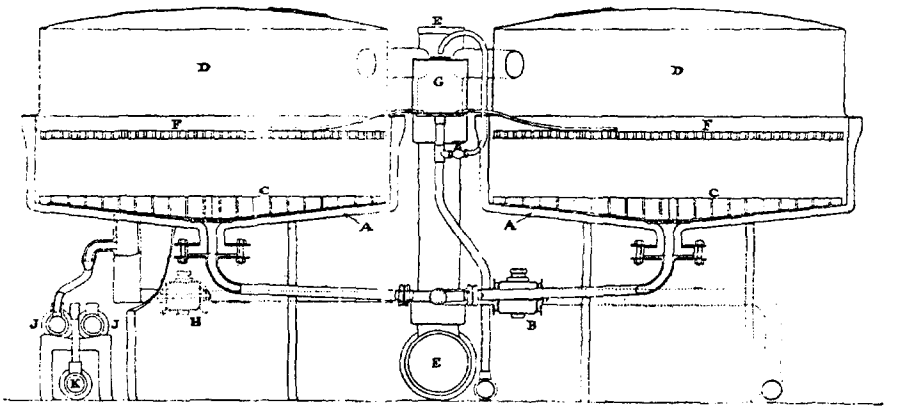
7/8th of apparatus is filled, Then cotton waste is immersed in the acid, a little at a time, until there is about 1 part of cotton per 50 parts of acid. After replacing the cover, c, the nitration is allowed to proceed for ca 30 mins. During this time, the acid is constantly circulating thru the cotton, thus achieving its uniform nitration. After this the stopcock, sc, is opened so that part of the waste acid can be drained by gravity. Then the rate of rotation is gradually increased so as to wring out the superfluous acid. This wringing should not,however, be carried too far, so as to avoid danger of the charge fuming off. As a rule, the acid content of nitrated cotton is not reduced below 50-60% before drowning it in w, which is done in the tank (provided with paddles), in the manner similar to the method which is briefly described under Direct Dipping Process (Ref 2, p 181; Ref 3, pp 170-71; Ref 4, p 48; Ref 6, pp 255-56; Ref 6, pp 131-32; Ref 7, pp 378-80; Ref 8, pp 100-02). Method of stabilization of NC by boiling in w is described in Ref 2, p 183. The yield on dry cotton waste, at Dartford Works, was only 160.0% when centrifuges were used w/o acid circulation

D. Displacement Process of Thomson, called also Nathan and Thomson's Displacement Process. It has been used since 1905 at Waltham Abbey, England. Most of the British plants manufg NC for use in Cordite are still using this method. For example, the Cordite plant of Canadian Industries Ltd at Beloeil, near Montreal used this process during WWII. Thomson's nitrators were installed in 1908 at Picatinny Arsenal, but were later replaced by the DuPont mechanical dipper equipment, briefly discussed in Vol 2, p C102-R of this Encycl

Thomson's nitrator (See Fig, which is a copy of Fig given in Ref 2) is a shallow earthenware pan, 3.5 ft diam, 10" deep at the sides and 12" deep at the center, where a run-off cock is located. The pan has a false bottom of perforated earthenware segments. Four nitrators are usually connected together by lead pipes so as to be worked as one unit. These pipes are also connected, (thru a gage-box) to the mixed acid storage tanks, to the waste acid tanks, and to the drain system. The mixed acid used at Waltham Abbey consists usually of HNO_3 21.0, H_2SO_4 70.5, HNO_2 0.6 & H_2O 7.9%

In carrying out the nitration, 650 lbs of mixed acid is allowed to run into each pan, and then 20 lbs of cotton waste, added in single handfuls, quickly pushed under the surface of the acid by means of an Al fork. During this manipulation the pan is covered by a light portable Al hood connected with a fume pipe so that toxic gases are drawn off. When all the cotton waste has been drowned in acid, perforated earthenware plates in segments are placed on top of the charge. Then a layer of w, cooled to ca 5°, is added very slowly, thus sealing off the nitrator in order to prevent escape of toxic fumes into the atmosphere. The time required for dipping a charge is ca 15 mins. At the expiration of a 2.5-hr period, the cock leading to the gage-box is opened, and the waste acid is allowed to run off at the rate of ca 1

WALTHAM ABBEY GUNCOTTON DISPLACEMENT PLANT



A —Earthenware nitrating pans
B —Acid inlet cock
C —Perforated earthenware plates
D —Aluminium fume hoods
E —Fume pipe
F —Perforated earthenware plates on top of guncotton
G —Water distributor
H —Outlet cock
J —Waste acid pipes
K —Waste water pipe

gallon per minute. At the same time and at the same speed, cold w is run on the top of the perforated plate thru the distributor. Because water is much lighter than acid, the line of demarkation between them remains fairly sharp. About 80% of waste acid can be revivified with concd nitric acid and oleum for use as a mixed acid in the next batch. As the remaining 20% of waste acid is too week to be rivived, it is sent to the recovery house where it is denitrated and concentrated. A small amt of w which follows the waste acid contains only ca 0.1 lb of acid per 1 lb of w and this is the total quantity of acid which is lost during the process. The whole of the acid is displaced in 3 hrs, and the w, which should fill the pan, is run thru NC. Then w is drained off and NC is sent to the boiling house (Ref 1; Ref 2, pp 181-83; Ref 3, pp 174-79; Ref 4, pp 46-8; Ref 5, pp 359-62; Ref 6, pp 135-36)

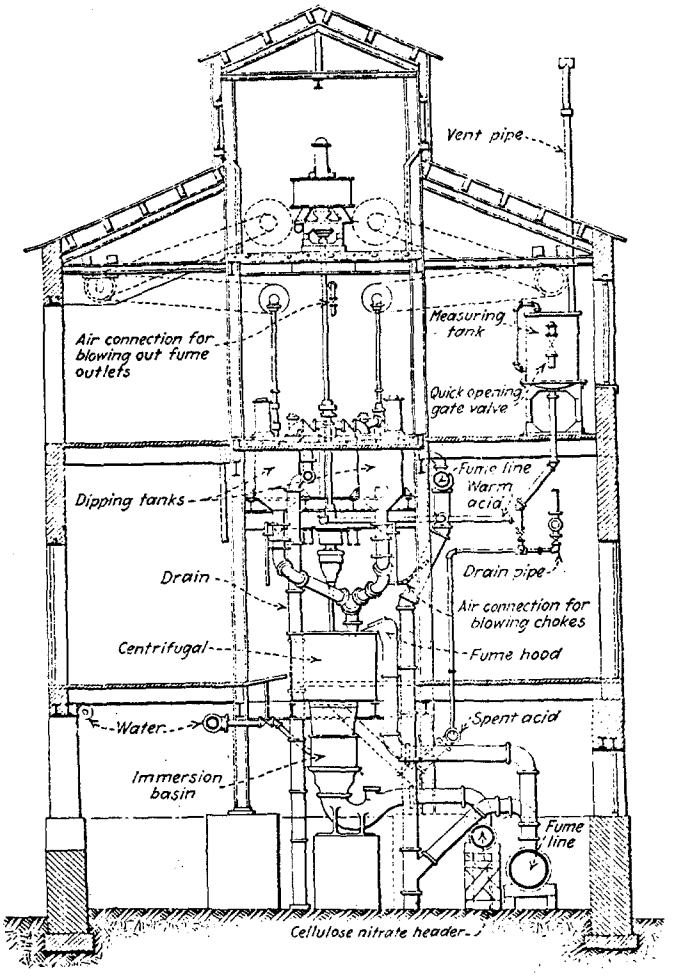
Nitrators can be made also of enamelled cast iron or stainless steel. Naked cast iron or lead cannot be used because, although they would not be attacked during nitration, they would be seriously affected on dilution of acid during the later stages of displacement

Stabilization of NC by boiling in w is described in Ref 2, p 183. The yield on dry cotton waste was 170.0% at Waltham Abbey. The output per man per week was 1742 lb , as against 458 by Abel's Method and 1112 by Direct Dipping Method (Ref 2, p 182)

E. DuPont's Process. Some Cordite plants replaced the Thomson method with the "DuPont's Mechanical Dipper Process". This method was briefly described under CELLULOSE AND DERIVATIVES in Vol 2, p C102-R of this Encycl. A more detailed description and a schematic view (See Fig) of the process are given in Ref 4a, p 13

Refs: 1) G. Lunge, "Das Verdrängungsverfahren von F. u. W. Thomson zur Herstellung von Nitrozellulosen, SS **1**, 2-4 (1906) 2)Sir F.L.Nathan, "Guncotton and Its Manufacture," JSCI **28**, 177-87 (1909) 3) Marshall **1** (1917), 169-79 4) Barnett (1919), 45-48 4a) J.R. DuPont, ChemMetEngrg **26**, 11-16 (1922) (varions methods of prepg NC) 5) Vennin, Burlot & Lécorché (1932) 353-61 6) Stettbacher (1933), 130-36 7) Pérez Ara (1945), 378-82 6) Belgrano (1952), 100-02

Cordite, Analytical Procedures. The analysis of a typical Cordite proplnt having the compn



CELLULOSE NITRATE PLANT USING MECHANICAL DIPPING TANKS

of *Cordite WM* (given under CORDITE) is as follows:

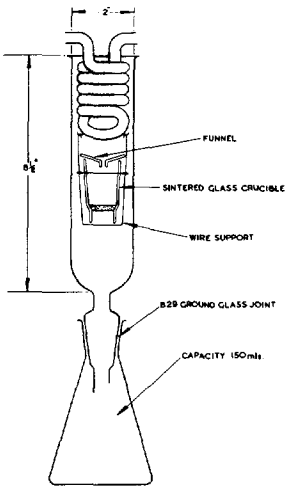
Nitrocellulose (Ref 1).Place a sintered glass crucible in an opened weighing bottle. Dry in an oven at 103-105°C for 1 hour. Cool in a desiccator for 40 minutes and weigh. Transfer to thin glass crucible a 5 ± 0.1 g sample of propellant and reweigh the assembly to obtain an accurate weight of the propellant taken

Transfer the crucible to the wire support of a Wiley-type extraction apparatus (See fig) and extract the propellant for 3 hours with ethyl ether using a tared 150 ml conical flask as the extraction vessel. Remove the ether from the crucible by suction. Then dry the crucible in the weighing bottle at 100°C for 2 hours. Cool in a desiccator and weigh. Repeat the drying until the difference in successive weighings does not exceed 2 mg.

Calculate the % NC (including chalk) from the formula: $\%NC = \frac{W_1 \times 100 \times 100}{\text{Wt of proplnt} \times (100 - \% VM)}$

where W_1 wt of dry residue after extraction and VM = Volatile matter in sample

MODIFIED WILEY-TYPE EXTRACTION APPARATUS



Mineral Jelly (Ref 1). Evaporate most of the ether from the extract on a water bath. Remove the remainder with a stream of air. Dry the residue in an oven at 60° C for 1 hour. Then add 20 ml of 80% methyl alcohol (by volume) and heat to gentle boiling on a water bath. Decant the hot solvent thru a small funnel fitted with a 7 cm No 41 Whatman filter paper and collect the filtrate in an iodine flask. Retain as much mineral jelly in the extraction flask as possible. Repeat the extraction twice, using 10 ml of 80% methyl alcohol making 40 ml in all. Finally wash the filter paper six times with hot 80% methyl alcohol

Transfer the filter paper to a Soxhlet extractor using the original extracting flask containing the mineral jelly. Extract the paper for 2 hours with ethyl ether. Distil the ether from the flask on a water bath and then in an oven at 100°C to constant weight. Calculate the % MJ from the formula:

$$\% \text{ Mineral Jelly} = \frac{\text{Wt of MJ} \times 100 \times 100}{\text{Wt of proplnt} \times (100 - \% VM)}$$

EthylCentralite (Carbamite) (Ref 1). Distill the methyl alcohol from the iodine flask (above) on a water bath. Take care that all methyl alcohol is removed, but do not overheat the Carbamite, which is volatile in steam and may be lost. Add 50 ml of carbon tetrachloride, followed by 25 ml of a 0.2N aqueous solution of potassium bromate containing potassium bromide (5.568 g of potassium bromate and 30 g of potassium bromide per liter). Enclose the flask in a lightproof bag during the subsequent bromination of the Carbamite. Add 10 ml of concentrated hydrochloric acid to the flask, replace the stopper at once and shake vigorously for exactly 5 minutes. Add 20 ml of 15% potassium iodide solution by pouring it around the stopper, a little at a time, and easing the stopper gently so as to admit the solution without losing bromine. Shake the flask again vigorously, remove the light-proof bag and titrate the liberated iodine with 0.1N sodium thiosulphate, shaking the flask continuously during the titration. It is important to ensure that at no stage before the end-point is sodium thiosulphate present in excess in any part of the flask, otherwise it will be decomposed in the strongly acid medium. The end-point is reached when the last drop of sodium thiosulphate solution added causes the carbon tetrachloride layer to turn from pale pink to colorless. The flask should be stoppered, inverted and held up to the light to observe the end-point

Carry out a blank test, in duplicate. When making a series of Carbamite estimations, perform one blank test before and the other at the completion of the series

1 ml of 0.1N sodium thiosulphate = 0.01342 g of Carbamite. Calculate the % Carbamite using the formula:

$$\% \text{ Carbamite} = \frac{\text{Net titer x factor of 0.1N } Na_2S_2O_3 \times 134.}{\text{Wt of propellant x } (100 - \% VM)}$$

Nitroglycerin (Ref 2). The NG may be calc by difference or determined directly as follows:

Extract a 2 ± 0.02g sample with ethyl ether as described above. After evaporation of the ether, make the extract up to 100 ml in a graduated cylinder with alcohol. Displace the air from an empty 250 ml titration flask fitted with an inlet tube through which carbon dioxide can be passed, add a 25 ml aliquot portion of the extract above, followed by 25 ml of concentrated hydrochloric acid and 25 ml of 0.7 N ferrous ammonium sulphate which has been freshly reduced by shaking with liquid zinc amalgam. Connect the flask to a reflux condenser and gradually bring the contents to incipient boiling and, when the color begins to

lighten, increase the heat to produce moderately vigorous boiling. Continue boiling until the color ceases to become lighter and then for a further five minutes. Increase the rate of flow of carbon dioxide, cool the flask to room temperature by means of a water bath and wash down the condenser with 30 ml of alcohol

Titrate the contents of the flask with 0.2N titanous chloride, adding 10 ml of 10% ammonium thiocyanate solution near the end point. Titrate slowly in the final stages allowing 10 seconds with good agitation between each drop. Carry out a blank determination on the reagents used and subtract from the above titer

1 ml of 0.2N $TiCl_3$ = 0.005047 g NG

$$\% \text{ NG} = \frac{\text{Net titer} \times 0.2\text{N}TiCl_3 \times 50.47 \times 4}{\text{Wt of proplnt} \times (100 - \% \text{ VM} - \% \text{ cryolite})}$$

Calcium Carbonate (Ref 1). Add 5 ± 0.1 g of the finely ground propellant very gradually to 100 ml of acetone contained in a 400 ml beaker, stirring vigorously by mechanical means during the addition and for the remainder of the determination. When the solution is complete, add 50 ml of 0.05N hydrochloric acid, the first 10 ml drop by drop to precipitate the NC in a fine state of division, and then the remainder quickly until the whole quantity has been added. After 10 minutes add 2 ml of bromo-phenol blue solution and titrate with 0.1N sodium hydroxide. To confirm the endpoint, stop the stirrer, allow the NC to settle and compare the color of the supernatant liquid with that in a blank experiment, which must be carried out in an exactly similar manner, except that the propellant is omitted

$$\% \text{ Calcium Carbonate} = \frac{\text{Titer diff} \times \text{Factor of 0.1N NaOH}}{10}$$

(This section on Alanlytical Procedures of Cordite was written by E.F. Reese, FRL, Picatinny Arsenal)

Refs: 1) Method EA 2, "The Examination of Propellants WM and CD/T "Amendment 1 (Jan 1, 1956) Ministry of Supply, Chemical Inspectorate (United Kingdom) 2) Method EA 8, "The Examination of Propellants DU and DUK", (Apr. 12, 1954) Ministry of Supply, Chemical Inspectorate (United Kingdom)

Cord-Like Ignition Device for Explosive Charges. Such devices suitable for use in initiating a number of expl chges, as in mining, military, or pyrotechnic operations, were patented by the Ensign-Bickford Co. These devices consist of a readily combustible core made with a plurality of yarns (each individually coated with deflagrating pdr) confined by a tape of less-combustible material. The tape is spirally wound about the core and secured so as to leave a relatively narrow spiral flame escape area running along the cord. The ensemble is coated with a waterproof material which is destroyed by the hot combustible gases. The deflagrating powder consists of mealed Blk-Pdr & a NC binder

Ref: Ensign-Bickford Co, Brit P 541202 & 541 363 (1941) & CA **36,** 5018 (1942)

Cordtex Detonating Fuse. See under Cord, Detonating

Core Charges or Core Explosives. See under COMMERCIAL OR INDUSTRIAL EXPLOSIVES, Section II, Coal Mining Explosives, Permissible

Cork (or Suber). The outer tissues of the stems of the cork oak or the exterior layers of the bark beneath the epidermis. In young stems it consists of epidermis, cortical tissues & periderm; and in older stems of secondary phloem & periderm. Cork is used in some expls mixts described below

Refs: 1) Hackh's (1944), 227 2) Merriam-Websters (1961), 507

Cork, Carbonized. Davis (Ref) reports that liquid oxygen explosives (LOX) made from carbonized cork and from kieselguhr mixed with petroleum were used in blasting the Simplon tunnel in 1899

Ref: Davis (1943), 356

Cork Powder Explosives. Aubert & Nauckhoff (Ref) patented the addn of cork powder to gelatinized expls, such as mixts of NC & NG. The cork permanently retains a certain quantity of occluded air and thereby ensures rapid transmission of deton thruout the expl. One example of such a blasting expl consists of NG 56, NC 2.3, NB (nitrobenzene) 1.7, AN 35 & cork pdr 5%

Ref: A. Aubert & S.A.G. Nauckhoff, BritP 1283 (1915); USP 1186065 (1915) & CA **10,** 1932 & 2045 (1916)

Cornaro's Explosives. Cornaro patented the following expl mixts: a) Finely divided metals, such as Al & Sb, and oxidizing agents (not specified); b) Al 20-21.5, As 20-21.5 & KNO_3 or $KClO_4$ 60-57%, the particles of Al & As preferably being coated with 2-4% of varnish (1/1/1 soot/boiled linseed oil/turpentine) (Ref 3): c) Al 20-21.5, Zn 20-21.5 & KNO_3 or $KClO_4$ 60-57%, the metals preferably being coated as above (Ref 4); e) Al 8-12.5, Sb 8-12.5 & MnO_2, KNO_3 or $KClO_4$ 75-84%, the metals preferably being coated as above (Ref 5)

Refs: 1) G. Cornaro, Brit P 9170 (1907) & CA **2,** 205 (1908) 2) ibid, FrP 376607 (1907) & CA **3,** 249 (1909) 3) ibid, USP 989947 (1910) & CA **5,** 2332 (1911) 4) ibid, USP 989948 (1910) & CA **5,** 2332 (1911) 5) ibid, USP 990036 (1910) & CA **5,** 2332 (1911)

Corncob Cellulose. The possible use of corncob cellulose in the expl industry is discussed by Marsh (Ref). It appears that the only use for it is as a carbonaceous absorbent for liq ingredients such as NG, used in the manuf of dynamites. See also Cornstalk

Ref: L.G. Marsh, IEC **13,** 296-98 (1921) & CA **15,** 1400 (1921)

Corner's Equation of State. At the high temps & pressures encountered in explosives technology, the perfect gas law is not applicable. The calculation of pressures developed by expls therefore requires the adoption of a suitable equation of state. Furthermore, in calculating the explosion products it is necessary to correct the ideal thermodynamic equilibria of the relevant chem reactions for the effect of the gas imperfection. This correction also depends on the equation of state adopted. Most equations of state, whether empirical or theoretical, are not suitable for application at the high temps & pressures developed by expls. Corner has recently discussed a theoretical equation of state applicable to propellent expls (Ref 3)

The internal energy, heat content, sp heat & equil constants of the normal products of combustion are expanded as a power series, in the density, by Corner (Ref 1). Corner's equation of state is as follows (Ref 1 & 2):

$$\frac{PV}{nRT} = 1 + \frac{B}{V} \times \frac{nC}{V^2} + \ldots .$$

where P = pressure, n = moles of gas, R = gas constant, T = Temp in ^{o}K, B & C = virial coefficients, and V = volume of gases. At pressures up to 40 tons/in^2, corresponding to density of 0.35 g/cc, only the first 3 terms in the equation need be kept. Thus the pressure dependence of the thermodynamic props can be evaluated from a knowledge of the 2nd & 3rd virial coeffs of the various gaseous products. Tables are presented which cover the range 1600^{o} - 4000^{o} K, and which have found considerable application in internal ballistics. These tables give "covolumes" of propellants with a systematic error of less than 5%. The basis of Corner's theory is the expression of the 2nd virial coefficient of a gas as a simple function of the parameters of the intermolecular field

Murgai (Ref 4) modified Corner's equation and used it to calc some props of "condensed explosives". The equation of state for the mixts of detonation products of HE was written as:

$$\frac{PV}{nRT} = 1 + \frac{nB}{V} + \frac{n^2C}{V^2} + \psi$$

All terms except the last are known from Corner's equation. The last term, corresponding to the 4th virial coefficient, was evaluated by analysis of deton vel for PETN as a function of loading density. It turns out that ψ can be absorbed in the 3rd virial coefficient C

In later work, Murgai (Ref 5) extended Corner's equation of state to the more extreme regions of pressure obtd in the deton of condensed expls

See also DETONATION AND EXPLOSION, Equations of State

Refs: 1) J. Corner, ProcPhysSoc (London) **58** 737-58 (1946) & CA **41,** 1513-14 (1947) 2) Corner, Ballistics (1950), 104 3) Taylor (1952), 33 4) M.P. Murgai, ProcNatlInstSciIndia **20,** 548-56 (1954) & CA **49,** 11281 (1955) 5) M.P. Murgai, JChemPhys **24,** 635 (1956) & CA **50,** 9078 (1956)

Cornet Powder. An expl composition consisting of $KClO_3$ 75 & rosin 25%. Cf with Comet Powder

Refs: 1) Daniel (1902), 171 2)Giua., Trattato **6,** (1959), 399

Cornhusker Ordnance Plant. A US Army Munitions Command, field installation located at Grand Island, Nebraska
Ref: OrdTechTerm (1962), 85

Cornil or Poudre Blanche. A Favier-type Belgian safety expl consisting of AN 77, Amm chloride 18, DNN 3, KNO_3 1 & Pb chromate 1%. The chge limit in Gt Britain was 500 g
Refs: 1) Gody (1907), 714 2) Marshall 1 (1917) 390 3) Marshall, Dict (1920), 25

Corning Mill Dust. This is also called *meal powder,* or by the Fr *pulvérin.* Corning mill dust is the finest and the most intimately incorporated BkPdr dust remaining in the mill after the "corning" or granulating operations. Various types of machines are used to manuf & granulate BlkPdr, such as "Wheel Mill" & "Stamp Mill" illustrated by Davis (Ref 2), and "Corning Machines" illustrated by Marshall (Ref 1)

Corning meal dust is used in the manuf of fireworks & fuse powers. For certain purposes, some manufacturers find it is superior to other kinds of meal powders
Refs: 1) Marshall **1** (1917), 25 & 82 2) Davis (1943), 5, 44 & 46

Cornish Powder. A Brit coal-mining expl, formerly on the "Permitted List." It consists of NG 55, NC 3, KNO_3 18, woodmeal 7 & Mg sulfate 17%. This powder was manufd by the National Explosives Co, Ltd.
Ref: Marshall, Dict (1920), 25

Cornstalk Explosives. A low density dynamite which was made from coarse AN, ground cornstalk & NG. Cartridges 1¼" x 8" were loaded so as to weigh less than 146 g. Their vel of deton was less than 2500 m/sec (Ref 2). Kirst & Marshall (Ref 3) patented another low density dynamite consisting of AN 58, $NaNO_3$ 9, NG 15 & cornstalk pith 18%

The possibility of producing refined cellulose in Italy was reviewed by Contardi & Ciocca (Ref 4). It is reported that the cellulose obtained from corn fodder & corn straw could be satisfactorily processed to produce artificial fiber, expls celluloid, films & varnishes

Worden describes procedures by which raw pith of maize or cornstalk was successfully nitrated and used in the manuf of dynamites (Ref 1). See also Bebie (Ref 5) and Ott et al (Ref 6)
Refs: 1) E.C. Worden, "Technology of Cellulose Esters", Eschenbach Print Co , Easton, Pa (1921), 1805-06 2) J. Marshall, USP 1614447 (1927) & CA **21,** 824 (1927) 3) W.E. Kirst & J. Marshall, Can P 339433 (1934) & CA **28,** 2908 (1934) 4) A. Contardi & B. Ciocca, Ricerca Sci **8II,** 155-59 (1937) & CA **34,** 4899 (1940) 5) Bebie (1943), 52 6) Ott, Vol 5, Part 1 (1954), 21

Coronite or Permittite. A Brit coal-mining expl which, at one time, was on the "Permitted List." It consisted of NG 38 - 40, NC 0.8 - 1.3, AN 26-28, KNO_3 3-5, liq hydrocarbon 2-4, rye flour 8-11, woodmeal 2-4 & Al stearate 11-14 parts. This mixt is a *Carbonite* (qv) type expl. It also has been called *Permittite*

Coronite is also one of the Swedish names given to Picric Acid

Bebie (Ref 5) and Warren et al (Ref 6) list, under this name, the Ger expl called *Koronit* (qv)
Refs: 1) Marshall **2** (1917), 557 2) Marshall, Dict (1920), 25 3) Naoúm, NG (1928), 409 4) CondChemDict (1942), 288 5) Bebie (1943), 52 6) F.A. Warren et al, "Chlorates and Perchlorates Their Manufacture, Properties and Uses" NAVORD Report **7147,** Vol 1 (1960), p 205, Contract NOrd 18471

Corporal (Missile). The name applies to a US Army surface-to-surface ballistic missile designed for use as a tactical weapon against enemy troop concentrations or installations. It can carry either a conventional HE or a nuclear warhead
Ref: OrdTechTerm (1962), 85

Corpse Light. It is the blue flame inside the Miner's Safety Lamp, also called *Davy Lamp.*

See under COAL MINE EXPLOSIONS AND FIRES. The Corpse light is used to detect & indicate the presence of firedamp which occurs in coal mines
Ref: Hackh's (1944), 228 & 342 (under firedamp)

Correlation of Explosive Properties with Other Parameters. "The Relationship between Performance and Constitution of Pure Organic Explosive Compounds" is the subject of a review by Lothrop & Handrick (Ref 1). Price (Ref 2) has reviewed the "Dependence of Damage Effects upon Detonation Parameters of Organic High Explosives", and Martin & Yallop (Ref 3) have reported on "The Correlation of Explosive Power with Molecular Structure"
Refs: 1) W.C. Lothrop & G.R. Handrick, ChemRevs **44,** 419-45 (1949) 2) D. Price, Chem Revs **59,** 801-25 (1959) 3) A.R. Martin & H.J. Yallop, JApplChem **9,** 310-15 (1959)

Corrosion. A process of gradual disintegration or decomposition of a chem nature. The most common example of corrosion is the rusting of iron in air or acidic water. Corrosion of metals is electrochemical in nature. Extensive info is available in the literature regarding measures that have been and can be taken to minimize or prevent various forms of corrosion which occur in industries. It is important to recognize the various forms of corrosion and to effectively apply the available info in order to overcome corrosion problems. See the Refs given below

Corrosion of ammunition, weapons & other items of ordnance is prevented by some type of treatment (such as painting, plating or other surface treatment) of many components, in addn to proper packing & sealing to protect the components. The usual rust preventative & lubricating oil preservative compds are not used on ammo components because they might prevent proper functioning, and also because ammo components contain many inaccesible cavities from which these materials cannot easily be removed (Ref 12a)

The common surface treatments for ammo are plating, dipping & coating. Small components of steel are usually plated with Zn or Cd. Various chromate dippings (such as "Cronak," "Irridite" & "Yellow ano-zinc") may also be used, especially when a plating is applied first. The outside of larger components (such as shells, bombs, rockets & mines) are painted for protection as well as for identification. The inside of such components may be coated with acidproof black paint. Certain small components like detonator cups may be coated with lacquer to prevent corrosion. No one coating is satisfactory for all components, as some may require special coatings(Ref 12a). See also other Refs given below
Refs: 1) P. Regnauld, MAF **13,** 241-65 (1934) & CA **28,** 6415(1934) (Includes 3 methods of measuring corrosion) 2) H.E. Searle & F.L. LaQue, ProcAmSocTesting Materials No **12,** 12 pp (1935) & CA **29,** 5402 (1935) (Corrosion testing methods) 3) F.N.Speller, "Corrosion. Causes and Prevention", McGraw-Hill, NY (1935), 687 pp 4) O. Bauer, O, Kröhnke & G. Masing, "Die Korrosion metallischer Werkstoffe", Hirzel, Leipzig, 3 volumes (1936) (Reproduced by J.W. Edwards, Ann Arbor, Mich, 1944) 5) R.J. McKay & R. Worthington, "Corrosion Resistance of Metals and Alloys", Reinhold, NY (1936) (ACS Monograph No 71) 6) J. Lucas, MAF **16,** 703-32 (1937) & CA **33,** 1646 (1939) (Describes various corrosion test methods) 7) A. Foulon, Nitrocellulose **9,** 44-46 (1938) (Corrosion problem in the expl industry) 8) Thorpe **3** (1939), 366-98 (Corrosion of metals and protective measures) 9) J.C. Hudson, "The Corrosion of Iron and Steel," Chapman & Hall, London (1940) 10) F. Seitz, "The Physics of Metals," McGraw-Hill, NY (1943) 11) G. Schikorr, "Die Zersetzungserscheinungen der Metalle," Barth, Leipzig (1943) (Reproduced by J.W. Edwards, AnnArbor Mich) 12) Hackh's (1944), 228 12a) Ohart (1946), 13-15. 13) H.H. Uhlig, Edit, "Corrosion Handbook" Wiley, NY (1948) 14) R.D. Misch et al, "Bibliographic Survey of Corrosion", NatlAssocCorrosion-Engrs, Bremen, Ga(1948) 15) N. Kaplan & R.J. Andrus, IEC **40,** 1946-47 (1948) (Corrosion of metals in acids) 16) Anon, ChemEngrg **55,** 233 (Feb 1948); 225 (March 1948) & 219 (April 1948) (The Corrosion Forum) 17) ASTM Standards **1949.** Part 1, p 865, Test **A224-46** (Recommended Practice for Conducting Plant Corrosion Tests) Part **1**, p 874, Test **A262-44T** ("Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels"); Part 1, p 878, Test **A279-44T** (Total Immersion Corrosion Test for Stainless Steels) 18) Kirk & Othmer **4** (1949)

487-529; "Corrosion," W.D. Robertson 18a) H. Ficheroulle & A. Kovache, MP **32**, 377-81 (1950) & CA **47**, 9014 (1953) (Corrosion of guns by primer compns) 19) F.N. Speller, "Corrosion - Causes and Prevention," McGraw-Hill, NY (1951), 688pp 20) E. Rabald, "Corrosion Guide" Elsevier Press, Houston, Texas (1951), 629 pp 21) K. Tator, ChemEngrg **59**, No 12, 144-90 (1952) (Symposium: How Protective Coatings Fight Corrosion) 22) F.A. Champion, "Corrosion Testing Procedures", Wiley & Sons, NY (1952), 369pp 23) J. Szanto, Ordnance **38**, 526-28 (1953) (Volatile corrosion inhibitors improve military packaging) 24) A.R. Black & A. Wachter, Ordnance **37**, 1052-54 (1953) (Defeating corrosion) 25) M.F. Mueller & A.D. Laehder, ChemEngrg **60**, 193-95 (October 1953) 26) E.A. Stockbower, Ordnance **39**, 502-05 (1954) (Preventing corrosion of military equipment) 27) F. Tödt, "Korrosion und Korrosionsschutz", W.de Gruyter, Berlin (1955), 1102pp 28) J.D. Clark & M.A. Walsh, TransNYAcadSci **17**, 279-88 (1955) & CA **50**, 147 (1956) (Corrosivity of fuming HNO_3 used as a rocket fuel) 29) J.L. English et al, Corrosion **12**, No 6, TechCommActivities, 65-68 (1956) & CA **50**, 9988 (1956) (Corrosion by HNO_3: A progress rept by NACE Task Group T-SA-5) 30) Anon, "Ordnance Materials Handbook Corrosion and Corrosion Protection of Metals", Ordnance Corps Pamphlet **ORDP 20-311** (1957) 31) L.L. Shreir, "Corrosion," Wiley & Sons, NY (1963) 32) Perry (1963), Sectn 23 33) W.M. Gauntt & J. Derbyshire, Ordnance **48**, 656-59 (1964) (Corrosion of space vehicles)

Cortex. A Fr detonating cord (cordeau détonant) consisting of PETN (Core) in a fabric covering. Its external diam is 4.8 mm & detonation velocity 5990 to 6410 m/sec. See also Cord, Detonating
Ref: M. Dutour, MAF **24**, 578 & 583 (1950)

Corteza de seguridad. Spanish for "sheath" and **cartucho con corteza** is a "sheathed cartridge". See under COMMERCIAL AND INDUSTRIAL EXPLOSIVES in this volume

Corvus. A US Navy air-to-surface missile with a range of about 100 miles. It is powered by a prepackaged liq propellant motor. The corvus was developed for use by carrier aircraft as a stand-off weapon against heavily defended strong points
Ref: OrdTechTerm (1962), 85

Cosilit. A German coal-mining expl of the Carbonite type (See Vol 2, p C61-R). It consists of NG 30, KNO_3 22.3, wood or corn-meal 40.5 & Na chloride 7.2%
Refs: *1)* Marshall, Dict (1920), 25-26 2) Ullmann **4** (1926), 784

Cosmotron. An apparatus similar in purpose to the Bevatron (See Vol 2, p B109-R) but on a smaller scale
Ref: OrdTechTerm (1962), 85

Coton azotique. One of the French names for Nitrocellulose. See CA_1 and CA_2 in Vol 2, p C1-R

Coton Collodion. One of the French names for Collodion Cotton. See Collodion Cotton or Pyroxylin in Vol 2, p C103-R

Coton poudre or Fulmicoton. French names for NC, corresponding to endecanitrocellulose, $C_{24}H_{29}O_9(ONO_2)_{11}$. See Guncotton in Vol. 2, p C106-R

Coton, Poudre au. French name for Tonite, a blasting expl based on Guncotton mixed with Ba nitrate
Ref: Daniel (1902), 171 & 769

Coton pyrique. French designation for Pyrocellulose or Pyrocotton. See Vol 2, p C105-L

Cotter Powder. An expl mixt consisting of $KClO_3$ 50% & realgar (As_2S_2) 50%
Refs: 1) Daniel (1902), 172 2) Giua, Trattato **6** (1) (1959), 399

COTTON

A plant of the genus Gossypium which pro-

duces the seed hairs of great economic importance known generally as cotton fibres. Cotton plants may be classified under two broad groups: the American or New World species and the Asiatic or Old World species. Under each of these groups hundreds of varieties have been reported. Since the number of ways in which the fibres from these numerous varieties differ from each other is almost infinite, rigid classifications of cotton fibres are impossible. Some chemical tests are used for establishing the type and the origin of cottons. One of these is the methylene blue absorption test, which gives a value of about 0.46 for American cotton, 0.69 for Egyptian and 0.84 for Sea Island. The five most important types of cotton in order of quality and spinning values are: Sea Island, Egyptian, American upland long-staple, American upland short-staple and Asiatic. Commercial classification is arbitrarily based upon appearance (grade), staple length (the length of a typical portion of the fibre measured under specific conditions of temperature and humidity) and character (physical properties). American cotton staple lengths vary from 7/8" to 1½"; Asiatic cottons have staple lengths ranging from 3/8" to 3/4"

The chem compn of a typical cotton fibre is as follows: Cellulose 86.1, protein 1.2, water 8.3, pectins 1.1, ash 1.1, wax 0.6, sugars 0.3, pigments - trace, others 1.3. Purified cotton is practically pure cellulose, while some cottons which have simply been dried contain as high as 96% cellulose. For chem prop of cellulose, see under Cellulose

The cotton fibres are removed from seeds by a process called "ginning". The recovered fibres are called "lint". The seeds, to which very short fibres called "linters" are still attached, are sent to a cotton-seed mill where the seeds and "linters" are separated from each other

Cotton "lint", the substance from which cloth is made, is too expensive for use in the explosive industry. It has also been found that these long fibres are much more difficult to nitrate than the shorter and less expensive "linters". Hence the purified "linters" constitute the principle source of cellulose for the manufacture of nitrocellulose explosives and plastics (See also under Cotton, Chemical)

The principal cotton-producing countries of the world are the US, India, Russia, China, Brazil and Egypt. For an excellent review of this subject see Ref 4

Refs: 1)H.B. Brown, "Cotton History, Species, Varieties, Morphology, Breeding, Culture, Diseases, Marketing and Uses", McGraw-Hill, NY (1938), 605 pp 2) G.R. Merrill, A.R. Macormac & H.R. Mauersberger, "American Cotton Handbook", AmerCottonHdbk Co, NY (1941), 1024 pp 3) Dorée (1947), pp 1ff & 105 4) Kirk & Othmer **4** (1949), 563-88 5) A.R. Macormac, &.R. Mauersberger & G.R. Merrill, "American Cotton Handbook", Textile Book, PubgCo, NY (1949), 940 pp 6) S.V. Shmelev, "Khimicheskaya Tekhnologiya Khlopka" (Chemical Technology of Cotton), GosNauch-TekhIzdatLegkoiProm, Moskva (1951), 328 pp 7) K. Word Jr, "The Chemistry and Chemical Technology of Cotton", Interscience (1955) 782 pp 8) H.B. Brown & J.O. Ware, "Cotton", McGraw-Hill, NY (1958), 500 pp 9) Encycl Britannica **6** (1963), 613-17. See also Refs under Cellulose, Vol 2, C97

Cotton, Chemical. The basic raw material from which NC and cellulose acetate are made. Chemical cotton is chemically purified cotton linters (See under). The raw linters contain impurities such as proteins, fats, waxes, pectins, sugars, inorganic salts and mechanically entrapped vegetation, dirt, wood etc , all of which must be removed

The raw linters are first air washed and mechanically cleaned and are then digested in dil solns of NaOH (1.0 to 10.0%) for 2 to 6 hrs at temps up to 168° (335°) F under closely controlled conditions of pH. After digesting and washing, the linters are subjected to multi-stage bleaching in dil hypochlorite solutions (2.5%) under closely controlled conditions. Rigid chem control is required in both the digestion and bleaching opns in order to prevent excessive formation of hydrocellulose and oxycellulose, both of which are undesirable. These impurities appear in the final product as "alkali-soluble cellulose". Final opns consist of treatment with dil sulfuric acid, washing and drying. The yield is about 75% by wt of the original linters

Chemical cotton is one of the purest forms of cellulose obtainable, containing 96-98% cellulose (dry basis)

Specifications for purified cotton linters are covered in US Spec MIL-C-206. The requirements are as follows: moisture, max 7%; alkali sol mat-

ter, max 5.0; ether extractive matter, max 0.4; ash, max 0.5; viscosity (Gd I) 3-9 poises; viscosity (Gd II) 10-36 poises; and lime, chlorides, sulfates, hypochlorites, max traces. The absorbency shall be satisfactory when tested acc ording to the special method prescribed
Refs: 1) Hayes (1938), 17 2) Anon, Chemical Cotton Flow Sheet of Hercules Powder Co, Chem & MetEngrg **48**, 92-94 (April 1941) 3) Dorée (1947), 4 4) Kirk & Othmer **3** (1949), 352-57 5) TM 9-1910 (1955), 127ff.See also Refs under Cellulose, Vol 2, C97

Cotton Linters. The short fibres left on cotton seeds after the first ginning. These fibres are removed from the seed by mechanical means at the cotton seed oil mills. Cotton linters are graded as follows, with respect to average fibre lengths, in mm: first cut 3.1, second cut 2.3, mill run 2.8 & hull fibre 1.9. First cuts and the longer grades of mill run linters are too expensive to be used in the manuf of expls. However, they are used for producing padding materials, medical cotton, surgical dressings & yarns. NC & cellulose acetate are made exclusively from the shorter mill run lengths and second cut linters. These linters must be carefully selected and then subjected to chem purification before they are suitable for use in the expls & plastics industries. The purified linters are called *Chemical Cotton.* From 100-180 lbs of second cut cotton linters are obtd per ton of cotton seed
Refs: Kirk & Othmer **3** (1949), 352 ff

Cotton (or Cellulose), Copper Index. A number which represents the amt of Cu (calcd as grams Cu) reduced from the cupric to cuprous state in alk soln by 100g of dry cotton. It indicates any appreciable changes that have taken place in the cotton during the purification & bleaching processes. There are several methods for detg this value: Schwalbe, Schwalbe-Braidy, Knecht-Thompson, and Hägglund Methods. The Copper Index Number for untreated cotton, as detd by the Schwalbe-Braidy Method, is as low as 0.04g, while for cotton treated with acid the value is as high as 2.34 g (Ref 1)

The formula used for caluclating this number is

$$\text{Copper Number} = \frac{6.36x \text{ cc } KMnO_4 \times N}{W}$$

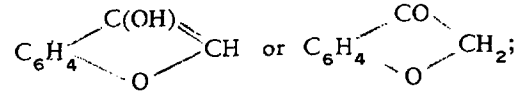
where N = normality of $KMnO_4$ and W = Wt of sample in grams, after deduction of wt for moisture & ash (Ref 2)
Refs: 1) Dorée (1947), pp 26-36 2) Std Methods-ChemAnalysis **2B** (1963), 1772 & 1828

Cottrell, Frederick G. (1877-1948). American scientist, inventor of an electrostatic precipitator, now known as *Cottrell Precipitator,* for smoke, dust & fumes. Among other inventions are the pebble bed furnace, boiling point apparatus & the Cottrell-Daniels process for fixation of atmospheric nitrogen. Cottrell was Director of US Bureau of Mines; Director of the Fixed Nitrogen Research Laboratory, and founder of the Research Corporation, a non-profit organization
Refs: 1) W.R. Whitney & F.G. Cottrell, Willard Gibbs Medal Award, Presentation & Medal Address, IEC **12**, 697-700 (1920) 2)Anon, Biography with portrait of F.G.Cottrell, Elec World **81**, 141 (1923) 3)Hackh's (1944), 229 4)F.Cameron, "Cottrell-Samaritan of Science", Doubleday & Co, NY (1952), 403pp (Biography of a great chemist)

Cottrell-Paterson Equation of State. An equation of state, applicable to gases at densities near that of the solids and to temps far above the critical, is derived by Cottrell & Paterson (Ref 1). It is shown that this equation is likely to hold in the range of density & temperature characteristics of the detonation wave in condensed expls. The hydrodynamic equations of deton are developed on the basis of the equation of state. They were applied to PETN and the theory predictions were shown to agree with observations. Murgai (Ref 2) extended the application of the equations to oxygen-deficient expls, specifically TNT
Refs: 1)T.L.Cottrell & S.Paterson, ProcRoySoc **213A**, 214-25 (1952) & CA **46**, 10735 (1952) 2)M.P.Murgai, ProcNatlInstSci India **19**, 541-45 (1953) & CA **48**, 3691 (1954) 3)Cook (1958), 66

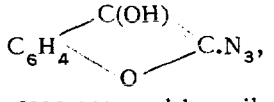
Coumaranone and Derivatives

Coumaranone or 3(2H)-Benzofuranone [called Cumaranon-(3); 3-Oxo-cumaran or 3-Oxy-cumaron in Ger]



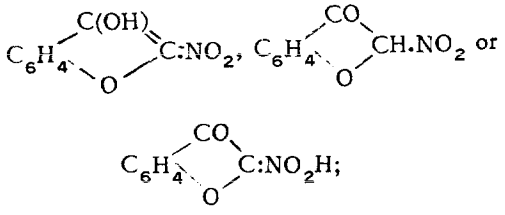
mw 134.13, O 23.86%; ndls (from alc), having an odor like hyacinth when pure; mp 101-02°, bp 153° at 16 mm press; readily sol in most solvs, except diffc sol in w & petr eth; other props & methods of prepn are given in Beil
Ref: Beil **17**, 118, (59) & [126]

2-Azido-3-coumaranone or 2-Azide - 3 - hydroxycoumarone (called 2-Azido-3-oxycumaron or 2-Azido-3-oxocumaran in Ger),

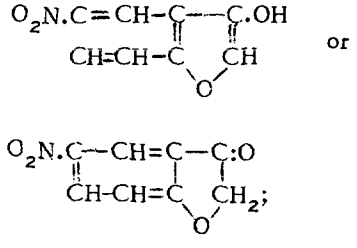


mw 175.15, N23.99%; yel-brn oil which decomposes explosively ca 160°. It can be prepd from 2-bromo-3-cumaranone and NaN_3 in acetone
Refs: 1)Beil **17**, (127) 2)K.Fries & S.Saftien Ber **59**, 1250 (1926)

Mononitrocoumaranone, $C_8H_5NO_4$; mw 179.13, N 7.82% Two isomers are described in the literature:
2- Nitrocoumaranone,

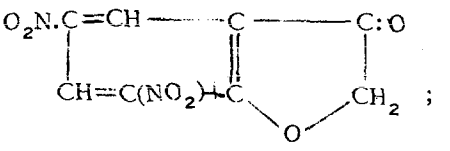


obtained as the K salt; citron-yel ndls, by treating 3-bromo-2-nitrocoumaron with diisobutylamine and heating the resulting 2-nitro-3-diisobutylaminocoumarone with alc K (Ref 1)
5-Nitrocoumaranone,



pltlts (from benz), mp 152°; vol with steam; mod sol in alc, sl sol in benz, insol in alc; was prepd by treating the caustic Na soln of 4-Nitro-2-chloroacetylphenol with aq acetone (Ref 2)
Refs: 1) Beil **17**, 119 & (60) 2) Beil **17**, [127]

5,7 - Dinitro-3-coumaranone,



mw 224.13,N12.50%;
light tan ndls (from AcOH-H_2O), mp 183-84°; was prepd by refluxing for 12 hrs a soln of 5,7-dinitro-3-coumaranone-2-carboxylate in 50% AcOH-HCl contg a catalytic amt of $ZnCl_2$, filtering hot & cooling to ppt the product (Ref 2)
Refs: 1) Beil - not found 2) P.D. Bartlett & E.N. Trachtenberg, JACS **80**, 5808-12 (1958) & CA **53**, 11363 (1959)

Trinitrocoumaranones, $C_8H_3N_3O_8$, were not found in Beil or in CA thru 1961

See also 2, 3-Benzofuran or Coumarone in Vol 2, p B67-R

Coumaric Acid and Derivatives

Coumaric Acid, Coumarinic Acid or Hydroxycinnamic Acid [called Cumarsäure, Cumarinsäure, (Oxyphenyl)-acrylsäure or Oxy-zimtsäure in Ger], $C_9H_8O_3$; mw 164.15, O 29.24%. This compd exists as ortho-, meta - & para - derivs. The *ortho* deriv is known in both trans & cis forms:

$HO.C_6H_4.CH$
‖
$HC.COOH$ (trans form); cryst (from w or dil alc) + 1 mol H_2O, mp (becomes anhyd after heating 8 hrs at 120°) 208°; mod sol in alc; sl sol in cold w; v sl sol in eth; insol in chlf & CS_2; forms numerous salts; other props & methods of prepn are given in Beil (Ref 1)

$HC.C_6H_4.OH$
‖
$HC.COOH$ (cis form), not yet isolated in the free state, being resolved into coumarin & water at the moment of liberation from its salts. A number of salts & other derivs are known and described in the literature (Refs 2 & 5)

m-Coumaric Acid, prisms (from w), mp 191°; sol in hot w, alc, eth & benz; diffc sol in cold w; other props & methods of prepn are given in Beil (Ref 3)

p-Coumaric Acid, col crysts (from concd H_2O soln) mp 210-15° (dec); sol in hot w; sl sol in hot alc & eth; diffc sol in cold w or benz; insol in petr eth; other props & methods of prepn are given in

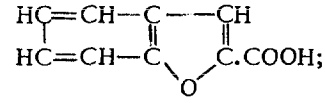
Beil (Ref 4)
Refs: 1) Beil **10**, 288, (122) & [174] 2) Beil **10**, 291 & (125) 3) Beil **10**, 294, (128) & [178] 4) Beil **10**, 297, (129) & (178) 5) A. Clayton, JChemSoc **97II**, 2102-10 (1910)

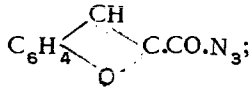
Azidocoumaric Acid, $C_9H_7N_3O_3$, not found in Beil or in CA thru 1961

Mononitrocoumaric Acid, $C_9H_7NO_5$; mw 209.15, N 6.70%. Several isomers are known: *3-Nitrocoumaric Acid*, $HO.C_6H_3(NO_2).CH{=}HC.COOH$; yel crysts (from alc), mp 241-42° (dec)
3-Nitrocoumarinic Acid, $HC.C_6H_3(NO_2).OH{=}HC.COOH$; yel prisms (from alc), mp ca 150°, by rapid heating, losing water; forms colored cryst salts
5-Nitrocoumaric Acid, ndls, becoming yel in the light, mp 247° (dec); was obtd by heating 5-nitro-β-2-hydroxyhydrocinnamic acid-β-sulfonate with 20% KOH soln; *5-Nitro-coumarinic Acid; 2-Nitro-m-coumaric Acid*, wh ndls (from w), mp 218°; *4-Nitro-m-coumaric Acid*, golden yel ndls (from alc), mp 248°; *5-Nitro-m-coumaric Acid*, crysts, having a taste not sweet, mp dec; *6-Nitro-m-coumaric Acid*, microsc ndls (from alc), mp 216° and *3-Nitro-p-coumaric Acid*, col ndls (from w), yel ndls (from alc), mp 223° (dec); was prepd by heating 3-nitro-r-methoxycinnamic acid with an acetic acid soln of HBr for 3 hrs at 100°
Ref: Beil **10**, 294-96, 299, (127,132) & [177]

Dinitrocoumaric Acid, $C_9H_6N_2O_7$; mw 254.15, N 11.02%. Only one isomer is described in the literature: *3,5-Dinitro-coumarinic Acid*, $HC.C_6H_2(NO_2)_2.OH{=}HC.COOH$; yel ndls (from eth), mp 153-55° (dec); was prepd by heating 6, 8-dinitrocoumarin with NaOH. It is rapidly converted to 6, 8-dinitrocoumarin when its soln in w or alc is heated (Refs)
Refs: 1) Beil **10**, (128) & [178] 2) A. Clayton, JChemSoc **97I**, 1407(1910) 3) B.B. Dey & K.K. Row, JChemSoc **125I**, 561(1924)
Trinitrocoumaric Acids, $C_9H_5N_3O_9$, were not found in Beil or in CA thru 1961

Coumarilic Acid and Derivatives

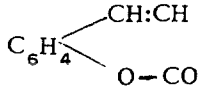
Coumarilic Acid or 2-Benzofurancarboxylic Acid [called Cumarilsäure or Cumaron-carbonsäure - (2) in Ger],  ; mw 162.14, O 29.60%; ndls (from w), mp 190-93° (subl), can be distilled w/o decompn at 310-15°; sol in alc & hot w; sl sol in chlf & CS_2; was prepd by heating 3-chlorocoumarin or 3-bromo-coumarin with alc KOH and by other methods. Coumarilic Acid forms several cryst metallic salts
Ref: Beil **18**, 307 & (276)

Coumarilic Acid Azide or Coumaril Azide (Called Cumarilsäure-azid in Ger),  ; mw 203.15, N 20.69%; lfts (from dil alc), mp 109°; explodes mildly when heated on a Pt block; sol in alc, eth & benz; was prepd by treating an aq soln of coumaril hydrazide with a calcd amt of $NaNO_2$, under cooling, and addg of acetic acid
Refs: 1) Beil **18**, 308 2) R. Stoermer & G. Calov, Ber **34**, 774 (1901)

Mononitrocoumarilic Acid, $C_9H_5NO_5$; mw 207.14, N 6.76%. Several isomers are described in the literature: *4-Nitro -*, mp 243-44°(Ref 6); *5-Nitro -*, col ndles (from dil AcOH), mp 274-75° (dec) (Refs 1,2,3,4 & 6); *6-Nitro*, yel powd, mp 246-47° (dec) (Ref 5); and *7-Nitro -* mp 256-57 (Refs 4 & 6. Their methods of prepn & other props are given in the Refs
Refs: 1) Beil **18**, [276] 2) B.B. Dey & K.K. Row, JChemSoc **49**, 564 (1923) & CA **18**, 679 (1924) 3) H. Erlenmeyer et al, Helv **31**, 76 (1948) & CA **42**, 4167 (1948) 4) S. Tanaka, JChemSocJapan, PureChemSect **73**, 284 (1952) & CA **47**, 9957 (1953) 5) P. Rumpf & C. Gansser, Helv **37**, 435 (1954) & CA **49**, 6216 (1955) 6) R. Andrisano et al, Gazz **86**, 1267(1956) & CA **52**, 16032 (1958) (UV spectra)
Dinitro, $C_9H_4N_2O_7$, and *Trinitro*, $C_9H_3N_3O_9$, acids of coumarilic Acid were not found in Beil or in CA thru 1961

Coumariloyl Peroxide. See Dicoumariloyl Peroxide

Coumarin and Derivatives

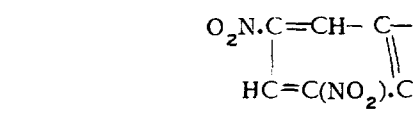
Coumarin or 1, 2 - Benzopyrone [called 2-Oxo-1,2-chromen; 5,6 - Benzo-pyron - (2); and Cumarin in Ger],  ; mw 146.14, O 21.90%; ndls (from

alc), having a characteristic odor; mp 70-71°, bp 391° at 750 mm, d 0.935 at 20°; sol in alc, eth, trichloroethylene & chlf; insol in w; prepd by extracting tonka beans with 80% alc, or by heating Na salicyl-aldehyde with acetic aldehyde. Other methods of prepn are given in Beil (Ref 1)

On nitration, it forms *6-Nitrocoumarin; 8-Nitrocoumarin; 3, 6- Dinitrocoumarin; and 6, 8-Dinitrocoumarin* (Ref 2). Some of the nitro derivs might find application in the expls industry

Refs: 1) Beil **17**, 328, (170) & [356] 2) Beil **17**, 333, (171) & [360-61) 3) Hackh's (1944) 230 4) Kirk & Othmer **4** (1949), 590-93 5) Sax (1957) 510 (Toxicity & props)

3, 6, 8 - Trinitrocoumarin,



; mw 281.13, N 14.95%; mp 158-59°. This compd appears to have been first prepd by Mangini & Passerini (Ref 2) who detd its chromophoric props & UV spectra. Its method of prepn is not reported

Refs: 1) Beil - not found 2) A. Mangini & R. Passerini, Gazz **87**, 252, 273 (1957) & CA **52**, 10716-17 (1958)

Coumarinic Acid. See Coumaric Acid

Coumarone. Same as 2, 3-Benzofuran. See Vol 2, p B67-R

Coumarone - Indene Resins. These are thermoplastic resins of relatively low mw, varying from fairly viscous liqs to hard resins. Their color varies from pale yel to nearly black. They are obtd by heating mixts of coumarone & indene with H_2SO_4 so as to cause polymerization. Coumarone -indene resins are one of the oldest types of synthetic resins, having been produced in Germany as early as 1900. The manuf & props are described in Ref 2

Coumarone-indene resins are sold under various trade names: *Cumar, Neville, Nevindene, Paradene & Picoumaron.* Numerous applications in industry include uses in plastics, protective coatings, adhesives, lacquers, and as plasticizers for cellulose esters & vinyl resins

Refs: 1) Beil **17**, 56 & [57] 2) Kirk & Othmer **4** (1949), 594-99 3) CondChemDict (1956), 310

Counter Chronograph. See Item D, under Chronographs and Detectors Currently Used for Velocity Measurements of Prejectiles, in this volume, p C309-R

Counterinsurgency Warfare. Title of a book by D. Galula, including theory & practice, published by Praeger, NY (1964)

Coup complet (Fr).Complete round of ammunition

Coup de poussière (Fr).Dust explosion

Courteille Powder. A kind of BkPdr invented in 1875. It contained K or Na nitrate, peat, oily substances & metallic sulfates

Ref: Daniel (1902), 172

COV. Concentrated oil of vitriol or sulfuric acid of 95-96% strength, by weight, as distinguished from NOV (Nordhausen oil of vitriol) or oleum which is a soln of SO_3 in anhydrous sulfuric acid

Refs: 1) Marshall 1 (1917), 96 2) Hackh's (1944), 230 & 593

Covolume (Covolumen in Ger).This is a term applied in certain equations of state to correct the vol of gas for *the effect* of the vol of the molecules. This term is not the molecular vol itself (Ref 12)

In *Van der Waal's equation of state,*

$$RT = (p + a/v^2) - (v - b),$$

it is the quantity b which corresponds to four times the square root of the space occupied by the molecules themselves (Ref 6). The classical equation of state used by ballisticians and known as the *Noble - Abel equation of state* is written

$$P(V - \alpha W) = \frac{WR_0T}{M}$$

where R_0 = the universal gas constant
W = weight of propInt
M = molecular wt of combstn gases
T = absolute temp and

where the term α is the covolume (Ref 16). It may be thought of as the smallest vol into which a unit wt of gas can be compressed. The significance of the covolume correction can be illustrated by the following examples. At a temp of 2730° K & a pressure of 68 atm, the conditions typical of rocket ballistics, the value $(V - \alpha)$ does not differ significantly from V. It is customary, therefore, in rocket ballistics to ignore the covolume correction. On the

other hand, in gun ballistics the pressures are considerably higher (3000 atm) and the covolume correction must be made (Ref 16). Also in the equation of state applied to the study of condensed explosives, covolume corrections must be used (Ref 14). The covolume when properly evaluated is found to be no longer a constant value, but is a variable with a value still in the neighborhood of Noble-Abel's α (Ref 16)

In studying the gaseous products of expln, some investigators use a covolume value assumed to be 1/1000 th of the specific vol (Refs 6, 12, 15 & 16). Some values of the covolumes of expls & proplnts are reported in the literature (Refs 3,7,8,9,10,11 & 13)

See also Equations of State under DETONATIONS AND EXPLOSIONS

Refs: 1) Venin, Burlot & Lécorché (1932), 53-55 2) Stettbacher (1933), 71 3) Pepin Lehalleur (1935) 40 & 50 4) Beyling & Drekopf (1936), 53 5) Hayes (1938), 50 6) Hackh's (1944), 230 & 890 7) H. Muraour, "Poudres et Explosifs", Presses Univs de France, Paris (1947), 60-92 8) H. Muraour & G. Aunis, MAF **22**, 133-70 (1948) (Covolumes of colloidal propellants) 9) Corner, Ballistics (1950) 100ff 10) SACMS, Ballistics (1951), 18 11) Taylor (1952), 87ff 12) Van Nostrand Chemists' Dict (1953), 186-87 13) P. Tavernier, MP **37**, 270-304 (1955) & CA **51**, 1610 (1957) (Imperfect character of the explosion gases & covolume of proplnts) 14) Cook (1958), 63ff 15) Anon, "Sources of Energy", ORDP 20-106 (1960), 2-2 16) A.M. Ball "Solid Propellants," ORDP 20-175 (1961), 3

COX. Code name for 1, 5-Dinitroxy-trimethylene -2, 4 - dinitramine, to be described under Dihydroxy-trimethylenediamine and Derivatives

Coyote Tunnels. These are usually horizontal tunnels about 4 x 5 ft in cross section which is just about large enough to provide working space. The simplest coyote layout consists of a main stem perpendicular to the hillside or quarry face with a single wing or crosscut at the back end driven at 90° to the right or left. A great many modifications of this arrangement are used, depending on local requirements. Tunnel driving is done by any one of the conventional methods applicable to small headings

The coyote tunnel method of blasting is used in some quarries & heavy sidehill cuts where conventional drilling methods are impractical because of high costs. In this method, a number of relatively large concentrated charges are located in one or more small tunnels under the rock formation. The best expl for this purpose is considered by Dupont to be "Nitramite" FR (free running). More details of the description of coyote tunnels and of the expls used are given in the Ref

Ref: Blaster's Hdb (1958), pp 215-20 & 390-92 and (1966), pp 240-44 & 343-44

CP_1 and CP_2. French abbreviations for "Coton poudre 1" (eth-alc insol NC, contg 13% N) and "Coton poudre 2" (eth-alc sol NC, cong 12% N). See also under Collodion Cotton or Pyroxylin in Vol 2, p C103-R

CP_2SD. French abbreviation for "Coton poudre 2 sans dissolvant" (NC contg 11.6-11.7% N, solventless). This NC is suitable for prepg solventless proplnts by gelatinizing the NC with NG without the use of a volatile solvent. See Vol 2, p 105-L

CP_3. French abbreviation for "Coton poudre 3" (NC contg to ca 11.5% N). See under Collodion Cotton or Pyroxylin in Vol 2, p C105-L

Cp100. French designation for percentage of carbon in a compd or compn

CPN-112C. A compression molded proplnt developed by Phillips Petroleum Co for use in rockets. Its compn & props are given in Conf "Prepllant Manual" SPIA/M2 (1962), Unit No 489

CPN-115A. Same type as above, but compn is different. Unit No 490

CPN-123. Same type as above, but compn is different. Unit No 491

CPN-127A. An extruded composite proplnt developed by Phillips Petroleum Co for use in rockets & Jets units. Its compn & props are given in Conf "Propellant Manual," SPIA/M2 (1962), Unit No 542

CPN-135. Same type as above, but compn is different. Unit No 543

"C" Process (Croning Process of Precision Casting). During WWII, Croning of Germany developed a low cost process of manufg molds and cores for casting objects having intricate contours. This method made possible the production of precision cast ammo components in larger sizes than was formerly considered practicable
Refs: 1) J. Croning, GerPatApplication 48679 (1944) 2) W.W. McCulloch, FIAT FR-1168 (Info obtained on interrogation of J. Croning in 1947) 3) Anon, "New Process", Industrial Bull, A.D. Little, Inc, Cambridge, Mass (1950) 4) PB Report 105131, "Plastic Bonded Shell Molding Simplified" US OffTechServices, Washington 25, DC (1951) 5) IR-7734, "Present Status of the Shell-Mold or "C" Process of Precision Casting of Metals", US OTS (1952)

CQE. A cast double-base propellant developed at ABL for rocket motors. Its compn & props are described in Conf "Propellant Manual", SPIA/M2 (1962), Unit No 633

Cracking. A process of decomposing petroleum or heavy petroleum fractions by heat, with or without catalysis, and thereby producing lower-boiling materials that are useful as motor fuels, domestic fuel oil, and other needed products

The term is also applied more generally to thermal decompn processes; thus ammonia may be cracked to give N_2 & H_2; and natural gas hydrocarbons (such as methane, CH_4) are cracked into C & H_2 or into other hydrocarbons
Refs: 1) Davis (1943), 129 & 224 2) Hackh's (1944), 231 3) Ullmann **10** (1958), 476-83 (Cracking of oils to Reichgasen) 4) CondChemDict (1961), 312

Cracking Gas. This is produced when heavy petroleum is cracked to produce gasoline. Cracking gas consists, in a large part, of olefins particularly ethylene & propylene. It is being used more and more for the manuf of glycol & glycerin which are nitrated to produce expls such as NGc, NG, Ethanolamine Nitrates (See under Aminoethanol in Vol 1, p A200) and Pentryl (See in Vol 2, Table, p B286). It is also used for the manuf of Mustard Gas (See Vol 2, p C168-L)
Ref: Davis (1943), 199

Crack-Preventing Additives to Cast Explosives. See Anticracking Additives to Cast Explosives, Vol 1, p A461 to A462

Cradle, Bomb. A rigid wood and/or metal frame structure, designed with a concave load bearing surface, to prevent movement of a bomb during handling. It may be carried manually or be transported by attachment to a vehicle. See also BOMBS, Vol 2, p B225-R and Bombs, Packing, Storage, Handling, etc, Vol 2, p B237-R
Ref: OrdTechTerm (1962), 87

Cradle, Cannon. An item designed to support a cannon and which allows it to recoil & counterrecoil. It also provides a means of securing the recuperator and recoil cylinders or the recoil piston rod. It has facilities for attaching it to a mount or carriage of a gun or howitzer. See also CANNON, Vol 2, p 26-L
Ref: OrdTechTerm (1962), 87

Crankcase Explosions in Oil Engines. These result from ignition of the medium which is normally an aerosol of lubricating oil, but whose formation may be accompanied by cracking & oxidation of the oil to form gases more hazardous than the mist itself. This subject is discussed in some detail in the Refs listed
Refs: 1) F.V. Cook, Petroleum Processing **5,** 1304-06 (1950) & CA **45,** 2669-70 (1951) (Crankcase explosions, causes & remedies) 2) J.H. Burgoyne, TranInstMarineEngrs (London) **67,** 255-70 (1955) & CA **49,** 16412-13 (1955) (Crankcase explns in marine engines) 3) H.D. Taylor, J ImpCollChemEngrSoc **9,** 82-95 (1955) & CA **50,** 12481 (1956) (Crankcase explns in oil engines)

Cranz, Carl Julius, Dr - Ing (1858-1945). A Ger ballistician & mathematician; an author of a comprehensive book in four volumes, entitled "Lehrbuch der Ballistik" (Refs 1 & 4) and of numerous papers on interior & exterior ballistics. He was responsi-

ble for designing many devices used in ballistics, such as Apparatus for Photographic Registration of Recoil (Ref 1, Vol 3, p 244), Ballistic Cinematographs (Ref 1, Vol 3, pp 350 & 356), Cranz & Schardin Spark Cinematograph (Ref 2), and others

A brief biography was published in 1943 (Ref 3) on his 85th birthday. Nothing is found in Chem Absts from 1943-1961, but the date of his death is found in Ref 4

Refs: 1) C. Cranz, "Lehrbuch der Ballistik, Springer, Berlin **1** (1925), **2** (1926), **3** (1927) & Ergänzungsband (1936) 2) C. Cranz & H. Schardin, ZPhysik **56** 147-49 (1929 3) Anon, "Geheimrat Cranz 85 Jahre," SS **38** 18(1943) 4) "Der Neue Brockhaus" F.A. Brockhaus, Wiesbaden, Vol **1** (1958), p 482-R

Crater, Cratering and Cratering Effect. A crater is a hole in the ground caused by an explosion. Craters can be produced by any expl chge (such as a demolition chge) and by any projectile (bomb, shell or rocket) exploding on the ground or slightly above it. In addn to the size of the expl chge, other determining factors in crater formation are the type of ground and the type of expl. An explosion on the surface of the ground produces a shallow crater. If an expl is buried deeply in the ground, the pressure of the gases on expln will be exerted entirely on the earth surrounding the charge and will cause effects for a measurable distance, known as *radius of rupture* (Ref 1)

If the expl is buried deeper in the earth than its radius of rupture, there is then formed a roughly circular depression, known as *camouflet*, considerably greater in vol than the expl chge producing it. Soft, low-density soil is lifted, scoured, and blown away more easily than firm or rocky earth formation. An expln in soft, swampy ground produces a relatively huge crater

Cratering effect may be considered as a measure of the demolition effects of General Purpose Bombs (See in Vol 2, pp B227-R to B228-L). These effects are sometimes detd by the following equation: 1) for commercial Dynamite

$$V = 0.4\ Q^{8/7}$$

and 2) for 500 & 1000-lb TNT loaded Bombs

$$V = 4.13W$$

where V = volume of crater in cubic feet (average soil), Q = weight of Dynamite in lbs, and W = weight of expl in lbs (Ref 2)

See Also Cratering Effect Tests

Refs: 1) Meyer (1943), 379-80 2) TM9-1910 (1955), 76-78 3) Cook (1958), 262-64 4) M.A. Cook, JApplPhys **30**, 725-35 (1959) (Mechanism of cratering in ultra-high velocity impact) 5) G.H.S. Jones et al, "Cratering by Ground Burst TNT at Suffield-Experimental Station Ralston Alberta", Suffield-Tech Paper **158** (October 1959) (Project D89-16- 01-12) 6) W.H. Townsend et al, "Mechanics of Crater Formation in Sand and Clay Produced by Underground Explosions", BRL Memo Rept **1381** (December 1961) (Proj 503-04-002) 7) G.H.S. Jones et al, "Some Comments on Cratering", Suffield Exptl Station-Special Publication **22** (April 1962) 8) L.K. Vortmann, "Craters from two Charges Buried in Vertical Array, " Sandia Corp Rept **SC-RR-64-1239** (Nov 1964) 9)Addnl Ref: C.S. Robinson, Explosions. Their Anatomy and Destructiveness", McGraw-Hill, NY (1944), 40-44

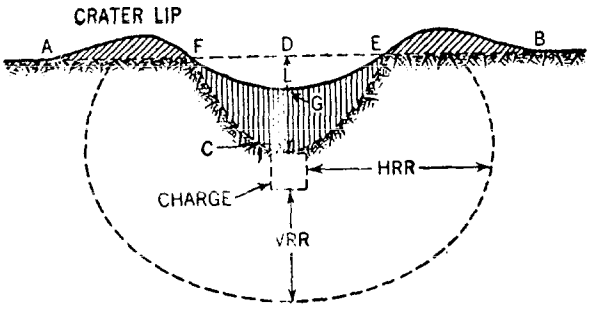
Cratering Charge or Cratering Explosive. This is usually a demolition charge used to blast craters in roads, or for other demolition purposes (Ref 8). During WWII, a cratering expl having the following compn (Ref 5) was used: AN 86.6, DNT 7.6, ferrosilicon 5.7 & red dye 0.1%. This expl is inexpensive and easily manufd. It is very insensitive to shock & initiation, very stable, has a high heaving force value, and a low rate of deton. The compn is somewhat hygroscopic and is packed, therefore, in hermetically sealed containers

A coml cratering charge, consisting of AN, TNT & Na nitrate in varying amounts was found, after tests at Ft Belvoir, Va and at PA, to be a very effective expl (Ref 4). This chge was standardized for military use by US Spec PA-PD-597, OAC-PD-63, and MIL-C-13879B (MU) for Charge, Cratering

Methods of analysis of a std cratering chge consisting of AN 65, Na nitrate 20 & TNT 15% are given in Refs 2,3,6 & 7

Refs: 1) US Corps of Engineers Rept **999** (1947) (Cratering with expls) 2) F.Pristera & S. Wachtell, PA ChemLabRept **120417** (1947) 3) N. Liszt, PA GenLabRept **54-HI-1718** (1954) 4) C. E. Jacobson & B. Zlotucha, "Development of an Explosive Charge for Production of Craters and Antitank Ditches", PATR **2263** (1955) 5) Anon, TM9-1910 (1955), 207 6) H.L. Herman, "Non-aqueous Titrimetric Analysis of Ammonium Nitrate, Sodium Nitrate, and TNT (Cratering Explosive)," PATR **2384** (1956) 7) N.S. Garman, PA GenLabRept **57-HI-109** (1957) 8) OrdTechTerm (1962), 65

Cratering Effect Tests. These tests are based on employment of the expl under actual operating conditions. Crater formation is illustrated in the following Fig:



Cross Section of Typical 2-lined Crater
HRR = horizontal radius of rupture; VRR = vertical radius of rupture.

C is the charge, and the ratio of surface diam of the crater to the depth of burial (FE/L) determines the nature of the crater. This ratio is 2 for a *common crater*, which is said to be 2-lined. A proper chge is one which produces a 2-lined crater; a 3-lined crater represents an overcharge; lower than 2, an undercharge. Based on experience, crater formation can be predicted by the formula

$$W = 1/2\ L^3 Z$$

where W = wt of expl in lbs; L = depth in ft to top of chge; and Z = a constant whose value depends on the type of crater to be formed and on the nature of the soil (Ref 4)

Meyer (Ref 4) has prepd a table for values of Z to be used with the above formula:

After W is calcd in the above formula, a further amt is added to it, depending on its value, as follows: for W = 25 lbs, add 100%; 25-100 lbs, add 50%; 100-250 lbs, add 25%; and 250 lbs, add 10%. The formula applies directly to TNT, but it can be converted for use with other expls, by employing appropriate factors

The French Test, called *Essai dans la terre* (test in the ground), may be considered as a modification of the Trauzl test. It is conducted as follows: A 100g cartridge of the test expl, provided with a No 8 detonator & fuse, is buried underground in uniform soil to a depth of 1.5 meters. The soil is tamped by using dry, sifted sand. After expln of the cartridge, the diameter (D) and height of cavity (H) are measured in centimeters. The volume (V) of the cavity is calcd from the formula:

$$V = 1/6\ \pi D^2 H$$
$$= 0.523\ D^2 H$$

The ratio of the value (V - v) to the original volume of the cartridge (v)

$$(V - v)/v$$

gives a value expressing the *puissance pratique du travail* (practical performance) (Ref 3). See also Refs 1 & 8

The German test for determining the efficiency of bombs & land mines on expln underground was conducted by burying an item, such as a 250 kg bomb, and exploding it. The vol of the resulting crater, measured in cubic meters, gave a value for the power of the expl chge (Ref 5). See also Refs 2,6 & 7

Kind of Soil	Camouflet 1-Lined	Under-charged 1½-Lined	Common 2-Lined	Over-charged 3-Lined	4-Lin
Light earth	0.010	0.024	0.054	0.162	0.36
Common earth	.012	.030	.066	.188	.44
Hard sand	.014	.038	.084	.252	.56
Hardpan or heavy clay	.016	.046	.100	.300	.67

The Cratering Effect Test is described in Italian, under the name *Saggio del Trauzl nella terra,* by Belgrano (Ref 9).See also Crater and Cratering Charge
Refs: 1) Vennin, Burlot & Lécorché (1932), 184-85 2) Stettbacher (1933), 386-87 (Minenwirkung) 3) Pepin Lehalleur (1935), 67-68 4) Meyer (1943), 379-80 5) O.W. Stickland et al, "General Summary of Explosives Plants" (German), OTS/USDC PB Rept **925** (1945), Appendix 7 6) G. Römer, "Report on Explosives" (Germany), OTS/USDC PB Rept **85160** (1946), 22 7) Stettbacher (1948), 145-48 (Auswurftrichter) 8) L. Médard, MAF **22** 607-08 (1948) 9) Belgrano (1952), 28-29 10) Addnl Ref: C.S. Robinson, "Explosions. Their Anatomy and Destructiveness", McGraw-Hill, NY (1944), 40-44

Crater, W. de C. (1894-1966) American chemist born in Succasunna, NJ. He attended Lafayette College and since 1917 has been associated with the Hercules Powder Company, Wilmington, Del. Crater is the author of many publications & patents on nitration processes, expl compds & proplnts
Refs: 1)IEC **40**, 1554 (1948) 2)C & EN **44**, 79 (11 July 1966)

Cratiti. Expls developed in 1898 by Alvisi of Italy. They consisted of Amm Perchlorate in admixtures with NG & NC
Refs Giua, Trattato **6** (1) (1959), 451

Crawford Bomb. This devise was developed in USA during WWII for the rapid evaluation of burning rates of proplnts at various temps and pressures. It is described and some results of tests are given in Refs
Refs: 1) B.L. Crawford, Jr et al, "Observations on the Burning of Double-Base Powders", NDRC Sec H, Div 3, Armor & Ordnance Rept **A-268** (OSRD **3544** (1944) 2) B.L. Crawford, Jr & C.M. Huggett "Direct Measurement of Burning Rates by an Electric Timing Method" NDRC Sec H, Div 3, Armor & Ordnance Rept **A-286** (OSRD **4009** (1944)

Crawshaw - Jones Apparatus. This apparatus is used to determine the properties & composition of the gaseous products developed on initiation & detonation of a confined chge of expl. The details of the apparatus and method of conducting tests are described in the Ref
Ref: C.E. Munroe & J.E. Tiffany, "Physical Testing of Explosives", US BurMinesBull **346** (1931), 95-99

Creep. The forward motion of fuze parts, relative to the missile caused by deceleration of the missile during flight. In some fuze designs, a *creep spring* is used to prevent creep action

It is also the sl unintentional movement of a vehicle, or the tendency thereto in a mechanism
Ref: OrdTechTerm (1962), 87

Creep Behavior of Propellant Material. Subject of a report by D.A. George, NBS Rept **5688,** 8th Prog Rept (1 Sept to 31 Oct 1947), Proj No TU2-2Q

Creep Force of a Projectile. This force results from the gradual retardation of a projectile due to air resistance encountered during its flight. It has the effect of causing objects within the fuze, which are free to move, to move slowly (creep) forward toward the nose of the fuze
Ref: Anon, "Ammunition Inspection Guide", TM9-1904 (1944), 322

Crémieux, Israel (1869-1932), Ingénieur Général. French Naval officer who specialized in artillery. He was the author of several publications, among them the book "Les Poudres de Marine. Balistique Intérieure Appliquée," Challamel Éditeur, Paris (1914)

Cremonita (Cremonite). The name applied to several expls patented in 1898 by U. Alvisi of Italy. They consisted of Amm perchlorate in admixture with one or more parts of PA, Amm picrate, Trinitrocresylate & Trinitrocresol. These Cheddite-type blasting expls were claimed to be more powerful than PA. Two examples of specific compns are:
1) Amm perchlorate 48.8 & Amm picrate 51.2%
2) Amm perchlorate 51 & PA 49%
Refs: 1) Daniel (1902), 172 2) Molina (1930), 362 3) CondChemDict (1942), 288 4) Mangini, Esplosivi (1947), 226 5) Giua, Trattato **6**(1) (1959) 401

Cremonita. A blasting expl consisting of AN 80 & cannel coal 20%
Ref: Pérez Ara (1945), 219

CRESOL AND DERIVATIVES

Cresol, Cresylic Acid or Methylphenol (called Kresol or Oxytoluol in Ger), $CH_3.C_6H_4.OH$; mw 108.13, O 14.79%. A coml coal tar product of the middle & heavy oil fractions. It contains a mixt of o-, m- & p- isomers which may be separated by fractional distillation into two fractions: o- and a mixt of m- & p-cresols. The props of cresol depend upon the compn & proportion of isomers in the mixt. See Kirk & Othmer (Ref 5) for more details
o-Cresol, wh crysts, phenol-like odor, poisonous, mp 30.8°, bp 191°, d 1.048 at 20°; sol in alc, eth & chlf; sl sol in w (Refs 1 & 7). Prepn & other props are given in Beil (Ref 1)
m-Cresol, col to yellowish liq, phenol-like odor, poisonous, fr p 11.7°, bp 203°, d 1.034 at 20°; sol in alc, eth & chlf; sl sol in w (Refs 2 & 7). Prepn & other props are given in Beil (Ref 2). This isomer serves as the starting material for the prepn of 2, 4, 6 - Trinitro-m-cresol (qv)
p-Cresol, crysts, phenol-like odor, poisonous, mp 34.6°, bp 202.3°; sol in alc, eth & chlf; sl sol in w (Refs 3 & 7). Prepn & other props are given in Beil (Ref 3)

Cresols are rapidly absorbed thru the skin causing severe burns (Ref 6). They are used as disinfectants, flotation agents; ink, paint & varnish removers; lube oil additives, organic intermediates, and for the production of synthetic resins (Ref 7)

Refs: 1) Beil **6,** 349, (169 & [322] 2) Beil **6,** 373, (183 & [344] 3) Beil **6** 389, (196) & [368] 4) Davis (1943), 129-30 & 169 5) Kirk & Othmer **4** (1949), 601-06 5a) Anon, ChemEngrg **59,** 212-15 (Nov 1952) (Cresylic Acid) 6) Sax (1957), 510-12 7) CondChemDict (1961), 313

Azidocresol, $C_7H_7N_3O$, and *Diazidocresol,* $C_7H_6N_6O$, not found in Beil or in CA thru 1961

Nitro Derivatives of Cresol

Mononitrocresols, $CH_3.C_6H_3(NO_2).OH$; mw 153.13, N 9.15%. The ten possible isomers are known & described in Beil. They are the 3, 4, 5 & 6 -nitro - o - cresols (Ref 1); 2, 4, 5 & 6 - nitro-m-cresols (Ref 2); and 2, 3- nitro-p-cresols (Ref 3). The derivs of m-cresols are the more important of these, because they are intermediates for the prepn of the expl compd Trinitro-m-cresol (qv)
Refs: 1) Beil **6,** 365, 366, (178) [338, 339] 2) Beil **6,** 385, 386, (191) & [359, 361] 3) Beil **6** 411, 412, (205, 206) & [387, 388]
Dinitrocresols (DNCrs), $CH_3.C_6H_2(NO_2)_2.OH$; mw 198.13, N 14.14%. The following isomers are described in the literature:
3, 4 (or 5, 6)-Dinitro-o-cresol, col crysts (from abs alc), mp 89.5°. Its prepn is given in Beil [Ref 1, p (180)]
3, 5 (or 4, 6)-Dinitro-o-cresol, yel prisms, mp 85.5 - 87°; sol in alc, eth & acet; sl sol in w & ligroin; can be prepd by nitration of 4,6-bromo-o-cresol and by other methods given in Beil (Ref 1). It forms numerous salts & addn compounds, some of which are expl. The **Lead Salt** $2(C_7H_5N_2O_5\ PbOH) + PbO$, orn-yel crysts (Ref 5) prepd by interaction of the Na salt with $Pb(NO_3)_2$; was proposed by Brün (Ref 7) as an ingredient of priming compns contg MF, Ba nitrate & an abrasive material. Rubinstein & Taylor (Ref 8) patented the use of the basic Pb salt 90 & $KClO_3$ 10% as the deflagrating compn for the bridge wire of an electric detonator. Fifteen to 25% of the Pb salt was also used in mixts with ground proplnts as an ignition compn for electric blasting caps (Ref 9). The **Ammonium Salt,** $C_7H_9N_3O_5$, is listed by Sax (Ref 14) as of mod toxicity and of the same fire & expln hazard as nitrates
The **Thalliumdiethyl Hydroxide Salt,** $CH_3.C_6H_2(NO_2)_2.O.Tl(C_2H_5)$, brn-red pltlts, mp-blackened at 215° and exploded with great violence at 219°; was prepd by Goddard (Ref 6). This compd is insol in CCl_4 & petr eth, gives a yel soln in acet and

orange in other solvs

A method for the routine estimation of 3, 5-Dinitro-o-cresol in urine is reported by Parker (Ref 12)

2, 4 (or 2, 6) - Dinitro-m-cresol, crysts (from abs alc), mp 100°; can be prepd from 2, 4-dinitro-3-aminotoluene by diazotization & boiling with w. Other methods of prepn & props are given in Beil (Ref 2)

2, 6 (or 2, 4)-Dinitro-m-cresol, crysts (from AcOH) mp 74°; may be prepd by nitrating 6-nitro-m-cresol with HNO_3-AcOH mixt in the cold, and by other methods [Ref 2, p (193)]

4, 6-Dinitro-m-cresol, yel crysts (from glacial AcOH or dil alc), mp 73-74°; prepd by heating 2,4,5-TNT with crystalline Na acetate, and by other methods (Ref 2)

2, 3-Dinitro-p-cresol, yel nds (from glacial AcOH), mp 157-59°; prepd by nitrating 2-nitro-p-cresol in AcOH at 18-25° with HNO_3, and by other methods (Ref 3)

2, 5-Dinitro-p-cresol, yel-brn ndls (from alc), mp 112-13°; prepd by nitrating 2-nitro-p-cresol in AcOH at 18-25° with HNO_3, together with other products (Ref 3, p[391])

3, 5 (or 2, 6) - Dinitro-p-cresol, lt-yel crysts (from eth), mp 82-85°; prepd by nitration of p-cresol in AcOH with HNO_3 and by other methods (Ref 3). It forms numerous salts & addn compds, some of which are expl

Dr Otto Borgmann obtd patents in 1893 for the manuf of expls contg salts or additive compds similar to those produced from Dinitrophenols (cited in Ref 4). The shock sensitiveness of Dinitrocresols, and a number of other expls, was detd by Wöhler & Wenzelberg (Ref 6a). In addn to their use as expls, Dinitrocresols have been used in selective herbicides & insecticides (Ref 11). See also Cresylates

Refs: 1) Beil **6**, 368, (180) & [341] 2) Beil **6**, 387, (193) & [362, 363] 3) Beil **6**, 414, (207) & [391] 4) Colver (1918), 290 5) A.E. Goddard & J.B. Ward, JChemSoc **121** 265(1922) 6) A.E. Goddard JChemSoc **123**, 1167 (1923) 6a) L. Wöhler & O. Wenzelberg, Angew Chem **46**, 173-76 (1933) 7) W. Brün, USP 1943513 (1934) & CA **28**, 2188 (1934) 8) L. Rubinstein & W. Taylor, BritP 471907 & 473146(1937) & CA **32**, 1934, 1936 (1937) 9) L.A. Burrows & G.A. Noddin, USP 2268372 (1941) & CA **36**, 2726 (1942) 10) Davis (1943), 147 11) Kirk & Othmer **4** (1949) 606 12) V.H. Parker, Analyst **74**, 646-47 (1949) 13) J. Issoire, MP **36**, 27-36 (1954) (Manuf of Dinitro-o-cresol) 14) Sax (1957), 278-79

Trinitrocresols (TNCrs), $CH_3.C_6H(NO_2)_3.OH$; mw 243.13, N 17.28%. Two of the Trinitro derivs found in the literature are the following:

3,4 5 (or 4,5,6)-Trinitro-o-cresol, orn-yel prisms (from acet), mp 102°; sol in alc, eth, acet & ethyl acetate; sl sol in chlf; v sl sol in cold w (Ref 11); prepd by diazotizing an ice cold soln of 4-nitro-2-aminotoluene in HNO_3, then treating with moderately hot HNO_3 and finally boiling HNO_3 (Ref 1)

2,4,6-Trinitro-m-cresol or 3-Methylpicric Acid, golden-yel ndls (from alc or w), mp 106.5-110°, bp-explodes on heating to 150°, d 1.69; sol in alc, eth & acet; sl sol in w; can be prepd from m-cresol by a method similar to the prepn of PA from phenol, and by other methods (Refs 2 & 7). Its Ger name is **Kresylit**

This compd is a powerful expl. It was used at one time in Europe as a bursting chge in projectiles. Its expl props are reported as follows: *brisance* by Pb block compression 97% TNT (Ref 9); *heat of combustion* 771 cal/mol at C_v (Ref 17) & 777 cal/mol (Ref 15); *impact sensitivity* sl more sensitive than PA (Refs 3 & 14); *power* by Trauzl test (for 15g sample) 384 cc vs 452 cc for TNT or 85% TNT (Ref 18) and 90% PA (Refs 8 & 9): *specific energy* (f) 7595 vs 8080 for TNT; *specific volume* (v) 675 liters vs 690 liters for TNT; *temperature developed on explosion* (calcd, t) 2700° v s 2830° for TNT (Ref 6); *thermal stability* explodes at 252-55° when heated at the rate of 10°/min (Ref 10) and 405° as the expln temp (Ref 5); and *Velocity of detonation* 6620 m/sec at d 1.52 & 6850 at d 1.68 (Ref 4) Other props are given in Ref 8

The French designation for Trinitro-m-cresol alone and also for its mixts with PA is **Crésylite.** The mixt of TNCrs 60% & PA 40% is called **Crésylite No 2** (Ref 13). It becomes plastic at 65-70° and melts at 85°. On being remelted the mixt at first assumes a pasty consistency, after which (6 hrs) it solidifies to an extremely compact substance. It has been used as a HE bursting chge, cast loaded into some projectiles. The same mixt was adopted in Italy under the name **Cresilite.** It is called by Molina (Ref 12) **Ecrasite**

Trinitrocresols form numerous salts which are expl (Ref 16). See under Cresylates

Refs: 1) Beil **6**, 369 2) Beil **6**, 387, (194) & [363] 3) W. Will, SS **1**, 209 (1906) 4) H. Kast SS **8**, 173 (1913) 5) R.L. Datta & N.R. Chatterjee, JChemSoc **115**, 1006 (1919) 6) H. Kast, SS **15**, 173 & 183 (1920) 7) D. Wride, Arms & Expls **28** 47 (1920) 8) B.J. Flürsheim, JSCI **40** 103T (1921) 9) W.M. Dehn & A.A. Wagner, ArOrdn **8**, 35 (1927)

10) G. Tammann & C. Kröger, ZAnorgChem **169**, 1(1928) 11) L. Desvergnes, RevChimInd **38**, 66 (1929) 12) Molina (1930), 391 13) Vennin, Burlot & Lécorché (1932), 457 14) L. Wöhler & O. Wenzelberg, ZAngChem **46**, 173 (1933) 15) A. Schmidt, SS **29** 262 (1934) 16) Pepin Lehalleur (1935), 274 17) E. Burlot & M. Thomas, MP **29** 262 (1939) 18) Davis (1943), 132 & 169 19) A.I. Titov, ZhObshchKhim **22**, 1329 (1952) & CA **47**, 6360 (1953) (Prepn of Trinitro-m-cresol & other phenol derivs) 20) D.G. Harvey, JPharm & Pharmacol **5**, 497-510 (1953) & CA **47**, 11526 (1953) (Toxicity of nitrated cresols) 21) H. Brockmann & E. Meyer, Naturwissen-schaften **40**, 242 (1953) & CA **48**, 504 (1954) (Titration in anhydrous solvs) 22) M.M. Davis & H.B. Hetzer, JACS **76**, 4247 (1954) (Relative acidity of Trinitro-m-cresol & PA) 23) A. Langhans, Explosivst **1/2**, 3-11 (1954) (Thermal decompn of various cyclic nitrocompds 24) A.I. Titov, ZhObshchKhim **24** 2040 (1954 & CA **49**, 14659 (1955) (Prepn of Trinitro-m-cresol and other nitrated cresols) 25) C.E. Moore & R. Peck, JOC **20** 673 (1955) (UV absorption spectra & ionization constants) 26) Sax (1957), 1223 (Explosion hazard) 27) Giua, Trattato **6** (1) (1959), 316 (Trinitrocresilolo)

Tetranitrocresol (TeNCrs). Only one isomer is known: **2,4,5,6- Tetranitro-m-cresol,** $CH_3.C_6(NO_2)_4.OH$; mw 288.13, N 19.45%; col crysts (from chlf) having a bitter taste, mp 175°, bp explodes; readily sol in alc or w, giving yel solns; was prepd by nitrating 5-nitro-m-cresol with concd HNO_3 & concd H_2SO_4 at 50°. This compd is a powerful expl (Refs 1 & 2)

Refs: 1) Beil **6**, 388 2) J.J. Blanksma, Rec **27**, 34(1908) & CA **2**,1134 (1908)

Cresolsulfonic Acid and Derivatives

m-Cresolsulfonic Acid [called m-Kresol-sulfonsäure -(4) or 3-Oxy-toluol-sulfonsäure - (6) in Ger], $CH_3.C_6H_3(OH).SO_2OH$; mw 188.20, lfts or ndls (from cold w or better from dil H_2SO_4) with 2 mols H_2O, mp 95-96° the anhyd crysts melt at 118°, bp dec; readily sol in w, alc, benz & eth; was prepd by sulfonation of m-cresol, and by other methods (Ref 1). Its Na salt, **Sodium Cresolsulfonate,** $CH_3.C_6H_3(OH).SO_2O$ Na, was proposed by Voight as an ingredient of safety blasting expls (Ref 3). One compn, called by Colver (Ref 6) **Raschite IV** consisted of AN 87 & Na cresolsulfonate 13%. Marshall (Ref 5) reports a proposal by F. Raschig in 1912 to make a cheap expl consisting of $NaNO_3$ 65 & Na cresolsulphonate 35%. Some of its other salts (Ref 1) might also be suitable as an ingredient of expls

6-Mononitro-m-cresolsulfonate, $CH_3.C_6H_2(NO_2)$-$OH.SO_2OH$; mw 233.20, N 6.01%; col ndls (from eth acetate or acet & chlf); was prepd by sulfonation of 4-nitro-3-hydroxytoluene at 70°, or by nitration of 3-hydroxy-1-methylbenzene-6-sulfonic acid (Ref 2). The following salts have been proposed as ingredients of expls:

Ammonium Mononitrocresolsulfonate, $CH_3C_6H_2(NO_2).OH.SO_2ONH_4$, was used in **Raschite III,** AN 84 & Amm Mononitrocresolsulfonate 16% (Ref 6)

Sodium Mononitrocresolsulfonate, $CH_3.C_6H_2(NO_2).OH.SO_2ONa$, orn-yel prisms, was used in some safety expls: a) $NaNO_3$ 75 & Na Mononitrocresolsulfonate 25% (Ref 6) b) $KClO_4$ 50, KNO_3 25 & Na Mononitrocresolsulfonate 25% (Ref 6) and c) $NaNO_3$ or KNO_3 65, Na Mononitrocresolsulfonate 25 & $KClO_3$ or $KClO_4$ 10% (Ref 4)

Refs: 1) Beil **11** 256, (60) & [145] 2) Beil **11** 257 & [146] 3) A. Voight, BritP 1712 (1911) & CA **6**, 1991 (1912) 4) A. Voight, USP 1095302 (1913) & CA **8**, 2253 (1914) 5) Marshall **1** (1917) 90 6) Colver (1918), 351-52

Creuse Charge. See Charge creuse, Vol 2, p C151-R

CRESYLATES AND OTHER DERIVATIVES OF NITRATED CRESOLS

Nitrated cresols form a number of salts, by interaction with inorganic compds. These salts are called Cresylates. They possess props similar to those of Picrates, except that they are less powerful & less sensitive expls than Picrates. Colver (Ref 4) gives a fairly complete list of these compds, and Kast (Ref 3) describes their props. Some of the more important Cresylates are:

Aluminum Trinitrocresylate (Basic), $HO.Al-(C_7H_4N_3O_7)_2 + 5H_2O$; pale reddish ndls, mp defgr above 100°; sol in w & alc; resembles the corresponding picrate in other props (Refs 1 & 4). See also Ref 2

Ammonium Trinitrocresylate (Called Ecrasita in Spanish, Ecrasite in Ital, and Ekrasit in Ger & Rus), $CH_3.C_6H(NO_2)_3.ONH_4$; yel-red, nonhygr ndls, mp ca 100°, bp explodes; readily sol in w,

less sol in alc; may be prepd by addg NH_4OH, Liq NH_3 or $(NH_4)_2CO_3$ to an aqueous soln of Trinitro-m-cresol. This compd is a powerful expl, its brisance by sand test is 90% of TNT (Ref 5); and is fairly insensitive to impact & friction. This expl was used by the Austrians during WWI, under the name *Ekrasit*, for filling some shells, and by the Italians during WWII as a bursting chge in large caliber shells. The 60/40 mix of PA/Trinitrocresol is also called *Ecrasite* by Molina (Ref 6). Hallett (Ref 11) reports that when Ammonium Trinitrocresylate is mixed with a large proportion of NG, there are formed HE's which are less susceptible to extreme temps than dynamites. See also Ref 10

Barium Trinitrocresylate, $Ba(C_7H_4N_3O_7)_2 + H_2O$; small sulfur-yel pltlts (from 90% alc), mp-loses H_2O above 130° and then defgr; readily sol in w, diffc sol in alc (Refs 1 & 4)

Calcium Trinitrocresylate, $Ca(C_7H_4N_3O_7)_2 + 4H_2O$; small, pale reddish-yel ndls (from alc), mp-loses 1 mol H_2O above 130° and then defgr; readily sol in w & alc (Refs 1 & 4)

Cupric Trinitrocresylate, $Cu(C_7H_4N_3O_7)_2 + 2H_2O$; greenish-yel ndls (from abs alc or w), mp-loses $2H_2O$ above 130° and then defgr; readily sol in w & alc (Refs 1 & 4)

Ferric Trinitrocresylate, $Fe(C_7H_4N_3O_7)_3$; glittering dk-brn crysts, mp-decomposes very readily; sol in alc, diffc sol in w (Refs 1 & 4)

Ferrous Trinitrocresylate, $Fe(C_7H_4N_3O_7)_2 + 2H_2O$; greenish-yel ndls, mp-decomp above 100°; readily sol in alc & w (Refs 1 & 4)

Lead Trinitrocresylate, $Pb(C_7H_4N_3O_7)_2 + H_2O$; sulfur-yel lfts (from very dil alc), mp-loses H_2O above 130° and then explodes violently; readily sol in alc, diffc sol in w (Ref 1). It is extremely sensitive to friction & impact (Ref 4)

Lead Trinitrocresylate (Basic), $HO.PbC_7H_4N_3O_7$; yel microscopic ndls, mp -explodes on heating; sol in w; was prepd by gradually addg to the normal salt, aqueous solns of a base [such as KOH, NaOH, $Ca(OH)_2$, $Ba(OH)_2$ & NH_4OH] and a sol Pb salt (Ref 1, p 387 & Ref 7)

Rubenstein (Ref 7) proposed using this compd to make high-tension fuseheads for elec blasting detonators. The blanks were dipped in a suspension of basic Pb Trinitrocresylate 73 & graphite 27% in a 2.5% soln of NC. The resulting beads were dried and dipped in a suspension of the cresylate alone in NC sdn and again dried. This was followed by dips into charcoal + $KClO_3$, and finally into collodion

Magnesium Trinitrocresylate, $Mg(C_7H_4N_3O_7)_2 + 6H_2O$; reddish-yel crysts (from alc or w), mp - loses $3H_2O$ above 130°; readily sol in alc or w (Refs 1 & 4)

Potassium Trinitrocresylate, $KC_7H_4N_3O_7 + H_2O$; yel lfts (from 90% alc), mp-explodes violently on heating; readily sol in w & alc; was prepd by saturating a soln of Trinitrocresol with K_2CO_3 or other K salt (Refs 1 & 4)

Silver Trinitrocresylate, $AgC_7H_4N_3O_7$; reddish-yel crysts, mp-explodes mildly on heating; sol in alc & w (Ref 1)

Sodium Trinitrocresylate, $NaC_7H_4N_3O_7 + 2H_2O$; dk reddish-yel ndls (from alc), mp-explodes mildly on heating; readily sol in alc or w (Refs 1 & 4)

Thallium Trinitrocresylate, $TlC_7H_4N_3O_7$; dark yel crysts, mp-explodes on heating (Ref 1, p [365])

Zinc Trinitrocresylate, $Zn(C_7H_4N_3O_7)_2 + 6H_2O$; reddish-yel crysts, mp-loses $3H_2O$ above 130° readily sol in alc or w (Refs 1 & 4)

The double salt of 4,6-dinitro-o-cresol, **Lead nitrato-bis(basic lead - 4,6 - dinitro-o-cresylate) monohydrate,** $[(O_2N)_2C_6H_2(CH_3)O-]_2 Pb.2Pb-(OH)_2.Pb(NO_3)_2+H_2O$; was recommended by Burrows & Filbert (Ref 8) as an ingredient of ignition compns for elec blasting initiators. This compd, obtd as orn colored crysts, loses H_2O at 150°. It was prepd by addg a soln of 4,6-Dinitro-o-cresol in H_2O & NaOH to a mechanically stirred soln of $Pb(NO_3)_2$ at 60°. This complex salt is less sensitive to impact & friction than MF. It can be stored at 110°F without any appreciable change. It is readily ignited by the heat of an incandescent bridge with a low firing current. This salt can be used alone, or in admixtures with oxidizers & finely divided metals. Some examples of compn proposed by Burrows et al (Refs 8 & 9) are as follows: a) Pb salt of DNCrs 80 & zirconium 20% b) Pb DNCrs 80 & $KClO_3$ 20% c) Pb DNCrs 70, Zr 15 & $KClO_3$ 15% d) Pyrocellulose proplnt 65, double salt of $Pb(NO_3)_2$ & bis basic salt of 4,6-DNCrs 25, and $KClO_3$ 10% e) Pyrocellulose proplnt 50%, complex Pb salt as above 25 & $KClO_3$ 25% and f) Pyrocellulose proplnt 75 & complex Pb salt as above 25%. The mixture given in d) reqd 0.299 amp to ignite the loose powder, mixt e) reqd 0.318 amp and f) 0.309 amp minimum chge

Organic Cresylates

Trinitrocresols form a number of expl compds with organic hydrocarbons, of which the following are described in the literature:

Trinitrocresol + Allylamine, $C_7H_5N_3O_7 + 3C_3H_7N$; yel crysts (from alc), mp 165°, bp-explodes at 265° (Ref 1, p [365])

Trinitrocresol + Acenaphthene, $C_7H_5N_3O_7 + C_{12}H_{10}$; orn-red crysts or yel crysts (from alc), mp 117.9-120°, bp-explodes at 451° (Ref 1, p [365])

Trinitrocresol + Fluorene, $C_7H_5N_3O_7+C_{13}H_{10}$; gray or dk-yel crysts (from benz), mp 107.2-109, bp-decomp at 150° and explodes at 421°(Ref 1, p [365])

Trinitrocresol + Naphthalene, $C_7H_5N_3O_7+C_{10}H_8$; lt-yel ndls (from acet), mp 125-27°; decompd by w, diffc sol in acet (Ref 1, p 387). A similar salt is formed from Trinitro-o-cresol & naphthalene (Ref 4)

Refs: 1) Beil **6**, 387 (195) & [365] 2) Escales, Ammonspr (1909), 64 3) H. Kast, SS**6**, 7,31 & 67 (1911) 4) Colver (1918), 348-50 5) W.M. Dehn & A.A. Wagner, ArOrdn **8**, 35 (1927) & CA **21**, 3270 (1927) 6) Molina (1930), 391 7) L. Rubenstein, BritP 495411 (1938) & CA **33**, 3158 (1939) 8) L.A. Burrows & W.H. Filbert , USP 2175249 (1939) 9) L.A. Burrows & G.A. Noddin, USP 2268372 (1941) 10) All & EnExpls (1946), 114-15 11) C.S. Hallett, BritP 57514 (1946) & CA **41**, 4312 (1947) 12) Giua, Trattato **6** (1) (1959), 316

Cresylic Acid. Same as Cresol

Cresylite. See under 2,4,6-Trinitro-m-cresol

Criminal Devices (Engins Criminels or Bombes in Fr). See Explosives and Weapons Used by Anarchists and Revolutionists in Vol 4

Crimper, Blasting Cap. A manually operated tool designed for fastening blasting caps to safety fuse

Ref: OrdTechTerm (1962), 87

Crimping. 1) A mechanical operation on metal or other material by which the material is permanently deformed, usually by small undulations, frills or wrinkles 2) The process by which a cartridge case is secured to the projectile, either by a continuous folding in of the neck of the cartridge case into a crimping groove, or by a series of deformations (crimps) at a location somewhat below the cartridge case mouth 3) In blank ammo, the closing in of the neck of the cartridge case to secure the wadding over the chge

Refs: 1) Davis (1943), 113-14 2) OrdTechTerm (1962), 87

Critical Conditions in Thermal Explosion Theory with Reactant Consumption. Title of a report by J. Adler et al, US Naval Ord Lab Tech Rept **63-251** (Dec 1963)

Critical Constants of Liquid Explosives. These are temp, pressure, vol & density. Such critical constants of liq expls cannot be measured directly because of the intractable nature of the substances. Equations have been developed by Lewis (Ref) whereby the critical temps & critical densities may be detd with a high degree of accuracy from measurements made in an accessible range of densities & surface tensions of some liq expls. It is shown by Lewis in applying these equations to TNT, NG & mercury that they are of general applicability to a wide range of chemical substances

Ref: D.T. Lewis, JApplChem (London) **3**, 154-60 (1953)

Critical Diameter. The effect of the initial temp on the magnitude of the critical diam of NG & TNT was detd by Belyaev & Kurbangalina (Ref 1). These authors found that for NG & liq TNT the diam decreased with an increase in temp. For NG, as the temp was raised from -20°to 70°, the diam decreased from 3.9 to 1.1 mm. Liq TNT, near its mp, has a critical diam greater than NG, at RT, and consequently its deton capacity & sensitivity are quite different from those for NG; however, at the flash point for liq TNT, it approaches NG in expl props. It was concluded that the mechanism for the chem conversion for cryst TNT is different from that for liq TNT

The critical diam and rate of deton of liq

ozone solns were detd by Gribova et al (Ref 2). The values of $d_{critical}$ of liq O_3 solns contg 4, 42, 53, 63 & 68% O were < 0.15, < 0.9, < 4.5, < 30 & < 100 mm, respectively. These exptl values are in close agreement with those calcd by Khariton's theory:

$$d_{crit} = (ART^2/ET_e)e^{E/RT}$$

for a pure liq expl, and

$$d'_{crit} = d_{crit}\ \alpha e^{q}$$

for mixts; where

$$q = E/RT\ [(1 - \alpha)/\alpha]$$

and A = constant, T = temp of front of detón wave, T_e = temp of products of expln, E = energy of activation, R = gas constant, and α is the wt fraction of the expl component in the mixt. The exptl values of Gribova et al (Ref 2) indicate that deton of liq O_3 & its solns is governed by the kinetics of decompn, especially at low values of E, and a high frequency factor. The rates of deton of liq O_3 contg 53, 40, 19 & 4% O in a tube, having a diam 15-18 mm & a length of 250-300 mm are 1840, 4260, 6460 & 6840 m/sec, respectively

Dremin (Ref 3) detd the critical diam for the deton of Nitromethane & its mixts with acetone. Analysis of his exptl data showed that the luminosity of the deton-wave front was not uniform. This implied that the reaction did not originate homogeneously in the chge but in isolated areas. By analogy with gaseous detons, it is shown that deton waves in liq expls develop transverse shock waves with the initiating reaction transposed from the combustion surface to a point where the transverse shock waves impinge. On the basis of these ideas, a model of propagation of deton in a cylindrical chge of a liq expl is developed and an equation derived for calcg the critical diameter

Refs: 1) A.F. Belyaev & R Kh. Kurbangalina, ZhFizKhim **34,** 603-10 (1960) & CA **55,** 15931 (1961) 2) E.I. Gribova et al, ZhFizKhim **34,** 1395-1401 (1960) & CA **55,** 6867 (1961) 3) A.N. Dremin, DoklAkadN **147,** 870-73 (1962) & CA **58,** 7779-80 (1963)

Critical Mass. The min mass, relative to a specific shape & environment, of a fissionable material necessary to sustain a nuclear chain reaction. See also Atomic (or Nuclear) Bomb and Atomic (or Nuclear)Energy, Vol 1, pp A499-A504

Ref: OrdTechTerm (1962), 88

Critical Velocity. A Brit term used to evaluate the performance of an AP projectile against armor plate. For each combination of projectile and plate of certain physical characteristics & thickness, there is theoretically a striking vel at which complete penetration would just occur. This velocity, detd in a certain prescribed manner, is called the *critical velocity*

Ref: OrdTechTerm (1962), 88

Croning Process of Precision Casting. See "C" Process

Cronite. An Amer coal-mine expl, Nos 1 & 5, two varieties of which are on the Permissible List. Both are based on AN as the main ingredient (Ref) See COMMERCIAL OR INDUSTRIAL EXPLOSIVES Section II, for other US Permissible Coal Mining Explosives

Ref: Marshall, Dict(1920), 26

Cronquist, Albert Werner (1846-1910). Swedish scientist who specialized in the science of expls. Hig obituary & contributions are recorded by Klason (Ref 2) Cronquist (Ref 1) developed a "Pendulum for Determination of Inflammability of Propellants & Explosives" (Grad der Entzündungsleichtigkeit in Ger) (Ref 1)

Refs: 1) A.W. Cronquist, SS **1,** 106 (1906) 2) P. Klason, SS **6,** 43-44 (1911)

Cross and Bevan Cellulose. An impure cellulose isolated from the complex it forms with lignin, in materials such as wood & straw. The method of isolation is described by Dorée (Ref 1) & Ott (Ref 2). Cross & Bevan cellulose contains both furfural-yielding substances (pentosans), and hexosans of the mannan type. It retains, in the case of coniferous woods, 40-60% of the total furfural-yielding substances in the original wood, and about 55-66% in the case of hard woods.

Refs: 1) Dorée (1947), 348, 352 & 362 2) Ott **5,** Part 1 (1954), 35, 450 & 462-63 & Part 3 (1955), 1374

Crossbar Shot (Called Stangenkugel in Ger). A spherical shot designed with two sections attached together by a folded iron bar which runs thru the center of the shot. Also an obsolete projectile which folded into a sphere for muzzle loading, but on leaving the gun opened into a cross having a quarter ball attached at each end of the arms. These shots were designed to inflict max damage among massed personnel or to damage the rigging of sailing vessels.
Refs: 1) Farrow's Military Encyclopedia, Military - Naval Publg Co , NY (1895), Vol 1, p 274 2) Merriam - Webster's (1961), 541

Crossbow. See Arbalest, Vol 1, p A477-L

Cross, Charles F (1855-1935). British scientist, a pioneer in modern cellulose chemistry; his prepn of acetyl & benzoyl celluloses (1889-90) was a prelude to the great achievement of his career, the discovery of cellulose Na xanthate (viscose) which was patented under the names of Cross, Bevan & Beadle (1892). Cross & E.J. Bevan published "Textbook of Papermaking" (1887), "Cellulose, an Outline of the Structural Elements of Plants" (1895), " Wood Pulp and Its Applications" (1911), and numerous other works on every aspect of cellulose. The pioneer work and the persistent & successful efforts of Cross were duly recognized during his life. He was awarded the medal of the Society of Chemical Industry (1916) for "conspicuous service to chemical industry", elected a Fellow of the Royal Society (1917), awarded a medal by the Worshipful Company of Dyers (1919), and was elected President of the Society of Dyers and Colourists for 1920-21
Ref: C. Dorée, Analyst **60,** 437-39 (1935) & JChemSoc **1935,** 1337-40

Crosslinking Agents for Nitrocellulose. The crosslinking of cellulose & its derivs is mentioned briefly by Ott (Ref 3). Jullander & Blom-Sallin (Ref 2) studied the crosslinking of NC by means of $TiCl_4$ & $Ti(OEt)_4$ and butyl acetate as solv. The props of such gels were detd by means of the ultracentrifuge and a consistometer. The crosslinking of NC (13.47% N) was achieved by Bouchez & Champetier (Ref 4) by action of sebacoyl chloride with benz as the suspending agent. Other studies on the subject are reported by Sihtola et al (Ref 5), Bouchez (Ref 6), and McDonnell & Matushko (Ref 7)

Reynolds et al (Ref 1) proposed the use of NC crosslinking agents such as *hexakis (methoxymethyl) melamine, bis(butoxymethyl) urea, hexamethylene diisocyanate,* and N, N^1-bis(hydroxymethyl) *adipamide* as ingredients of blasting gelatin (See Vol 2, p B211-R). It is claimed that quantities from 0.05 to 0.50% of these agents, incorporated in the gelatin mix, preserve the sensitivity to deton of blasting gelatin in storage

See also Cellulose Nitrate, Action of Solvents, Vol 2, pC115-R
Refs: 1) R.J.W. Reynolds, S. Fordham & ICI, BritP 593163 (1947) & CA **42,** 9180 (1948) 2) I. Jullander & B.S. Blom-Sallin, JPolymerSci **3,** 804-11 (1948) & CA **43,** 8673-74 (1949) 3) Ott, Vol **5,** Part 2 (1954), 700 & Part 3 (1955), 1088, 1348 4) É. Bouchez & G. Champetier, CR **242,** 635-37 (1956) & CA **50,** 12838-39 (1956) 5) H. Sihtola et al, JPolymerSci **30,** 1-4 (1958) & CA **52,** 21071 (1958) (Crosslinking of cellulose, impregnated with sebacic acid, during nitration) 6) E. Bouchez, MP **41,** Annexe 4218, 1-74 (1959) & CA **56,** 2606 (1962) (Crosslinking of NC by acid chlorides) 7) C.H. McDonnell & A.J. Matushko "Crosslinking Reactions of Nitrocellulose with Diepoxide and Divinyl Sulfone," Naval Propellant Plant Tech Rept **127** (April 1963) (NAVWEPS Rept **8027**) (Conf) (Not used as a source of info)

Crotonic Acid and Derivatives

Crotonic Acid or Butenoic Acid (called α-Crotonsäure in Ger), $CH_3.CH{:}CH.COOH$; mw 86.09. O 37.17%; col ndls (from w) or monoclinic prisms (by evapn), mp 71.4 - 71.7°, bp 189°, d 0.9730 at 20°, n_D^{77}/1.4249, heat of comb 478 kcal/mol; was first prepd in 1883 (Ref 1) but did not become available in coml quantities in North America until 1936 (Ref 2). Crotonic acid is prepd in the lab by condensation of acetaldehyde in dry eth soln with malonic acid in the presence of pyridine. It is prepd on a coml scale by oxidn of crotonaldehyde with air or oxygen

Crotonic acid forms numerous salts. It is used mainly in the prepn of synthetic resins, surface coatings, plasticizers & pharmaceuticals
Refs: 1) Beil **2,** 408, (187), [390] & {1255} 2) Kirk & Othmer **1** (1947), 143; **4** (1949), 613-17

3) Ullmann **5** (1954), 617-20 4) Sax (1957), 512-13

Crotonyl Azide (Called Crotonsäureazid in Ger), $CH_3.CH:CH.CO.N_3$; mw 111.10, N37.83%; clear liq having a sharp odor; bp-begins to decomp at 30°& rapidly evolves N at 75°; was prepd by treating crotonyl chloride in ether soln with NaN_3 (Refs 1 & 2)

Refs: 1) Beil **2**, [394] 2) L.W. Jones & J.P. Mason, JACS **49**, 2533 (1927)

Mononitro, $C_4H_5NO_4$, and *Dinitrocrotonic Acid,* $C_4H_4N_2O_6$, derivs were not found in Beil or in CA thru 1961. Derivs of 4,4-Dinitrocrotonic Acid or 4,4-Dinitrobutene-2-oic Acid were prepd from nitroform by the Michael's reaction in non-alkaline media (Ref)

Ref: L.A. Kaplan et al, NAVORD Rept **4536** (May 1957) (Conf)

Crotonyl Peroxide. See Dicrotonylperoxide

Crown Oil E. A lubricating oil formerly manufd by the Gulf Oil Corp. It was used during WWII in US experimental plastic expls such as *PEP-2* (85/15 PETN/Gulf Crown Oil E); *PEP-3* (86/14 PETN/Crown Oil E); *PIPE* (81/19 PETN/Crown Oil E); and *RIPE* 85/15 RDX/Crown Oil E). These expls were intended to be used, as fillers for special shells & rockets, against reinforced concrete (Refs 1, 3 & 4). These expls were prepd and evaluated at Picatinny Arsenal (Refs 2 & 5). They were found to have a high order of chem stability, but to undergo physical deterioration & consequent exudation at slightly elevated temps. Plastic expls employing Crown Oil E were not used in service ammo. Crown Oil E is not presently manufd as it has been replaced by other products of the Gulf Oil Corp (Ref 6)

Refs: 1) E.H. Eyster & M.A. Paul, "PEP-2, PETN Plastic Explosive," OSRD Rept **3240** (1944) 2) S. Livingston, "Properties of Explosives RIPE, PIPE and PEP-3", PATR **1517** (1945) 3) E.H. Eyster, "The Preparation and Properties of Plastic High Explosives", OSRD Rept **5631** (1946) 4) Summary Technical Report of Division 8, NDRC, Vol 1 (1946), 30 5) S. Livingston "Plastic Explosives - Comprehensive Study of the Plastic Explosives RIPE, PEP-3, and Composition C3", PATR **1695** (1948) 6) Private communication from representative of Gulf Oil Corp, NY (Nov 1964)

CR (Cyclonite Rifle) **Propellants.** Experimental rifle propellants, based on RDX & NC, were developed dury WWII by the DuPont Company. Their compns are as follows:

CR1 - NC(12.6% N) 47.0, RDX 47.0, butyl stearate 5.5 & DPhA 0.5%

CR2 - NC(12.6% N) 50.0, RDX 45.0, dibutyl tartrate 4.5 & DPhA 0.5% with 0.7% K_2SO_4 added

CR3 - NC(12.6%) 36.5, RDX 60.0, Triacetin 3.0 & DPhA 0.5% with 0.7% K_2SO_4 added

These proplnts exhibited superior thermochemical props, but showed a high pressure exponent in the burning rate equation. All the proplnts, when coated with DNT, gave long ignition times. They showed, in general, good velocity-pressure relationships, but poor uniformity from round to round (Ref 2). The burning stabilities of these proplnts were comparable with those of single-base proplnts, and much better than for proplnts contg NG (Ref 1)

The manuf & analytical proceedures used are described in Ref 2

See also CC (Cyclonite Cannon) Propellants in Vol 2, p C92-L; and Composite Propellants in this Vol

Refs: 1) R.M. Lemmon, OSRD Rept **5945** (1945) 2) Burnside Lab of DuPont, OSRD Rept **6216** (1945) 3) Anon, Summary Rept of Div 8, NDRC Vol 1 (1946), 110-11

Cruciform. A configuration in the form of a cross with equal legs, 90° apart. This shape is used for certain grains of rocket propellant. See Fig 3 under CANNON PROPELLANT, Vol 2, p C30-R

Refs: 1) OrdTechTerm (1962), 88 2) Wimpress, Ballistics (1950), 104 & 120

Cruciform Luminosity Produced by Detonation of Explosive Cartridges of Square Cross Section. If the axis of a photographic camera is placed in a direction parallel to the long axis of a cylindrical cartridge, the photographic plate shows that the luminosity produced, on explosion of the cartridge spreads evenly in all directions. When the expln

of a square cross-sectional cartridge is photographed, the photographic plate will show a luminous cruciform with its arms in directions perpendicular to the faces of the rectangular cartridge, and dark space (zone) all around the crucifix. The dark space is called the "dead zone" ("zone morte"in French). This phenomenon of a luminous cruciform shows that when a body other than round is exploded, the deton wave & the products of expln do not spread evenly in all directions, but follow certain lines (Ref 7). The explanation of this pehnomenon if given in Refs 2 to 6

Before this explanation was available, one did not understand why the expln of a square building, such as happened at Sevran-Livry in 1893, caused more damage to surrounding objects lying on the horizonal axes of the bldg than those facing the angles of the bldg (Ref 1)

If, in photographying the expln of cartridges, the camera is placed perpendicular to the long axis of the cartridge (verticle axis of a bldg), the photographic plate will show that the direction of luminous rays is not perpendicular to the axis, but inclined in a direction opposite to that of detonator (Ref 4)

Refs: 1) (?).Biffe, MP **8**, 2^{e} partie, "Documents administratifs" (1893), p 14 (In the expln at the Poudrerie de Sevran-Livry, the greatest damage was caused in four directions oriented exactly accdg to the four axes of the factory. No explanation of this phenomenon was given at the time) 2) E. Burlot, MAF **9**, 789-957 (1930) 3) P. Demougin, MAF **9**, 961-72 (1930) 4) H. Ahrens, SS **38**, 175-77 (1943) (Wirkungen bei der Detonation von Sprengkörpern) 5) H. Muraour, CR **222**, 1108 (1946) 6) A. Michel-Lévy, CR **223**, 62 (1946) 7) D. Riabouchinsky, CR **237**, 222-24 (1953) (Sur les luminsoités en forme de croix produits par la détonation d'une cartouche d'explosif de section carrée)

Crusher Gage. A device used to measure the pressure developed in a gun chamber by measuring the deformation of a Cu cylinder. See under CLOSED BOMB etc

Ref: OrdTechTerm (1962), 136

Crusher Tests. See Esop's Test for Determination of Efficiency of Detonators, Vol 1, p XI

See Compression (or Crusher) Tests for Determination of Brisance, in this vol, p C 492-L

See Compression (or Crusher) Tests for Determination of Pressures, under CLOSED BOMB (OR VESSEL) AND INSTRUMENTS etc, in this vol, p C 330-L

Crushing and Grinding. Crushing is the reduction in size of particles of a material by impact blows or by compression between two hard surfaces. If one-step crushing (primary) is done, then the resulting particles are rather coarse. In order to produce finer particles, the crushing operation usually has to be repeated (secondary), or the material is subjected to *grinding.* This is the reduction of a material to a powdery state (pulverization) in devices based on friction (such as millstones) or concussion or abrasion with metal balls (ball grinders), or pebbles (pebble grinders)

Crushers may be divided into the following types:

a)*Cone crushers* are secondary or reduction crushers. The two common types are Symons (Nordberg Mfg Co) and Telsmith (Smith Engrg Works)

b)*Corrugated- and Toothed-Roll crushers.* The simplest type consists of a single roll (toothed) operated against a breaker. It is used for coarser crushing. More complicated devices contain several corrugated or toothed cylinders rotating in a horizontal plane at the same or different speeds. Examples of these crushers are the Fairmount Single-Roll unit(Allis-Chalmers), Bartlett & Snow Single-Roll unit and Two-Roll crushers (Bartlett & Snow), Sawtooth crusher (Sprout, Waldron & Co), and Jeffrey Single-Roll crusher (Jeffrey Mfg Co)

c)*Gyratory crushers.* Type R & Superior McCully (Allis-Chalmers), Multi-stage Fine Reduction crusher & Type T Bulldog (Traylor), Gearless Gyratory (Kennedy-Van Saun), and Telsmith Breaker (Smith Engrg Works)

d)*Hammer crushers* consist of a horizontal metallic plate and several pivoted hammers mounted on a horizontal shaft. Crushing is achieved by impact between the hammers & plate. Some types are Double Impeller (New Holland Machine Co), Jeffrey (Jeffrey Mfg Co), Super-Jumbo (Williams Patent Crusher & Pulverizer Co), Impactor (Pennsylvania Crusher

Co), American Ring (American Pulverizer Co), Stedman Type B Heavy-Duty (Stedman's Foundry & Machine Works), and Joy-Hazemag Impact Crusher (Joy Mfg)

e)*Jaw crushers* are represented by those of Blake, Dodge, and Fine Reduction (Allis-Chalmers); Kue-Ken Balanced Jaw (Straub Mfg Co); Type H, Type HB, and Type S (Traylor); Universal (Universal Engrg Co); and Nertia (Southwest Engrg Co)

f)*Pan crushers* consist of one or more grinding wheels or mullers revolving in a pan which either remains stationary or is driven. In the first case the mullers are driven and in the latter case the mullers revolve by friction. An example of this type of crusher is the Bonnot Dry Pan (Bonnot Co)

g)*Rotary crushers* may be either horizontal, as in the Horizontal Crusher (Sprout, Waldron & Co), or vertical, as is that of Bartlett & Snow. They usually consist of a shaft which carries a cone having large teeth at the feed inlet and small teeth or furrows at the product outlet

h)*Smooth-Roll crushers* consist of two rolls of the same diam which revolve toward each other at the same speed. One of the shafts moves in fixed bearings, the other in movable bearings. The distance between the rolls is adjustable, and a nest of powerful springs hold the movable roll to the clearance at which it has been set

Grinding Mills or Grinders may be divided into the following types:

a)*Buhrstone mills* consist of hard circular stones which serve as grinding media. Material from a hopper is fed thru a center hole in one of the stones. It is distributed between the stone faces and ground while working its way to the periphery. The Buhrstone mill is probably one of the oldest types of grinding mill still is use. The Sturtevant Mill Co is one of the manufacturers of Buhrstone mills

b)*Disk Attrition mills* may be considered as modern counterparts of the earlier Buhrstone mill. The stones are replaced by steel disks or abrasive grinding plates rotating at high speeds. Examples of this type of millare Bauer Double Disk mill (Bauer Bros Co), Frigidisc Grinder (Young Machinery Co), and the Sprout-Waldron Attrition mill (Sprout, Waldron & Co)

c)*Dispersion and Colloid mills* are classed as grinders but they do not do much actual grinding. Their value lies in eleminating coarse particles (a few microns & up), while ensuring breakdown of agglomerates or, in the case of emulsions, the shearing of fluid phases to produce dispersed droplets of ca 1 micron. Some mills of this type are Morehouse (Morehouse-Cowles, Inc), Premier (Premier Mill Corp), Charlotte (Chemicolloid Corp), Tri-Homo Disperser-Homogenizer (Tri-Homo Corp), Manton-Gaulin (Manton-Gaulin Co), and the Viscolizer (Cherry-Burrell Co)

d)*Flash Pulverization mills.* This process for drying & comminuting minerals was introduced in 1945 by the Institute of Gas Technology. It was found that a wide variety of friable materials could be pulverized by introducing them into a stream of gas at mod pressure, and causing the stream to pass thru a nozzle into a zone of lower pressure. When superheated steam was used as the motive fluid, the product was dried to less than 1% moisture content

e)*Fluid-Energy or Jet mills.* These may be classed in terms of the nature of mill action. In one class, the fluid energy is admitted in high-velocity streams at an angle around a portion or all of the periphery of a grinding & classifying chamber. In this class are the Micronizer (Sturtevant Mill Corp), Jet Pulverizer (Jet Pulverizer Co), Jet-O-Mizer (Fluid Energy Processing & Equipment Co), and others. The other class of mills conveys the fluid stream of particles at high velocity into a chamber where two streams impact upon each other. The Majac mill (Majac Inc) and others of somewhat similar structure are in this class

f)*Hammer mills* consist of a rotor shaft carrying hammers (sometimes called beaters) and a housing contg grinding plates or liners. The rotor shaft, which may be vertical or horizontal, usually operates at high speed. The fineness of the product can be regulated by changing rotor speed, feed rate, clearance between hammer & grinding plates, the number & type of hammers used, and the size of the discharge openings. Examples of this type of mill are the Williams Helix-Seal mill (Williams Patent Crusher & Pulverizing Co), Stedman Type A Two-Stage Swing Hammer Grinder (Stedman's Foundry & Machine Works), Jeffrey Type A Swing Hammer Pulverizer (Jeffrey Mfg Co); Mikro-Pulverizer, SP (Semi-Plastic) Mikro-Pulverizer, Mikro-Atomizer & Micro-Bud Pulverizer (Pulverizing Machinery Co); Raymond Screen Pulverizer, Imp Pulver-

izer, Automatic Pulverizer & Raymond Vertical Mill (Raymond Div, Combustion Engrg Co); Blue Streak Dual Screen Pulverizer (Prater Pulverizing Co), Riley Atrita (Reily Stoker Corp), Aero (Foster Wheeler Corp), Rietz Disintegrator (Rietz Mfg Co), Tornado mill (F.J. Stokes Corp), Ftiz mill (W.J.Fitzpatrick Co), Turbo-Pulverizers & Turbo mills (Pullmann Pulverizer Co), Limited mill (Schutz-O'Neill Co), Pulvocron (Strong Scott Mfg Co), and the Entoleter Impact mills (Entoleter, Inc)

g)*Particle-Size Classifiers.* Most crushing & grinding equipment can be operated in "closed circuit " with size classifiers. Material from a machine is conveyed to a classifier, in which large particles are removed and returned to the machine. Particles of the desired size and smaller are discharged as product. By this procedure, a product is obtd with more uniform size distribution than would be obtd by bath or continuous "open-circuit" operation. External classifiers may be used independently as well as externally with grinding mills. The Whizzer (Raymond Pulverizer Div, Combustion Engrg Co), Spinner (Williams Patent Crusher & Pulverizer Co), Gayco (Universal Road Machinery Co), and Whirlwind (Sturtevant Mill Co) are classifiers of this type. Many operations incorporate grinding & classification functions so closely integrated that the classifier is termed an internal type. This type has particular application for dry grinding since the material remains in suspension and is conveyed pneumatically thruout its residence in the mill. The Whizzer classifer is a type particularly applicable to internal use with the Ring-Roller mill

h)*Pin-Type mills.* In contrast to peripheral hammers of the rigid or swing types, this is a class of high-speed mills having pin breakers in the grinding circuit. They may be on a rotor with stator pins between circular rows of pins on the rotor disk, or they may be on rotors operating in different directions. The Alpine Kolloplex (Alpine American Corp) is an example of this type mill

i)*Ring-Roller mills.* These are equipped with rollers that operate in conjunction with grinding rings, in either vertical or horizontal position. Grinding takes place between the surfaces of the ring & rollers. Some examples of this type of mill are the Sturtevant mill (Sturtevant Mill Co), Bradley Hercules Three-Roller mill & Griffin mill (Bradley Pulverizer Co); B & W Pulverizers, Types B & E (Babcock & Wilcox Co); Raymond Ring-Roller mill & Raymond Bowl mill (Raymond Div, Combustion Engrg, Inc) ; and the Williams Ring-Roller mill (Williams Patent Crusher & Pulverizer Co)

j)*Tumbling mills.* A generic term sometimes used in referring to ball, pebble, rod, tube & compartment mills, because of the action of the grinding medium. These mills consist of a cylindrical steel shell, sometimes stone-lined, contg a chge of steel balls or stone pebbles. The shell is rotated horizontally about its axis so that size reduction is effected by tumbling of the balls or pebbles on the material between them. These mills may be operated wet or dry, in either batch or open-circuit use or in closed circuit with size classifiers. Some examples of Tumbling mills are the Compeb, Ball Peb & Rod mill (Allis-Chalmers Mfg Co); Kominuter, Unikom, Unidan & Pyrator (F.L.Smith & Co); Marcy Ball & Open End Rod mills (Mine & Smelter Supply Co), Hardinge Conical, Ball & Pebble mills (Hardinge Co), Foster Wheeler Ball mill (Foster Wheeler Corp), the Attritor (Union Process Co) and others. A Ger development in "vibrating mills" has received considerable notice in the literature. Ball action in a cylindrical shell results from oscillating or vibrating the shell. Some recent mills of this class on the market are the Vibratom (Schutz-O'Neill Co), Vibrating Ball mill (Allis-Chalmers Mfg Co), and Sweco Vibro-Energy mill (Southwestern Engrg Co)

Crushing & grinding operations, employing the types of equipment described above, are used in the processing & preparation of a wide variety of expl compns and their ingredients

Refs: 1)W.L.Badger, "Elements of Chemical Engineering", McGraw-Hill, NY (1936), 522-57 (Crushing & Grinding) 2)W.H,Walker et al, "Principles of Chemical Engineering", McGraw-Hill, NY (1937), 251-88 (Crushing & Grinding) 3)J.Gross, "Crushing and Grinding", US BurMines Bull **402** (1938), 148pp 4)E.R.Riegel, "Chemical Machinery", Reinhold, NY (1944), 8-40 (Devices for reducing the size of solid particles) 5)C.E.Berry, IEC **38**, 672-78 (1946) (Modern machines for dry size reduction in fine size rang) 6)L.T.Work, IEC **39**, 11-31 (1947) (Crushing & Grinding) (See also succeding "Size Reduction" under Unit Operations)

6)W.Anselm, "Zerkleinerungstechnik und Staub", Deut Ing-Verlag, Düsseldorf (1950), 58pp (Crushing technology & dust) 7)C.Mittag, "Die Hartzerkleinerung", Springer-Verlag, Berlin (1953), 342 pp (Crushing hard materials) 8)Kirk & Othmer **12**, 498-520 (1954) (Size reduction) 9)Perry (1963), Section 8 byL.T. Work & A.L.Stern, "Size Reduction and Size Enlargement"'

Crushing Impact, Sensitivity of Explosives to. G.D.Dorough in Naval Weapons Lab Rept **1805** (April 1962) (Conf) describes the *Susan Test*

Cryogenic Liquid. Cryogenic pertains to very low temps of at least -50° and below. Cryogenic liquids are gases (such as oxygen, nitrogen & argon) which have been liquified at low temps. These liquids are used in missile systems for propulsion, pressurization & refrigeration
Ref: Ord Tech Term (1962), 88

Cryolite, Na_3AlF_6, a double fluoride of sodium & aluminum. It occurs as a white, sometimes red, brown or black vitreous substance in Greenland, Colorado & Russia; and as a synthetic made from fluorspar, H_2SO_4, hydrated alumina & Na carbonate (Refs 1 & 6)

Cryolite is used in *Cordite N* (See in this Volume), other propellants (Refs 5 & 8), and in some pyrotechnic compns (Ref 3), such as French compn for *Signal Pistol, Véry* - $Ba(NO_3)_2$ 57, Al 30, cryolite 6, Sb_2S_3 6 & castor oil 1%
White Star - $Ba(NO_3)_2$ 35, Al 43, Cryolite 10, Sb_2S_3 10 & vaseline 2%
Refs: 1)Hackh's (1944), 235 1a)Davis (1943), 63 & 70 2)Anon, Summary Tech Rept of Div 8, NDRC, Vol 1 (1946), 107 3)Izzo, Pirotecnia (1950), 226 & 228 4)P.Tavernier, MP **38**, 312 & 331 (1956) (Heat of formation at C_v 3604 cal/g) 5)Anon, MIL-STD-286A (1960), Method **307.1.2.** (Method for detg cryolite content of propInts) 6)CondChemDict (1961), 316 7)Conf "Propellant Manual", SPIA/M2 (1962), Unit Nos 21, 284, 353, 523 & 604 (Not used as a source of info) 8)Encyclopedia **2** (1962), p C-34, Table V (Use of cryolite in Propellants)

Cryoscopic Method. This is a method of determining the molecular weight (mw) of an organic substance by accurate determination of the depression, in freezing point of a solvent, produced by a small quantity of the solute in a solution of known concentration
Refs: 1)Hackh's (1944), 235 2)N.D.Cheronis & J.B.Entrikin, "Semimicro Qualitative Organic Analysis", Interscience, NY (1957), 145-49 3)A.Weissberger, "Physical Methods of Organic Chemistry", Interscience, NY, Vol 1 (1959), 247-48

Cryostat. This is a refrigerated bath held at a constant low temp. Its construction & operation vary widely depending on temp, reqd accuracy of temp regulation, and refrigerant. The simplest way to hold constant temps is to use a bath medium which either melts or boils at the desired temp. Ice-salt mixts are used for temps ca 0°. Egly (Ref) presents a table of selected materials, readily available & inexpensive, which are recommended for use in a wide temp range. If a cryostat is to be held at the same temp for extended periods of time, a bath liq should be chosen which freezes well below the desired temp, and the bath temp should be controlled. A large number of liq-hydrogen & liq-helium generators and cryostats are described in recent literature
Refs: 1)Kirk & Othmer **8**, (1952), 488 2)A. Weissberger, "Technique of Organic Chemistry", Interscience, NY, Vol **3**, Part 2 (1957), 166-70; R.S.Egly, "Cryostats"

Crystal. A smokeless shotgun propInt made by Curtis & Harvey. It contains NC (insol in eth-alc mixt) and is intended for cheap loading. The chge wt is 33 grains
Ref: Marshall, Dict (1920), 26

Crystal. See under Crystallography, Crystals and Chrystallochemical Analysis

Crystallization. The act or process of changing a substance from the dissolved, molten, liq, or gaseous state to a solid in the form of crystals. It may be accomplished either by precipitation of a solid material from soln, or by slow cooling of a liq material. One important advantage of crystallization over other methods (such as total evapn of solv) is that pure crystals can be obtd from a soln contg a relatively high percentage of impurities, provided that mixed crystals are

not formed

Many chemical industries use crystallization techniques at the present time. In the expl industry, nearly all expls, except liquids such as NG, are obtd in crystalline form (PA, Tetryl, TNT, PETN, LA, MF, AN, $KClO_3$, $KClO_4$ & others)

Some selected Refs on crystallization are given below. See also Crystallization of Explosives and Crystallography, etc

Refs: 1)W.L.McCabe, IEC **38**, 18-19 (1946) (Crystallization and succeeding years under Unit Operations) 2)Struther, Wels Corp, "Krystal Crystallization Equipment", Bulletin **50A**, Warren, Pa (1948) 3)Kirk & Othmer **4** (1948), 619-36; A.R.Thompson, Crystallization 4)A.Weissberger, "Physical Methods of Organic Chemistry", Interscience, NY, Vol 1 (1950), 363-84; R.Tipson, Crystallization and Recrystallization 5)V.D.Kuznetsov, "Kristally i Kristallizatsiya", Gosudarst Izdatel'stvo-Tekh-Teoret Lit, Moscow (1953), 411 pp 6)J. Reilly & W.R.Rae, "Physico-Chemical Methods" Van Nostrand, NY, Vol **2** (1954), 207-14 7)J. González del Tánago, "Evaporación y Cristalización", Ed Dossat, Madrid (1954), 296 pp 8)A.Weissberger, "Physical Methods of Organic Chemistry", Interscience, NY, Vol 1 (1959), 345-51; E.L.Skau et al, Chap 7, Sect V 9)V. L.Singleton, "A Crystallization Technique", Chem Anal **51**, 18 (1962) 10)Perry (1963), Section 17, 7-23

Crystallization of Explosives. The physical props of expls are detd to a large extent by their crystallographic props. The fact that large amts of RDX can be successfully used in expl compns is due to controlled crystn conditions which give proper size distribution & shape of the RDX crystals. Also different crystal forms of a given expl can behave as if they were different expls. HMX is a good example to illustrate this: the *beta* form of HMX is a safe HE, whereas the other three polymorphic forms are sufficiently sensitive to be useful as primary expls. Another example of the importance of controlling crystal structure is found in LA. Care must be taken to be sure that *alpha* LA is formed rather than beta LA. There is also a critical crystn velocity above which the growing LA cryst will detonate spontaneously. A study of the crystn of TNT has led to improved production methods for loading all calibers of HE rounds (Refs)

Bowden & Singh (Ref 7) studied the effect of size of primary expl crystals on their expl decompn when heated. Experiments with Cd azide, alpha LA & Ag azide showed that crystals having a thickness greater than a critical value always exploded when kept at a fixed temp. Crystals smaller in thickness than the critical value decompd slowly at that same temp

These examples of crystal studies in the expl field are but a few of many applications that have been reported. Propellants, primers, pyrotechnics & many HE's have been greatly improved by the development of processes which control crystal growth & stability of the individual materials. Some further info may be found in the Refs listed below. See also Crystallography, Crystals, etc

Refs: 1)J.R.Johnson, "Crystallographic Studies of RDX, HMX and Related Compounds" OSRD Rept **694** (July 1942) 2)J.R.Johnson, "The Sensitivity of RDX, HMX and RDX-HMX Mixtures", OSRD Rept **797** (Aug 1942) 3)A. T.Blomquist, "The Polymorphism of HMX", ORSD Rept **1227** (Feb 1943) 4)W.E.Bachmann, "The Preparation of HMX", OSRD Rept **1981** (Nov 1943) 5)A.T.Blomquist, "Microscopic Examination of High Explosives and Boosters", OSRD Rept **3014** (Aug 1944) 6)A.N.Campbell & E.J.Pritchard, CanJRes **25B**, 183-97 (1947) & CA **41**, 4647 (1947) [Crystallization velocities of: a)PA - 2,4-Dinitrophenol b)DNPh-TNT c)PA-DNPh-TNT] 7)F.P.Bowden & K.Singh, PrRoySoc **227A**, 23, 30-33 & 35-36 (1954) (Influence of crystal size on explosion) 8)A.B. Bofors, SwedP 152620 (1955) & CA **50**, 7462 (1956); BritP 758590 (1956) & CA **51**, 7014 (1957) (Crystallization of aromatic nitrocompds from HNO_3) 9)W.A.Gey et al, JACS **78**, 1803 -10 (1956) & CA **50**, 12479 (1956) (Linear crystallization of TNT systems) 10)W.C. McCrone, Ordn **44**, 506-07 (1959) (Explosives crystallization)

Crystallography, Crystals and Crystallochemical Analysis. The term *crystal* is applied to a body that is formed by the solidification of a chem element, compd or mixt and has a regularly repeating internal arrangement of

its atoms and often external planes, called faces. *Crystallographic systems* are classifications of crystal forms on the basis of the combinations of interfacial angles and lengths of the axes. The seven classes of crystals are:

a)**cubic** - three equal rectangular axes

b)**hexagonal** - three equal coplaner axes, inclined 60° to each other, and a fourth axis different in length and perpendicular to the other three

c)**monoclinic** - three unequal axes, two of which are inclined, but the third is perpendicular to the other two

d)**orthorhombic** - three unequal rectangular axes

e)**tetragonal** - three rectangular axes, two of which are equal and different in length from the third

f)**triclinic** - three mutually inclined and unequal axes, all three angles unequal, and other than 90, 60 or 30°

g)**trigonal** - three equal and equally inclined axes

Crystallography is the science of crystals which deals with the study of their internal, molecular & atomic arrangements

Crystallochemistry or Crystal Chemistry is the application of chemical science to the study & identification of various crystals. *Crystallochemical Analysis* is the name given by Fedorov (Ref 2) to the identification of noncubic cryst substances by means of goniometric data. The method is comparatively simple, rapid & requires a very small amt of substance. According to Donnay (Ref 30), "although, since the advent of x-ray diffraction methods in crystallography, the scope of crystallochemical analysis might well be expanded to include all methods of identification based on crystallographic constants, it seems best to retain the term in its original sense". Crystallochemical analysis may be applied in the examination of expl mixts contg crystalline ingredients

Refs: 1)P.Groth, "Chemische Krystallographie" 5 Vols, Englemann, Leipzig (1906-19) 2)E.S. Fedorov, ZKrystMin **50**, 513-75 (1912) & CA **6**, 3252-53 (1912); ZKrystMin **52**, 97-136 (1913) & CA **7**, 2714 (1913); and ZKrystMin **53**, 337 -88 (1914) & CA **8**, 1369 (1914) 3)E.S.Fedorov, Izvestiya Akademii Nauk **1916**, 435-54 & 547-53; Ca **12**, 8-10 (1912) (Fundamental law of crystallochemistry) 4)E.S.Fedorov et al, "The Crystal Kingdom; Tables for Crystallo-chemical Analyses", AcadSci (Russia), Petrograd (192?) & CA **18**, 215 (1924) 5)A.E.H.Tutton, "Crystallography and Practical Crystal Measurement", Macmillan, London, 2 Vols (1922) 6)A.K. Boldyrev, MémSocRusseMin **53**, 251-342 (French résumé, 337-42 (1924) & CA **21**, 1421 (1927) (Modification of Fedorov's method) 7)G. Friedel, "Leçons de Cristallographie", Berger-Levrault, Paris (1926) 8)E.S.Fedorov, "Crystallography", (in Russian), AkadNauk, Petrograd (1926) 9)W.Voigt, "Lehrbuch der Krystallphysik", Teubner, Leipzig (1928) (Reproduced by Edwards Bros, Ann Arbor, Mich) 10)T.V.Barker, "The Study of Crystals", and "Systematic Crystallography", Murby, London (1930) 11)P.P.Ewald et al, "Strukturbericht 1913-1939", AkadVerlagsges, Leipzig 7 Vols (1931-43) (Reproduced by Edwards Bros, Ann Arbor, Mich) 12)J.D.H.Donnay & J.Mélon, AnnSocGeolBelg-Bull **57**, 39-52 (1933-34) & CA **28**, 6603 (1934) (Application of the method of Barker in studying a cryst homeomorphous series) 13)V.Goldschmidt, "Kursus der Kristallometrie", Bornträger, Berlin (1934) 14)P.Terpstra et al, Z Krist **87**, 281-305 (1934) & CA **28**, 6603 (1934) (Deficiencies of Barker's method) 15)R.W.G. Wycoff, "The Structure of Crystals", Reinhold, NY (1935) 16)A.K.Boldyrev & V.V.Dolivo -Dobrovolskii, ZKrist **93**, 321-67 (1936) & CA **30**, 6258 (1936) (Determinative tables for crystals) 17)J.D.H.Donnay, ZKrist **94**, 410 -12 (1936) (Über die Bestimmungstabellen für Kristalle) 18)W.A.Wooster, "Textbook on Crystal Physics", Macmillan, NY (1938) 19)C.W.Stillwell, "Crystal Chemistry", McGraw-Hill, NY (1938) 20)P.Terpstra, Rec **57**, 893-904 (1938) & CA **32**, 8869 (1938) (Cryst measurements with a new model two -circle goniometer, especially adapted to use in chem labs) 21)M.W.Porter & R.C.Spiller, Nature **144**, 298-302 (1939) (Crystallochemical analysis) 22)R.C.Evans, "Crystal Chemistry", Cambridge Univ Press, London (1939) 23)H.F.Miller & S.E.Q.Ashley, Jr., "Crystallochemical Analysis", Data Folder from Pittsfield Lab of Gen Elec Co (1941) 24)A. N.Winchell, "The Optical Properties of Organic Compounds", Univ of Wisconsin Press,

Madison, Wisconsin (1943) 25)E.E.Walstrom, "Optical Crystallography", Wiley, NY (1943) 26)A.F.Wells, "Structural Inorganic Chemistry", Clarendon Press, Oxford (1945) 27)C. W.Bunn, "Chemical Crystallography, An Introduction to Optical and X-ray Methods", Oxford Univ Press, London (1945) 28)F.C.Phillips, "An Introduction to Crystallography", Longmans Green, London (1946) 29)F.A.Bannister, Nature **157**, 234-36 (1946); JSciInstr **23**, 36 (1946) (Physical methods for the identification of materials) 30)A.Weissberger, "Physical Methods of Organic Chemistry", Interscience, NY, Vol 1 (1945), 531-58 & Vol 1 (1949), 878-971; E.E.Jelley, Crystallographic Microscopy; Vol 1 (1945), 561-83 & Vol 1 (1949) 1017-39; J.D.H. Donnay, Crystallochemical Analysis; and Vol **1** (1949), 983; M.A.Peacock, Determination of Crystal Form 31)R.W.G. Wycoff, "Crystal Structures", Interscience, NY, Vol **1** (1948); 1st Supplement to Vol 1 (1951); Section 3 (1953); 2nd Supplement (1953) 32)Kirk & Othmer, Vol **4** (1949), 636 -62; A.F.Wells, Crystals 33)Armour Res Foundation of IIT, "Crystallographic Properties of Primary Explosives", Summary Rept **11** (1955). [Ord Proj TA3-5101, Contract DA-11-022-ORD-(P)-18] 34)J.F.Nye, "Physical Properties of Crystals", Oxford Univ Press, NY, (1956), 250pp 35)J.Krc, Jr, T.A.Erikson & R.J.Hinch, Jr, "Investigation of Crystallographic Properties of Primary Explosives", Armour Res Foundation, IIT Quarterly Rept No **1** (1958), [Ord Proj TB3-0115A, Contract DA-11-022-501-ORD-2731] 36)W.C.McCrone Assoc Inc, "The Crystallography of Explosives", Final Rept (1962) (Contract DA-11-022-ORD-4090) 37)R.C.Evans, "An Introduction to Crystal Chemistry", Cambridge Univ Press, NY (1964)

CS. A US CWS code designation for *o-chlorobenzylidene malononitrile* (called 2-Chlor -benzylidenmalonsäure-dinitril in Ger), $Cl.C_6H_4.CH:C(CN)_2$; col crysts (from alc, CS_2 or CCl_4), mp 95-96°, bp 310-15°; sol in acet & benz; sl sol in w; can be prepd by condensing 2-chlorobenzaldehyde with malononitrile. The physiological effect of this compd is to cause sneezing & skin irritation; the smarting effect is intensified by washing. Most of the discomfort can be avoided if a gas mask is worn whenever the dry solid is handled (Refs 1 & 2)

As a CWA (Chemical Warfare Agent), CS can be dispersed in burning & bursting grenades, by hand-held dispersers (modified flame thrower), helicopter-mounted dispersers, and skid mounted dispersers. Its chief use is for riot & mob control; however, military applications are indicated (Ref 5)

See also other CWA under Chemical Agents, Vol 2, pC165-R

Refs: 1)Beil **9**, [641] 2)B.R.Corson & R.W. Stoughton, JACS **50**, 2828-30 (1928) 3)H.G. Sturz & C.R.Noller, JACS **71**, 2949 (1949) (Substituted benzylidenemalononitriles) 4)S.Patai & Y.Israeli, JChemSoc **1960**, 2020 (Prepn of p-chlorobenzylidenemalononitrile) 5)M.V.Malin, Chem Warfare Lab Spec Publication **4-20** (June 1960)

CSC. A British double-base propellant called Cordite Solventless Carbamite. See Cordite SC under CORDITE

CSE (Fr). Abbr for Coefficient of Self-Excitation. See under Gap Test, Vol 1, p XIV

CSE (Fr). Abbr for Commision des Substances Explosives

CSE (Explosifs). These expls were developed after WWII at the laboratory of Commission des Substances Explosives (CSE) in France. One group of expls based on chlorates or perchlorates is described uhder CHEDDITES OR STREETITES. See compn designated 55-CSE -1948 & 58-CSE-1948 in Table 4, Vol 2, p C159. These are called "Explosifs du type OC"

Examples of another group of expls based on AN are the following (Ref 1):

a)*63-CSE-1949*: AN 67, Pentolite (20/80) 12 & Al powd 21%; CUP (qv) value 147%

b)*68-CSE-1949*: AN 20, PETN 20, DNN 1.5 & NaCl 58.5%; CUP value 43%

c)*123-CSE-1948*; AN 17, PETN 23 & NaCl 60%; CUP value 43%

d)*133-CSE-1948*: AN 22, PETN 20, TNT 3 & NaCl 55%; CUP value 46%

The above compns all belong to the "Explosifs du Type N". Compns b), c) & d) are safe for use in any fiery coal mine. The mix desig-

nated 123-CSE-1948 was authorized for use in 1950 under the official name Explosif du Type N n° 63, and the 133-CSE-1948 as Explosif du Type N n° 64

Examples of a class of CSE expls contg silicon (silicium) and called "Explosifs au Silicium", are given below. Their compn & props are compared with similar expls not contg silicon (Ref 3):

Composition & Properties	Designation of Explosive n°78 CSE-1950	n°88 CSE-1950	Nn°0	Nn°31
AN(NH_4NO_3)	79.0	82.0	78.7	78.5
Silicon(pulverized)	5.5	7.4	-	-
Aluminum (granular)	-	-	-	9.2
TNT	-	10.6	21.3	-
Pentolite (20/80)	15.5	-	-	12.3
Density, g/cc	1.40	1.24	1.40	1.40
Gap test (CSE), cm	-	6	9	-
Sensitivity to initiation, g of MF	1.50	-	-	1.50
Trauzl test (CUP) PA = 100	127.5	124	121.5	136.5
Velocity of deton, meters/sec	4700	4000	4460	4150

The following compns are representative of a group of plastic expls developed by the CSE (Ref 2):

a) *33-CSE-1951*; RDX 75, NG 21 & nitropolystyrene 4; semi-plastic

b) *34-CSE-1951*: AN (screen thru No 5 silk) 88 & nitropolystyrene (screened thru No 8 silk) 12%; gravimetric denisty 0.33, packing density 0.65; at d = 0.6 its coefficient de self-excitation (CSE) is 3.5cm, and CUP value 114%. This is a low d coal mining expl

c) *36-CSE-1951*: PETN (pulverized) 82% & polystyrene soln (40%) in liq DNT 18%; density 1.58g/cc; vel of deton 7520 m/sec; readily detonated by No 8 cap

d) *37-CSE-1951*: AN 85 & nitropolystyrene 15%; CUP value ca 120%; O balance ca 0%; low density coal mining expl

e) *38-CSE-1951*: AN 72.25, nitropolystyrene 12.75 & NaCl 15.00%; packing density 0.72, cartridge d 0.75; CSE value 2.5 cm; vel of deton 2720; low density coal mining expl

Several expl compns based on 2-nitro-2-ethyl-1,3-propanediol dinitrate (NEPD) were also prepd & examined in France at the lab of CSE. These expls are called "Explosifs à base de Dinitrate de Nitroéthylpropanediol". Some examples of NEPD expls are given in Table on p C 572 (Ref 4)

Refs: 1)L.Médard, MP **32**, 209-24 (1950) 2)L. Médard, MP **34**, 104-05 (1952) 3)R.Sartorius, MP **34**, 211-12 (1952) 4)L.Médard, MP **35**, 152-53 (1953)

CSP 2 (Chilworth Smokeless Powder, No 2). This is a tubular double-base proplnt which may be considered as a modification of Cordite (qv). It consists of NC (N content 12.70±0.15%) 70.5, NG 23.5, petroleum jelly 5 & Na bicarbonate 1% (Ref 3). The density of the proplnt is 1.56 g/cc. Some other props are calcd by Alvaro Alberto (Ref 2) and applied to the problem of erosion of proplnts. CSP 2 has been known since ca 1902 and has been manufd or adopted by several countries, including Germany, Gt Britain, Brazil (1908), Italy (1910) & Spain. It was manufd in Italy by the Società Italiana Dinamite Nobel, Avigliana under the name *C2(Polvere)* (Refs 4 & 5); and in Spain at the Fábrica de Galdácano of the Union Española de Explosivos under the name *Pólvora sin Humo de Chilworth* (Ref 3). This proplnt has been used in various cannons such as cal 76.2mm, 101.6mm, 15 cm, 30.5cm & others

See also C-2 (Polvere), Vol **2**, p C1-L

Refs: 1)Marshall, Dict (1920), 26 2)Alvaro Alberto, Anais Acad BrasilCienc **14**, 327-42 (1942); **15**, 39-61, 187-89, 239-49 (1943) & CA **40**, 2629-30 (1946) 3)Vivas, Feigenspan & Ladreda **3** (1948), 167-68 & 395-405 4)P. Tavernier, MP **32**, 253 (1950) 5)Admiral Alvaro Alberto, private communication (14 Oct, 1958)

CS Powder. Abbr for Chilworth Smokeless Powder. See CSP 2

C-Stoff. A Ger liq mixt constg of hydrazine hydrate 30, methanol 57 & water 13%. This mixt was used as a rocket fuel (Refs)

Refs: 1)H.S.Seifert et al, AmJPhys **15**, 121-40 (1947) & CA **41**, 6383-85 (1947) 2)J.G.Tschinkel, C & EN **32**, 2586-87 (1954) 3)PATR **2510** (1958), p Ger 32

CU. A double base rocket proplnt developed at the Bishopton Ordn Factory, ERDE. Its

CSE Designation of Explosives

Composition and Properties	79-1950	80-1950	7-1952	8-1952	31-1952	36-1952	38-1952	76-1952	77-1952	78-195
NEPD	6.0	8.0	6.0	36.5	27.6	25.9	31.9	27.6	34.3	32.
AN(NH_4NO_3)	80.6	83.0	82.3	61.0	70.0	57.4	23.6	58.0	51.0	47.
Wood flour	13.4	9.0	11.7	2.5	–	–	3.3	2.0	3.0	10.
Rhodopas HV 2	–	–	–	–	2.0	2.0	5.0	2.0	1.2	1.
Centralite	–	–	–	–	0.4	0.4	0.5	0.4	0.5	0.
PETN	–	–	–	–	–	14.3	–	–	–	–
Na nitrate	–	–	–	–	–	–	35.7	10.0	10.0	9.
Density (packing),g/cc	0.90	0.95	0.90							
Sensitivity to initiation, g of MF	0.40	0.30	0.30							
Gap test (CUP), cm	5	5	5							
Remarks*				Exudes						

*Most of these expls are very difficult to initiate. Some are of unsatisfactory plasticity

compn & props are given in conf "Propellant Manual", SPIA/M2 (1962), Unit No 586

Cube Powder. A smokeless proplnt similar to *Imperial Schultze Powder* which contains NC (made from wood fiber), $Ba(NO_3)_2$ & petroleum jelly
Ref: Cond Chem Dict (1942), 288

Cugnite. A French blasting expl consisting of AN 30, $NaNO_3$ 30, NG 27, NC 0.7, wood meal 11% & $BaSO_4$ 1.3%. This expl was manufd by the Société Française des Explosifs
Ref: Marshall, Dict (1920), 26-27

Cuirasse (Fr). Armor (of a vessel)

Cuirassé (Fr). Armored vessel; battleship

Culasse (Fr). Breechblock

Culverin. A light, long, muzzle-loading artillery weapon developed during the 15th century. A piece used by the Brit Navy in the 16th century had a bore of 5½", a length of 32 calibers & a max range of 2500 paces. It fired an iron shot and belonged to the class of "seventeen-pounders", weighing 17-lbs. There were also lighter culverins, such as *Demi-culverin* (a 9 to 10-pounder), the *Saker* (a 5-pounder), the *Minion* (a 4-pounder) and the *Falkon* (a 2½ to 3 -pounder). The heaviest weapon was called a cannon(qv). The 16th century Brit Navy cannon was a muzzle-loader, having a 7½" bore, 18 calibers long, & a max range of 2000 paces. It fired an iron shot weighing 50-lbs, hence it was called a fifty-pounder
Ref: J.F.C.Fuller, "A Military History of the Western World", Funk & Wagnall, Vol **2** (1955). p 12

Cumaranone. See Coumaranone

Cumaric Acid. See Coumaric Acid

Cumarin. See Coumarin

Cumene and Derivatives

Cumene or Isopropylbenzene (called Isopropylbenzol, Cumol or 2-Pheynl-propan in Ger); $C_6H_5.CH(CH_3)_2$; mw 120.19, H 10.06%; col liq, fr p -96°, bp 152.5°, flash p 102°F (39°C), d 0.864 at 20°, n_D 1.489 at 25°; sol in alc, CCl_4, eth & benz; insol in w; can be prepd by a)interaction of benz & isopropyl iodide b)distilling cuminic acid c)interaction of benzyl dichloride & Zn methyl and d)catalyzed interaction of benz & propylene (Refs 1 & 2). Other props & methods of prepn are given in Beil (Ref 1)

Cumene has a potent narcotic action characterized by a slow induction period. It is a depressant to the central nervous system. The long duration of its action indicates a possible slow rate of elimination, meaning that cumulative effects must be considered. Cumene is thought to have a greater acute toxicity than either benz or toluene (Ref 4)

Cumene is used in org synthesis; as a solvent, diluent, and as an additive to aviation gasoline
Refs: 1)Beil **5**, 393, (191),[306] & {877} 2)Cond Chem Dict (1950), 200 3)Faith, Keyes & Clark (1957), 587 4)Sax (1957), 514

α-Azidocumene, $C_6H_5.C(N_3)(CH_3)_2$; mw 161.20, N26.07%. This compd is described in the original article, which is not currently available to the authors. It is not found in the abstract (Ref 2)
Refs: 1)Beil - not found 2)Y.Yukawa & K. Tanaka, NipponKagakuZasshi **78**, 1049-52 (1957) & CA **54**, 5555 (1960)

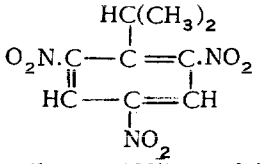
Nitro Derivatives of Cumene

Mononitrocumene, $C_9H_{11}NO_2$; mw 165.19, N 8.48%. Several isomers are described in the literature:
α-Nitrocumene, $C_6H_5.C(NO_2)(CH_3)_2$; oil, bp 224° (dec), d 1.1025 at 20°, n_D 1.5209 at 25° (Ref 1, p 396);
2-Nitrocumene, $O_2N.C_6H_4.CH(CH_3)_2$; pale yel oil, bp 118° at 12mm, d 1.101 at 12°, n_D 1.5259 at 20° (Ref 1, pp [207] & {890}); *3-Nitrocumene*, oil, bp 92° at 3.5mm, d 1.0830 at 25°, n_D 1.5301 at 20° (Ref 2). See also Refs 3, 4, 5 & 6); *4-Nitrocumene,* pale yel oil, bp 128° at 12mm. d 1.0830 at 20°, n_D 1.5361 at 20° (Ref 1, pp [308]&{890}). Other props & methods of prepn are given in the refs
Refs: 1)Beil **5**, 396, [307-08]&{890} 2)M. S.Carpenter et al, JOC **16**, 593, 606 (1951) & CA **46**, 1493 (1952) 3)H.C.Brown & W.H. Bonner, JACS **76**, 605-06 (1954) & CA **49**,

10871 (1955) 4)H.Gilman et al, JOC **19**, 1072 (1954) & CA **50**, 225 (1956) 5)H.Hock & H. Kropf, ChemBer **89**, 2436-38 (1956) & CA **51**, 8026 (1957) 6)J.R.Knowles, JChemSoc **1960**, 4894-95 & CA **55**, 22218 (1961)

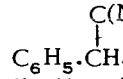
Dinitrocumene, $C_9H_{10}N_2O_4$; mw 210.19, N 13.33%. Only one isomer is known: *2,4-Dinitrocumene,* yel crysts, mp 18.5°, bp 169° at 12mm & 136° at 2mm (Refs 1 & 2); bp 142-43° at 3mm (Ref 4); n_D 1.5528 at 25° (Ref 4); was obtd, in addn to 2-Nitro- & 4-Nitro -cumene, by treating cumene with a mixt of HNO_3/H_2SO_4 at 10-20° (Refs 1 & 2). Wright & Allan (Ref 3) proposed the use of Dinitrocumene as one of the ingredients of a plastic dynamite consisting of: PETN or HNMnt & RDX distributed in a mixt of Dinitrocumene, dinitro-tert-butylbenzene and dinitro-sec -butylbenzene. See also Refs 5 & 6
Refs: 1)Beil **5**, {890} 2)E.C.Sterling & M. T.Bogart, JOC **4**, 25 (1939) & CA **33**, 4236 (1939) 3)H.R.Wright & W.G.Allan, USP 2439328 (1948) & CA **42**, 4350 (1948) 4)C.Hansch & G. Helmkamp, JACS **73**, 3081 (1951) & CA **46**, 3999 (1952) 5)B.M.Wepster, Rec **76**, 352 (1957) & CA **51**, 14612 (1957) (UV spectra & other props) 6)M.Osowiecki et al, Helv **41**, 1621 (1958) & CA **53**, 12343 (1959)

2,4,6-Trinitrocumene,



; mw 255.19, N 16.47%; col ndls, mp 109°; sol in hot alc; difc sol in cold alc; was prepd by nitration of cumene with mixed acid (Ref 1). Absorption spectra data are reported by Liss & Lohmann (Ref 2). Its expl props were not detd
Refs: 1)Beil **5**, 396 2)E.Liss & K.Lohmann, ChemBer **89**, 2547 (1956) & CA **51**, 14577 (1957)

β,β,β,β'-Tetranitrocumene,



; mw 300.19, N 18.67%; wh ndls (from hexane), mp 87-88°; was prepd by adding nitroform to a soln of β-nitrostyrene in MeOH, addg w & heating at 40° for 4 hrs, and cooling & filtering the product. The kinetics of decompn into nitroform & β-nitrostyrene were studied. The decompn was found to be subject to general base catalysis. Its expl props were not detd
Refs: 1)Beil - not found 2)J.Hine & L. A. Kaplan, JACS **82**, 2915-21 (1960) & CA **55**, 17550 (1961)

Monohalogen Derivatives of Cumene

Several monohalogen derivs of cumen are unstable oils or expl substances: *α-Chlorocumene,* $C_6H_5.C.Cl(CH_3)_2$; watery oil, decomp on heating (Ref 2); *α-Iodosocumene,* $OI.C_6H_4.CH(CH_3)_2$, unstable oil (Ref 1); 4-Iodosocumene, $OI.C_6H_4.CH(CH_3)_2$, wh mas, decompg at 165° (Ref 4); and *4-Iodoxycumene,* $O_2I.C_6H_4CH(CH_3)_2$, ndls (from w), explodes at 191° (Ref 4)
Iodoyxcumene, $9_2I.C_6H_4.CH(CH_3L_2$, ndls (from w), explodes at 191° (Ref 4)
Refs: 1)Beil **5**, 395 2)Beil **5**, 395, [307] & {884} 3)Beil **5**, (191) 4)Beil **5** (192)

Cumene Hydroperoxide; Cumyl Hydroperoxide; α,α-Dimethylbenzyl Hydroperoxide or Isopropylbenzene Hydroperoxide, $C_6H_5.C.OOH(CH_3)_2$; mw 152.19, O 21.02%; col oil, having an odor of H_2O_2, bp 65° at 0.18mm, begins to decomp at 165-70°, flash point 198°F, n_D 1.5237 at 20°; sol in org solvs; diffc sol in w; and constricts the respiratory passage when inhaled; was first described by Hock & Lang (Ref 2) who gave details of the oxidation of cumene with dry O in the light of a Hg vapor lamp

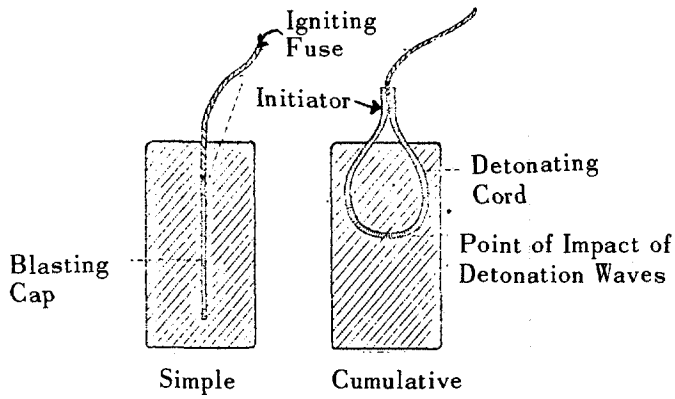
The thermal decompn of Cumene Hydroperoxide has been studied by several authors (cited in Refs 6 & 8). Realization of the potential industrial importance of this compd and its acid-catalyzed conversion to phenol & acetone, caused the appearance of many papers & many patents involving the efficiency of both the oxidation process and the decompn stage (Ref 3)

Anderson (Ref 5) detd the expl props of Cumene Hydroperoxide: *explosion temp,* 170° (smoke); *impact sensitivity,* BM App, 2 kg wt - 100+ cm; *sand test* (brisance) 1.5g sand crushed vs 49g for TNT

Cumene Hydroperoxide is used as a catalyst for free radical reactions, such as polymerization; as a vulcanization accelerator & in rubber-reclaiming flotation; and as a poly-

merization catalyst & ingredient of some rocket proplnts (Ref 9)
Refs: 1)Beil - not found 2)H.Hock & S.Lang, Ber **77B**, 257 (1944) & CA **39**, 3526 (1945) 3)E.J.Vandenberg & G.E.Hulse, IEC **40**, 932-37 (1948) (Redox emulsion polymerization) 4)C.S.Marvel,et al, JPolymerSci **3**, 433-37 (1948) (Emulsion copolymerization of butadiene & styrene) 5)W.Anderson, PACL Rept 127153 (1949) 6)Tobolsky &Mesrobian (1954), 88, 97 & 117 7)Sax (1957), 514 8)E.G.E.Hawkins, "Organic Peroxides", Van Nostrand, Princeton, NJ (1961), 84 & others 9)Conf Propellant Manual SPIA/M2 (1962), Unit Nos 352, 356, 358 & 359 (Not used as a source of info)

Cumulative Initiation or Ignition. (Called Kumulative Zündung or Gegenläufige Zündung in Ger). In order to increase the efficiency of an expl chge, it may be initiated simultaneously from opposite ends, using two elec blasting caps or pieces of detonating fuse (Refs 2 & 3). According to Drs B. & H. Walter (Ref 5), Dr H. Brunswig (See Vol 2, pB318-R) is claimed to be the discoverer of the phenomenon of cumulative initiation. For the detonation of insensitive expls, Brunswig (Ref 1) advocated that advantage should be taken of the fact that at the point where two deton waves meet in a piece of fuse initiated at both ends, the effect is cumulative and much greater than that obtd by deton with a single piece of fuse. See fig



TYPES OF INITIATION

Refs: 1)Marshall **2** (1917), 541-42 2)Stettbacher (1833), 47-8 3)Stettbacher (1948), 135 4)PATR **2510** (1958), p Ger 66 5)Drs Benno & Hans Walter, formerly of Picatinny Arsenal, private communication (July 1963)

CUP or cup (Fr). Abbr for "Coefficient d'Utilisation Pratique". See Vol **1**, pIX

Cupola Furnace Explosion. A violent expln occurred in one of the cupola furnaces of Germany, shortly after starting the blast. The cross section of the shaft had previously become constricted by sprayed molten Fe, so that the 3rd portion of the chge jammed. This formed a cavity in the furnace in which an expl gas/air mixt accumulated & ignited. Calcn showed that a total pressure of 2.9atm developed during the expln. The collapse of the supporting posts, which corresponded to a buckling load of 9 tons, confirmed these calcns. Back pressure forced the gaseous mixt into the blast main, where a 2nd expln destroyed the blower
Ref: A.Sachse, Giessereitech **2**, 17 (1956) & CA **50**, 8418 (1956)

Cupprammonium Solution. This solvent for NC used in determination of its viscosity by the falling sphere method, contains 30±2 g of pure Cu, 165 ±2g of NH_4OH (26 to 25% NH_3) & 10g of sucrose, plus distd water to make a total of 1 liter. Details of its prepn and of the NC vicosity test are described in Ref
Ref: US Military Specification **MIL-C-206A**, (1962), pp 4-6

Cuprates with Organic Cations. Copper (II) Azide, $Cu(N_3)_2$, dissolves more or less easily in many water-sol azides to form normal addn compds, called cuprates. The presence of cuprates can be detected by the blackening produced in water by pptn of $Cu(N_3)_2$. The water-sol cuprates are good conductors of electricity. Because of their N content, they are more expl than the corresponding nonelectrolytes. Compds with org amines as cations are described by Cirulis & Straumanis (Ref 1). See Ref 2

See also Cupric Azide Complexes, Vol 1, p A533-L; and the Copper Azide Explosive Complexes of Cirulis & Straumanis in this Vol, p C 517-R

Refs: 1)A.Cirulis & M.Straumanis, Ber **76B**, 825-30 (1943) & CA **38**, 1970-71 (1944) 2)C.M.Harris, JProcRoySoc NS Wales **85**, 138-41, 145-47 (1952) & CA **47**, 1525 (1953) (Some cuprates from acet soln)

Cuprene or Carbene (Called Cupren or Carben in Ger). In addn to the info given under *Carbene* (Vol 2, p C50-51), the following uses in expls & the indicated Refs should be noted. Foulon (Ref 2) has reviewed the patent literature on the uses of cuprene in the expl industry. Here is discussed the possible use of cuprene as an absorbent for NG or liq O, as a substitute for charcoal in BkPdr, and as an ingredient of chlorate or AN explosives
Refs: 1)Beil **1**, 232,[220] & {914} 2)A. Foulon, SS **27**, 48-50 (1932) & CA **26**, 2596 (1932) 3)Marshall **3** (1932), 227 4)Thorpe **1** (1937) 87 5)Kirk & Othmer **1** (1947), 121-23 6)Ullmann **3** (1953), 67-8

Cuprene Tar. A grn-blue liq by-product, resembling tar, is obtd in the prepn of cumene by passing C_2H_2 (acetylene) over Cu or CuO heated to 200-300°. Yield is 5.5 to 7%, density 0.83 to 0.91. A similar tarry substance is evolved when cuprene is heated above 300° leaving a residue of carbon. The tar is a strongly unsaturated, neutral liq consisting mostly of aromatic hydrocarbons, especially of the C_6H_6 & $C_{10}H_8$ series. Small quantities of anthracene derivs, olefins, styrenes & aliphatic hydrocarbons are also present. This tar can be nitrated directly to form an expl mixt
Refs: 1)P.Schläpfer & O.Stadler, Helv **9**, 185-99 (1926) & CA **20**, 1384 (1926) 2)W. Herzog, Kunststoffe **21**, 49-53 (1931) & CA **25**, 2683 (1931)

Cupric Acetylide. See under ACETYLIDES AND CARBIDES, Vol 1, p A74-R; and in this Volume under List of Copper Compounds, p C 516-L

Cupric Azide. See under AZIDES, INORGANIC Vol 1, p A552ff

Cupric Chlorate. See under CHLORATES, Vol 2, p C188-L

Cupric Compounds. Same as Copper (II) Compounds. See in this volume, p C 515-R

Cupric Explosive Salts. See under AMMINE, Tables D & E, Vol 1, p A280-81; Cupric Azide Complexes, Vol 1, p A533-L; under Cupric Chlorate, Vol 2, p C188-L; under List of Copper Compounds in this Volume; and others, p C 515-R

Cupric Tetramminothiosulfate (called Kupferammonthiosulfat in Ger), $[Cu(NH_3)_4]\ S_2O_3$; dk blue fine crysts, fairly stable on heating; insensitive to light, air & moisture; sol in water; was prepd by mixing hot solns of $Na_2S_2O_3$ & ammoniacal $CuSO_4$ in mol proportions, and cooling the mixt (Refs 2 & 3). Herz (Ref 1) proposed the use of this compd in mixts with oxidg agents as detonating compds for primers & initiators. The following mixts were proposed: $[Cu(NH_3)_4]\ S_2O_3$ 42.7 & $KClO_3$ 57.3% b) $[Cu(NH_3)_4]\ S_2O_3$ 40.5, $KClO_3$ 54.5 & powdered glass 5.0% c) $[Cu(NH_3)_4]\ S_2O_3$ 37.05, PbS_2O_3 7.41, $KClO_3$ 51.54 & powd glass 4.00% d) $[Cu(NH_3)_4]\ S_2O_3$ 27.5, PbS_2O_3 5.5, Sb_2S_3 11.0, $KClO_3$ 53.0 & powd glass 3.0% These mixts are reported to be very brisant and considered suitable to replace MF detong compns. Other advantages noted are low cost, simplicity in prepn, and safety in manuf, handling & use
Refs: 1)E.Herz, SS **7**, 284-85 (1912) & CA **6**, 2841-42 (1912) 2)Mellor **10** (1930, 535 3)Gmelin, Syst Nr **60**, Tl B, Lfg **1** (1958), 591-92

Cuprous Acetylide. See under ACETYLIDES AND CARBIDES, Vol 1, p A72-R; and in this Volume under List of Copper Compounds, p C 516-L

Cuprous Azide. See under AZIDES, INORGANIC Vol 1, p A534, p C 515-R

Cuprous Compounds. Same as Copper (I) Compounds. See in this Volume, p C 515-R

Cuprous Fulminate. See under List of Copper Compounds in this Volume, p C 520-R

Cuprous Sodium Acetylidothiosulfate [called Kupfer (I)-sodium-acetylenidthiosulfat in Ger],

5 $Na_2S_2O_3 \cdot 5Cu_2S_2O_3 \cdot 5Cu_2C_2 \cdot C_2H_2 \cdot 10H_2O$; brick-red solid, begins to decomp ca 140°, on heating in air it burns like gun powder; was obtd when a soln of $Na_2S_2O_3$ was added to cuprous acetate and acetylene passed thru the soln; the ppt was washed & dried. Whin its aq soln was treated with either K or Na hydroxide, a brn ppt was obtd which exploded more violently than the red compd

Refs: 1)K.Bhaduri, ZAnorgChem **76**, 419-21 (1912) & CA **8**, 304 (1914) 2)Gmelin, Syst Nr **60**, Tl B, Lfg 2 (1961), 648

Cups, Percussion or Primer; and Caps. They are small metallic cups (or caps) contg "primer compositions" which are covered with a paper disc, and a metallic "anvil" (See Vol 1, p A473-L). These cups are part of "explosive trains". On firing a weapon, the base of the cup is struck by the firing pin of the percussion lock. This action ignites by impact the primer mixt in the cup producing a flash, which in turn ignites either the proplnt (if the cup is part of a primer used in a propellant train), or the primer mixt of a detonator or delay element (if the cup is part of a primer used in an artillery ammunition train)

The invention of the percussion cap is attributed to an Englishman, Joseph Egg, who tried at the beginning of the 19th century to utilize MF, described in detail by Edward Howard (1800) but first prepd by John K.von Löwenstern (1630-1703). It is also probable that J. Shaw of Philadelphia, USA, independently invented similar cups in 1815. The Paris gunmakers, Prélat & Deboubert, patented in 1820 primer cups filled with Ag or Hg fulminates. However, the first practical use of such cups is attributed to E. Wright (ca 1823) and F.Joyce (1824). Percussion & primer caps were a great improvement over the old flintlocks. The first priming compns contd MF mixed with chlorates & abrasives, some of which are still used today, although more modern formulations, such as those contg LSt, are available in many countries

Refs: 1)Marshall **1** (1917), 37 & **2** (1917), 516-20 2)Colver (1918), 513 3)Marshall **3** (1932), 160 4)Hayes (1938), 574 5)Ohart (1946), 51 6)TM 9-1910 (1955), 113-18 & 209

Curds (called Caillebotte in French). The white ppt obtd by coagulating sour milk. It is used either directly, under the name "cottage cheese", or it may be processed into various kinds of cheese. Nitration of the dried product yields an expl ingredient called *Nitrocurds* (Nitrocaillebotte in Fr). Expls using Nitrocurds were proposed by Sjöberg (1886-87) as follows:

a)Nitrocurds 30, AN or Amm oxalate 55, oil 10 & naphthalene 5%

b)Nitrocurds 3, $KClO_3$ 27, AN or Amm oxalate 55, oil 10 & naphthalene 5%

Refs: 1)Cundill (1889) in MP **6** (1893), 47 & 95 2)Daniel (1902), 493 & 717

Cureña, Afuste or Montaje del cañón (Span). Gun carriage (mount)

Curie, Marie Sklodowska. (1867-1934). Physical chemist, born in Warsaw, received doctorate at the Sorbonne in 1904; succeeded husband, Pierre, as professor of physics at the Sorbonne in 1906. In 1911, Mme Currie was awarded the Nobel prize for chemistry for work on radium & its compds

Refs: 1)E.Curie, BritJRadiol **271**, 409 (1950) & CA **44**, 9197 (1950) (Discovery of radium) 2)A.Kerschagl, "Mme Curie. Pierre Curie", Springer-Verlag, Vienna (1950), 89pp 3)A. Dorabialska, Wiadomości Chem **8**, 305-16 (1954) & CA **49**, 730 (1955) (Biographical sketch with portrait) 4)I.J.Curie, "M.P.S. Curie", Państwowe WydawnNauk, Warsaw (1954), 685pp 5)I.Zlotowski, Przemysl Chem **33**, 553-55 (1954) (Survey of the development of radioactivity & its applications) 6)G. Collignon, "Marie Curie", Barmerlea, London (1955), 154pp 7)M.L.Schmied, "Die Magischem Strahlen. Lebensweg einer Forscherin (Marie Curie)", Neues Leben, Berlin (1955), 110 pp 8)W.Geddie & J.L.Geddie, "Chambers's Biographical Dictionary", Macmillan, NY (1957), 267 9)"Webster's Biographical Dictionary", Merriam Co, Springfield, Mass (1959), 375

Curie, Pierre (1859-1906). French chemist, educated at the Sorbonne, conducted researches on piezoelectricity & magnetism, and became professor of physics at the Sorbonne. Pierre is known expecially for his work with his wife, Maria, on radioactivity leading to their discovery of polonium & radium for which they were awarded with A.H.Becquerel the 1903 Nobel

prize for physics
Refs: 1)H.S.Klickstein, JChemEduc **24**, 278 -82 (1947) (Pierre Curie. An appreciation of his scientific achievements) 2)A.Kerschagl, "Mme Curie. Pierre Curie", Springer-Verlag, Vienna (1950), 89pp 3)I.Zlotowski, Przemysl-Chem **33**, 553-55 (1954) (Survey of the development of radioactivity & its applications) 4)M. S.Curie, "Pierre Curie", Denoël, Paris (1955), 125pp 5)W.Geddie & J.L.Geddie, Chambers's Biographical Dictionary", Macmillan, NY (1957), 267 6)"Webster's Biographical Dictionary", Merriam Co, Springfield, Mass (1959), 375

Curing of Propellants. Amer term equivalent to the Brit "Ageing of Propellants". See Vol 1, p A112-L

Curme, Jr, George Oliver (1888-). Amer chemist recognized as the leading authority on aliphatic chemistry and heads the list of those who have made the US the leader in org technology. He was born in Mount Vernon, Iowa, studied at Northwestern Univ, Harvard & the Univ of Chicago where he obtd the PhD degree. The fundamental research work conducted under his direction at the Mellon Institute of Industrial Research (1914-19) resulted in the formation of Carbide and Carbon Chemicals Corp (1920). Besides receiving tribute from the chem profession in the Chandler Medal (1933), the Perkin Medal (1935), the Elliott Cresson Medal (1936), and the Willard Gibbs Medal (1944), Curme witnessed the coml practice of his discoveries expand to an annual production of nine billion pounds in 1948 (Ref 1)
Refs: 1)A.D.McFadyen, C & EN **26**, 742 (1948) 2)American Men of Science, Bowker Co, NY, 11th edit (1965), p 1072

Current Non-Permitted Explosives (Brit). See under Coal Mining Explosives in this Volume; and in the books of Taylor (1952), p 14 and of Taylor & Gay (1958), p 26

Current Permitted Explosives (Brit). See under Coal Mining Explosives in this Volume; and in the books of Taylor (1952), pp 20-21 and of Taylor & Gay (1958), pp 97 & 103-06

Curtis & André Powder. Between 1897-1900, Curtis & André patented several expl compns:
Blasting expl of 1897: AN 92.5, wood flour 4.0 & starch 3.50%
Propellant for arms of small caliber (1900): Amm picrate 55.6, $Ba(NO_3)_2$ 33.3, KNO_2 2.8 & NC (13%N) 8.3
Blasting expl of 1900: "slow mixt" 88 & NC 12%. The slow mixt consisted of $Ba(NO_3)_2$ 62, KNO_3 20, paraffin 12 & powd coke or charcoal 6%
Refs: 1)Daniel (1902), 175-76 2)Giua, Trattato **6** (1959), 387

Curtis & Durnford Powder. There was patented in 1900 a method of prepg smokeless proplnts which consisted in partial drying of NC to about 15% H_2O content, then gelatinizing it with acet and blending with other ingredients. Some examples of the compns reported are as follows: a)NC 75 & chalk 25% b)NC 50, KNO_3 & charcoal 50% c)NC 75, chalk 15, KNO_3 & charcoal 10% d)NC 80, chalk 12, KNO_3 & charcoal 8%
Refs: 1)Daniel (1902), 176-77 2)Giua, Trattato **6** (1959), 387

Curtisite. A Brit coal-mining expl formerly on the "Permitted List". It was made by Curtis & Harvey and consisted of: AN 88, TNT 8 & MNN 4%. A modification of compn, called *Super-Curtisite*, enabled it to pass the Rotherham Test. The modified compn contd: AN 38.5, KNO_3 29.5, TNT 10 & Amm chloride 22%. The limit chge was 16oz & power (by ballistic pendulum) was 2.71" vs 3.27" for Gelignite contg 60% NG
Refs: 1)Barnett (1919), 132 2)Marshall, Dict (1920), 27

Curtius Rearrangement. A chem change resulting in the formation of a *primary amine* (RNH_2) or a *urethane* ($RCOONH_2$) from an acid azide. The process may be carried out by treating a sodium azide (NaN_3) in toluene, or by treating an ester successively with hydrazine & nitrous acid, and hydrolyzing the acid azide by heating with HCl. The isocyanate is an intermediate product (Ref 3):

$$RCOCl + NaN_3 \longrightarrow RCON_3 + NaCl$$
$$RCON_3 \longrightarrow RNCO + N_2$$
$$RNCO + H_2O \longrightarrow RNH_2 + CO_2$$

See also Refs 1 & 2 and others in CA
Refs: 1)F.L.Scott et al, "Azide Transfor-

mations", Nature **170**, 922-23 (1952) & CA **47**, 9923 (1953) 2)E.Lieber et al, "Migration of Nitrogen in the Schmidt Reaction", JACS **75**, 2023 (1953) & CA **49**, 5305 (1955) 3)"International Encyclopedia of Chemican Science", Van Nostrand, Princeton, NJ (1964), 316

Curtius, Theodor (1857-1928). *Ger scientist* who published many papers on the prepn & props of various org compds, many of which are explosive. Curtius was editor for many years of the Journal für Praktische Chemie
Refs: 1)A.Darapsky, ZAngChem **40**, 581-83 (1927) (Biographical note) 2)H.Wieland, Z AngChem **41**, 193-94 (1928) (Obituary) 3)A. Darapsky, JPraktChem **125**, 1-22 (1930) (A review of Curtius' work, with photograph)

Cushion Blasting. The stemming or tamping by means of various types of ceramic plugs & wedges of the expl chge used in shotholes of coal mines. The object of stemming is, of course, to close up the shothole & resist ejection to such an extent that the energy developed by the expl chge is utilized effectively in breaking us the coal to be blasted (Ref 3). This subject is discussed and the fundamentals reviewed by Denues (Refs 1 & 2)
Refs: 1)A.R.T.Denues, US Bur Mines RI **3510** (1940), 25pp & CA **34**, 5662 (1940) 2)A.R.T.Denues, US Bur Mines RI **3674** (1943), 37pp 3)Taylor & Gay (1958), 163-64 4)Blastors' Hdb (1966), 402-08

Cut Holes. Cut holes are fundamental to all underground blasting operations. The first & most difficult step in driving any heading is to make an opening into the solid ground, usually in the center of the face and as deep as practical. This opening is called the cut and is the most important part of the blast since the remainder of the holes cannot break effectively unless the cut comes out completely. There are three different types of cuts:
a)*the angled cut,* in which holes are drilled at an angle to the face to provide as much freedom as possible
b)*the burn or shatter cut,* in which a number of closely spaced holes are drilled straight into the face and only part of them blasted and c)*the combination* of these two. A few extra minutes spent in drilling a cut properly may mean the difference between breaking a full round or obtaining only a small part of the desired advance
See also Cut Shots
Ref: Blasters' Handbook (1958), 293ff

Cutocellulose. See under Adipocelluloses and Cutocelluloses, Vol 1, p A104-R

Cutoff Explosive Charge is that explosive charge in a dependent or secondary borehole that is unburdened or exposed as a result of the rock movement caused by a prior charge
Ref: Dr. R.W.Van Dolah, Research Director, Explosives Research Center, US Bur Mines, Pittsburgh, Penna; private communication, August 17, 1964

Cutoff Explosive Charge, Hazards of in Multiple Blasting. is discussed by J. Hagy et al in US Bur Mines Rept Inv **5613** (1961)

Cut Powders. These were formerly made by cutting BkPdr press-cakes into cubes. The glazing, stoving, finishing & blending were very much the same as for granulated powders, except that the stoving had to be continued for 30 hrs at 55°. These powders were designed for use in large cannons, 6 to 12-inch bore, but have since been replaced by smokeless propInts
Ref: Marshall **1** (1917), 84

Cut Shots & Drifting. Drifting is the coal-mining term given to the driving of underground tunnels thru stone. The normal practice is to fire a preliminary number of shots to blow out a cavity from the rock face. This cavity is called the cut. Its purpose is to provide an addnl free face for subsequent shots. The cut is then enlarged by 2 or 3 rings of "easer" shots, and the drift is dressed to its correct size by a final round of "trimmer" shots. The efficiency of blasting in stone depends primarily on the effectiveness of the cut shots
See also Cut Holes
Ref: McAdam & Westwater (1958), 119

Cutters. See under Cartridges and Cartridge -Actuated Devices, item c)*Special Purpose Devices* in Vol 2, p C71-R

Cutting. In addn to *Crushing and Grinding* (qv), there are other methods of size reduction & techniques of a specialized nature which take advantage of unique props of solid materials. Size reduction by shear cutting is employed where materials are tough or fibrous, such as wood chips, cotton, rags & NC. The most commonly used cutting machine is called the *Rotary Knife Cutter.* It consists of a rotar with knives spaced uniformly on the periphery so as to cut against stationary knives on the casing. The loading hopper and screens, which control the max particle size, constitute a cylindrical encasing for the rotor

Some examples of rotary cutting machines are the following: *Fabricated-Steel Rotary Knife Cutter* (Sprout, Waldron & Co); *Giant Dicing Cutter* (Taylor, Stiles & Co); *Mikro-Chipper* (Pulverizing Machinery Co); and others by Paul O.Abbe Engrg Co. Hollander & Jordan beaters for pulping NC may also be classed with these cutters

Ref: Perry (1963), Section 8 byL.T.Work & A.L.Stern, "Size Reduction and Size Enlargement"

Cutting Charge. See under CHARGE, Vol **2**, p C150-R

Cutting of Metal Plates with High Explosive Charges in analyzed & discussed by W.E. Drummond, JApplMechanics **25**, 184-88 (1958)

CYANAMIDE AND DERIVATIVES

Cyanamide, Carbondiimide or Cyanogenamide (called Cyanamid, Kohlensäure-amid-nitril or Carbodiimid in Ger), HN:C:NH or N:C.NH_2; mw 42.04, N 66.64%; col deliq ndls, mp 44° -46°, bp 140° at 19mm, flash p 285°F, d 1.282 at 20°; sp heat 0.547 at 20°, heat of fusion -2.1kcal/mol at 44°, Q_{form} -88 kcal/mol at 25°, Q_{comb} 172kcal/mol; v sol in w, alc & eth; sol in chlf & benz; sl sol in CS_2; prepd commercially by acidifying calcium cyanide with either sulfuric or CO_2 in water. Other props & methods are given in Beil (Ref 1)

Cryst cyanamide slowly polymerizes on standing to dicyandiamide; reacts with Amm salts to form the corresponding guanidine salts. Cyanamide attacks the skin like a caustic alkali and may cause eczematous dermatitis (Ref 9)

Metallic cyanamides are used as combustion catalysts in some composit proplnts (Ref 10)

Cyanamides may be identified by treatment with $AgNO_3$. A yel ppt, insol in w & NH_4OH, indicates the presence of cyanamide. Thiourea interferes, giving a blk ppt. In this case, add to the soln contg cyanamide a saturated Pb acetate soln followed by strong NH_4OH. A yel ppt indicates the presence of cyanamide, while a wh ppt indicates its absence (Refs 1a & 8a)

Refs: 1)Beil **3**, 74, (36), [63] & {149} 1a)L. A.Pinck, IEC **17**, 459-60 (1925) 2)J.A.Lee, ChemMetEngrg **38**, 564-67 (1931) 3)Franklin (1935), 93-7 4)Davis (1943), 369ff 5)Kirk & Othmer **4** (1949), 663-72 6)V.Grignard, Ed, "Traité de Chimie Organique ", Masson, Paris, Vol **14** (1949), 200-18 (A.Guillaumin) 7)Inorg Synth **3** (1950), 39-43 (L.A.Pink & J.M.Salisbury) 8)Cyanamide, New Product Bull, Amer Cyanamide Co, NY, Coll Vol **1** (1949) 8a)G. Bourjol & Mme Teindas, MP **31**, 60-1 (1949) 9)Sax (1957), 521 10)W.G.Stanley & W.A.Proell, USP 2988437 (1961) & CA **55**, 25258 (1961) [AN 73.3, oxidizable binder (cellulose acetate 1p & ethyleneglycol diglycolate polyester 2p) 24.7 & Cu cyanamide 2% are mixed and pressed into grains to give a burning rate of 0.12"/sec at 1000lb/sq in under N]

Salts and Other Derivatives of Cyanamide

The following metallic salts of cyanamide: *Barium,* $BaCN_2$; *Cerium,* $Ce(CN_2)_3$; *Lead,* $PbCN_2$; *Magnesium,* $MgCN_2$; *Mercuric,* $HgCN_2$; *Silver,* Ag_2CN_2; *Sodium,* $NaCHN_2$; and *Strontium,* $SrCN_2$; are some examples of those described in the literature (Ref 1). The prepn of metallic cyanamides is the subject of a patent issued to Sakai & Aihara (Ref 11). *Calcium Cyanamide* (sometimes called simply Cyanamide), $CaCN_2$; mw 80.11, N 34.97%; col rhmb crysts, mp 1300° (sublimes >1150°); currently manufd by passing gaseous N thru finely ground Ca carbide, preheated to 1000-1100° (Ref 1). Ca cyanamide acts locally on the skin as a primary irritant; the fatal dose, by ingestion, is estimated to be 20 to 30g for an adult (Ref 14). It is used in

the manuf of cyanamide derivs (Dicyandiamides & Guanidines), and the impure material as a fertilizer & weed killer (Ref 6)

The storing of $CaCN_2$ contg small amts of moisture might result in an expln. The presence of H_2O in $CaCN_2$ is usually due to absorption from the atm during pulverization of the finished product. The presence of impurities, such as CaC_2 & CaO, increases the hygroscopicity of the product. Absorption of moisture by $CaCN_2$ may be partly avoided by using pure CaO as a primary material, by ventilating with dry air, and by coating the grains of $CaCN_2$ with oil (Ref 9)

Calcium cyanamide treated with a halogen acid, such as HCl or HBr, and then by HNO_3 or $HClO_4$ results in explosive compds, such as **Bromoformamidine Nitrate, Bromoformamidine Perchlorate, Chloroformamidine Nitrate,** and **Chloroformamidine Perchlorate** (Ref 23)

See also Refs 2, 3, 4, 5, 6, 8, 10, 12, 13, 15, 16, 17, 18, 19 & 20

Silver Cyanamide, Ag_2CN_2, is reported to decomp with expl violence in the temp range 330-50°. The proposed reaction mechanism is a 2-step, process forming AgCN, Ag & N in the first step, followed by decompn of AgCN to Ag & $(CN)_2$ in the second step. The addn of metallic Ag, NaCl or CaF_2 moderated the reaction to measurable velocities of varying degrees (Ref 21). The prepn & props of *Silver Nitrocyanamide,* $NCN.NO_2Ag$, are given by Okazaki et al (Ref 22). The ignition temps & induction periods (160°, 16.3 sec; 180°, 6.0 sec & 210°, 1.2 sec) are comparable to those of MF; activation energy 22.1 kcal/mol; and Q_{comb} 752 cal/g. The impact & friction sensitivity props are similar to those of LSt & DADNPh. When used in caps, Ag Nitrocyanamide initiates Tetryl & TNT but not as satisfactorily as MF or LA (Ref 22)

Kurzer (Ref 7) has described the syntheses & props of substituted arylsufonylcyanamides.

Refs: 1)Beil **3**, 78, (38), [66] & {153} 2)Marshall **1**,(1917), 114 3)S.Nagai & G.Yamaguchi, JSocChem Ind, Japan **43**, Suppl Binding 219, 534-38; Suppl Binding 361-62, 799-802 (in English) (1940); & **44**, Suppl Binding 286-87, 660-62 (in Englsih) (1940); CA **35**, 1584, 3395 (1941) & **44**, 8067 (1950) (Synthesis of $CaCN_2$ from Ca carbonate & ammonia) 4)Davis (1943), 376 5)K.G.Herring et al, IEC**38**, 1317 (1946) 6)Kirk & Othmer **4** (1949), 666 7)F. Kurzer, JChemSoc **1949**, 1034-38; Chem & Ind **1949**, 522-23 & CA **44**, 572-73 (1950); JChemSoc **1949**, 3029-33 & CA **44**, 3933-34 (1950); JChemSoc **1950**, 3269-76 & CA **45**, 6166-67 (1951); and JChemSoc **1951**, 1258-62 & CA **46**, 2004 (1952) 8)G.Bourjol & Mme Teindas, MP **31**, 52-3 (1949) 9)M.Pieri, Chimica (Milano) **7**, 158-60 (1952) & CA **46**, 8335-36 (1952) 10)G.Desseigne & A.Audiffren, MP **35**, 15-38 (1953) & CA **50**, 4507 (1956) (Prepn of Guanidine Nitrate from Ca Cyanamide) 11)M.Sakai & A. Aihara, JapP 8020 (1954) & CA **50**, 13990 (1956) 12)M.Nord, ChemEngrg **61**, 238-40 (Dec 1954) (Continuous process for production of $CaCN_2$) [J.Daniels, USP 2687945 (1954)] 13)J.P.Picard & M.Blais, "A New Approach to the Synthesis of Calcium Cyanamide without Using Electrical Power", PATR **2261** (1955) (Conf) 14)Sax (1957), 426 15)J.P. Picard & V.I.Siele, "Mechanism of Formation of White Calcium Cyanamide by the Picatinny Process", PATR **2405** (1957) (Conf) 16)J.P. Picard et al, "Laboratory Pilot Plant Investigation of Picatinny Process for Producing White Calcium Cyanamide", PATR **2452** (1957) (Conf) 17)V.I.Siele et al, "Suitability of White Calcium Cyanamide for the Preparation of Guanidine Nitrate", PATR **2455** (1957) (Conf) 18)M.Blais & J.P.Picard, "Effect of Various Physical Properties of Lime on the Purity of White Calcium Cyanmide Made by the Picatinny Process", PATR **2457** (1857) (Conf) 19)S. Chard et al, "The Manufacture of Calcium Cyanamide Via Carbon Monoxide, Ammonia and Reactive Lime: Part I. Laboratory Work" ERDE Rept **2/R/57** (1957) (Conf), and "Part II. An Assessment of the Possible Procedure Involved", ERDE Rept **4/R/57** (1957) (Conf) 20)S.Chard et al, "Review of the Calcium Cyanamide (Via Co and NH_3) Project with an Outline Design for a 35 Ton/Week Unit", ERDE TechMemo **3/M/57** (1957) (Conf) 21)V. A.Shushunov & B.G.Zateev, UchZapGor'kovskii-GosUniv, Ser Khim **1958**, No 32, 3-7 & CA **54**, 6380 (1960) 22)K.Okazaki et al, Kogyo-KayakuKyokaishi **21**, 12-15 (1960); CA **55**, 11848 (1961) & Explosivst **1964**, 103 23)W. Sauermilch, Explosivst **9**, 71-74, 256-57 (1961); CA **55**, 21589 (1961) & **57**, 8434 (1962) 25)V. 8.Siele & F.C.Tompkins, PATR **3201** (Jan

1965) (Isothermal decompn of thallous cyanamide)
Note: None of the classified reports listed above was used as a source of info

Cyanamide Complexes as Combustion Catalysts from Composite Propellants. See under Combustion Rate (or Burning Rate) Catalysts

Cyanates are salts of Cyanic Acid contg the monovalent radical -CNO. See under Cyanic Acid

Cyanazide. See under Cyanocompounds

CYANIC ACID AND ITS SALTS (CYANATES)

Cyanic Acid (called Cyansäure, Isocyansäure or Carbonimid in Ger), HO.C:N or O:C:NH; mw 43.03, N 32.56%; gaseous, condensed to col vol liq, fr p -80 to -86°, bp 23.5° at 760 mm, d 1.140 at 0°; rapidly polymerizes, even below 0°, to a wh amor polymer, cyamelide, while at higher temps cyanuric acid is also formed; in aq soln Cyanic Acid is very rapidly hydrolyzed to NH_3 & CO_2; is formed in the oxidn of pure C by aq $KMnO_4$ in the presence of NH_3 (Refs 1, 2, 3 & 4). See also Ref 5

Free Cyanic Acid is highly toxic; it is also a severe expln hazard as it emits flammable vapors when heated (Ref 6)
Refs: 1)Beil **3**, 31, (15), [27] & {65} 2)Franklin (1935), 108-11 3)Sidgwick, OrgChem of N (1937), 322-26 4)Davis (1843), 370, 373-74 & 387 5)J.Vaganay & S.Wevert, Fr P 1071628 & 1071629 (1954); MP **38**, 409, 415 (1956) & CA **53**, 6155-56 (1959) (Prepn of Isocyanates) 6)Sax (1957), 521

Cyanates

Ammonium Cyanate, NH_4OCN, col ndls, mp 88° (dec), d 1.342 at 20°; can be prepd by adding NH_3 to Cyanic Acid in ether soln at -8°, in addn to some urea; and by other methods (Ref 1 & Ref 2, p 277)
Cobalt (II) Cyanate, $CO(OCN)_2$, pale lavender crysts, mp 80° (dec), d 2.432 at 25° (Ref 1, p {70})
Copper (II) Cyanate, $Cu(OCN)_2$, dk-grn crysts, mp 80° (dec), d 2.418 at 25° (Ref 1, p {69})
Lead (II) Cyanate, $Pb(OCN)_2$, col ndls, mp dec, d 4.938 at 20° (Ref 1 & Ref 2, p 819)
Nickel Cyanate, $Ni(OCN)_2$, pale grn crysts, mp 80° (dec) d 2.302 at 25° (Ref 1, p {70})
Potassium Cyanate, KOCN, col crysts, mp 700-900° (dec), d 2.056 at 20°, highly toxic (Ref 1 & Ref 2, p 1040)
Silver Cyanate, AgOCN, col crysts, mp dec, d 4.15 at 20°; explodes on heating with $AgIO_3$ at 200°; highly toxic (Ref 1 & Ref 2, p 1104)
Sodium Cyanate, NaOCN, col ndls, mp (?), d 1.937 at 20°, highly toxic (Ref 1 & Ref 2, p 1116)
See also Ref 3
Refs: 1)Beil **3**, 34, (16), [297] & {69} 2)Sax [29] (1957) 3)E.Söderbäch, ActaChemScand **11** 1622-34 (1957) (in Ger) & CA **52**, 18047-48 (1958) (Prepn of metal cyanates)

Cyanides

Cyanides. These are compds contg the monovalent radical, -CN, derived from hydrocyanic acid (HCN). See **AC** in Vol 2 of Encycl, p C 167-L. Some of the more important salts are the following:
Ammonium Cyanide, NH_4CN, wh crysts, mp 36°, bp sublimes, evolving a flammable gas (Ref 3, p 278)
Barium Cyanide, $Ba(CN)_2$, wh crysts, highly toxic (Ref 3, p 332)
Cadmium Cyanide, $Cd(CN)_2$, crysts, mp >200° (dec) (Ref 3, p 419)
Calcium Cyanide, $Ca(CN)_2$, rhmb crysts, mp >350° (dec) (Ref 3, p 426)
Copper Cyanide, $Cu(CN)_2$, yel-grn crysts, mp dec, produces toxic & flammable vapors (Ref 3, p 504)
Lead Cyanide, $Pb(CN)_2$, wh powd (Ref 3, p 819)
Magnesium Cyanide, $Mg(CN)_2$, solid, highly toxic (Ref 3, p 842)
Mercuric Cyanide, $Hg(CN)_2$, col prisms, darkened by light, mp dec, d 3.996, highly toxic (Ref 3, p 858)
Potassium Cyanide, KCN, wh deliq crysts, having a faint ordor of bitter almonds, mp 635°, d 1.52 at 16°(Ref 3, p 1040)
Silver Cyanide, AgCN, wh odorless & tasteless powd, mp 320° (dec), d 3.95, highly toxic (Ref 3, p 1104)
Sodium Cyanide, wh deliq powd, mp 564°, bp 1496°, vap press 1mm at 817°, highly toxic (Ref 3, p 1116)
Other props & methods of prepn are given in the Refs. See also Cyanocompounds
Refs: 1)Thorpe **3** (1939), 463-512 2)Kirk & Othmer **4** (1949), 677-80 3)Sax (1957)

Cyanides, Diazo. These compds are described in the following Refs:
Refs: 1)R.J.W.Le Fèvre & H.Vine, Chem & Ind **56**, 688 (1937) CA **31**, 7857 (1937); JChem Soc **1938**, 431-38 & CA **32**, 5386 (1938) (Configurations of isomeric diazocyanides) 2)O.Stephenson & W.A.Waters, JChem Soc **1939**, 1796-1804 & CA **34**, 999 (1940) (Decompn reactions of aromatic diazocyanides) 3)M.A.Sabor, Indian JPhys **17**, 223-28 (1943) & CA **38**, 4498-99 (1944) (Transformation of diazocyanides) 4)H.H.Hodgson & E.Marsden, JChem Soc **1944**, 395-98 & CA **39**, 64 (1945) (Structures of Hantzsch's aryl syn- & anti-diazocyanides) 5)D.Anderson et al, JChemSoc **1947**, 445-53 & CA **41**, 5471 (1947) (Structure of Hantzsch's isomeric diazocyanides); **1947**, 457-60 & CA **41**,5471-72 (1947) (Magnetic props of isomeric aromatic diazocyanides) 6)N. Sheppard & G.B.B.M. Sutherland, JChemSoc **1947**, 453-56 & CA **41**, 5472 (1947) (IR spectra & molecular structure of isomeric diazocyanides) 7)H.H.Hodgson, JChemSoc **1948**, 1097-1104 & CA **43**, 595 (1949) (Reaffirmation of the structure of Hantzsch's isomeric diazocyanides)

Cyanide Safety Explosives. Safety expls consisting of mixts of metal cyanides & salts of HNO_2 were patented by Eiter et al (Ref). Representative compns are as follows: a)$NaNO_2$ 53 & $K_4[Fe(CN)_6]$ 47% b)$NaNO_2$ 27 & $Hg(CN)_2$ 73% c)$Ba(NO_2)_2$ 72 & NaCN 28%. The ingredients are intimately mixed in powd form and the compn detonated either by heating or priming
Ref: K.Eiter, O.Vogl & H.Michl, Austrian P 176784 (1953) & CA **48**, 1004 (1954)

Cyanidine. Same as 1,3,5 (or sym)-Triazine

Cyano. The monovalent radical -CN, sometimes abbreviated Cy. It acts like a halogen, forming Cyanides (qv), and like ammonia forms many complex salts. Cyano or cyanogen compds may be considered as derived from Cyanogen (qv) or its derivs

CYANOCOMPOUNDS

Cyanocompounds contain the monovalent radical -CN and do not include the Cyanides (qv). Some of the org derivs of interest in the expl industry include the following:

Cyanoacetamide, Malonamide Nitrile or Propionamide Nitrile (called Cyanacetamid, Cyanessigsäure-amid or Malonsäure-amidnitril in Ger), $NC.CH_2CONH_2$; mw 84.08, N 33.32%; wh crysts, mp 120°, bp dec; sol in w & alc; prepd by ammonolysis of cyanoacetic ester or by dehydration of ammonium cyanoacetate. The compd is highly toxic. It is used in plastics and as an intermediate in general & in org pharmaceutical synthesis
Refs: 1)Beil **2**, 589, (256) [534] & {1632} 2)Kay-Fries Chemicals, Inc (NY) Tech Data Bull (preliminary) "Cyanoacetamide" (Feb 1951) 3)Sax (1957), 522 4)Cond Chem Dict (1961), 322

Azidocyanoacetamide, $NC.CH(N_3).CONH_2$. Its *Copper salt*, $Cu(C_3H_2N_5O)_2$, defgr on heating even a small quantity; *Silver salt*, $AgC_3H_2N_5O$, explodes on heating; *Mercurous & Lead* salts also prepd (Refs 1 & 2)
Refs: 1)Beil **2**, {1642} 2)E.Ott & H.Weissenburger, Ber **70B**, 1834 (1937)

Nitrocyanoacetamide, $NC.CH(NO_2)CONH_2$; mw 129.08, N 32.56%; prisms (from alc), mp 145° (dec); sol in w & alc; sl sol in eth; insol in benz, chlf & petr eth. Its *Potassium salt*, $KC_3H_2N_3O_3$, monoclinic prisms, dec & defgr on heating above 225°; *Copper salt*, $Cu(C_3H_2N_3O_3)_2$, dk-blue prisms, dec & defgr on heating above 150°
Refs: 1)Beil **2**, 598 & (258)

Cyanoacetic Acid or Malonic Nitrile (called Cyanessigsäure or Malonsäure-mononitril in Ger), $NC.CH_2.COOH$; mw 85.06, N 16.47; wh hygr crysts, mp 69-71°, bp 160° (dec); sol in w, alc & eth; prepd by interaction of Na chloroacetate & K cyanide soln. This compd forms numerous salts, some of which are unstable
Refs: 1)Beil **2**, 583, (253), [530] & {1626} 2)Sax (1957), 522 3)Cond Chem Dict (1961), 322

Cyanoacetyl Azide (called Cyanessigsäureazide in Ger), $NC.CH_2CON_3$; mw 126.08, N 44.44%; yel expl oil, having a pungent odor; can be prepd by treating cyanoacetyl hydrazide with $NaNO_2$ & HCl
Refs: 1)Beil **2**, (256), & {1637} 2)A.Darapsky & D.Hillers, JPraktChem **92**, 313 (1915) & CA **10**, 883 (1916) 3)A.Weissberger & H.D. Porter, JACS **65**, 53 (1943) & CA **37**, 1427 (1943); JACS **66**, 1851-55 (1944) & CA **39**, 934 (1943)

Nitrocyanoacetic Acid, $NC.CH(NO_2).COOH$; mw 130.05, N 21.54%; prepd by saponification of the ethyl ester of nitrocyanoacetic acid with $Ba(OH)_2$. The Barium salt, $BaC_3N_2O_4 + H_2O$, lt-yel cryets, insol in w & org solvs, on treatment with dil HCl loses CO_2 and forms nitrocyanomethane, $NC.CH_2.NO_2$
Refs: 1)Beil **2**, (258) 2)C.Ulpiani, Gazz **42**, 225 (1912) & CA **6**, 2235 (1912)

Nitrocyanoacetyl Azide, $NC.CH(NO_2).CON_3 + H_2O$; mw 155.08, N 45.16 (anhyd salt); dk-yel hygr crysts, mp defgr on heating; decompd violently by boiling in w, giving N, Co_2, HCN & CO; was prepd from nitrocyanoacetylhydrazide by treating with HNO_2. Its *Sodium salt,* yel ndls, explodes on heating
Refs: 1)Beil **2**, (259) 2)A.Darapsky & D. Hillers, JPraktChem **92**, 336 (1915) & CA **10**, 885 (1916)

Nitrocyanoacetic Acid Methyl Ester, $NC.CH(NO_2).CO_2CH_3$; mw 144.09, N 19.44%; lustrous ndls + H_2O, mp 76°; readily sol in w & MeOH; diffc sol in eth; was obtd by oxidn of isonitrosocyanoacetic acid methyl ester with $KMnO_4$ soln. Its *Hydrazine salt,* $C_4H_4N_2O_4 + H_2N.NH_2$, crysts (from MeOH), melts at 168°; and its *Potassium salt,* $NC.C(:NO_2K).CO_2.CH_3$, lusturous lfts, decomp at 264-66°
Refs: 1)Beil **2**, (258) 2)A.Darapsky & D. Hillers, JPraktChem **92**, 324 (1915)& CA **10**, 884 (1916)

Nitrocyanoacetic Acid Ethyl Ester, $NC.CH(NO_2).CO_2.C_2H_5$; mw 158.11, N 17.72%; col prisms (from w); sol in w & alc; insol in eth, chlf & benz; was pred by boiling diethyl furoxanedicarboxylate with w for 4-5 days until completely in soln. It forms numerous salts. The *Silver salt,* $AgC_5H_5N_2O_4$, becomes gray on the surface when exposed to light; explodes on heating
Refs: 1)Beil **2**, 598 & (258) 2)C.Ulpiani, Gazz **42**, 218-21 (1912) & CA **6**, 2235 (1912)

Isonitrosocyanoacetic Acid (called Isonitrosocyanessigsäure; Oximinocyanessigsäure; Oximinomalonsäure-mononitril; Isonitrosomalonsäure -mononitril; or Propanoximnitrilsäure in Ger), $NC.C(:N.OH).COOH$; mw 114.06, N 24.56%; water-clear crysts + H_2O (from eth + benz), mp - dec 120°; v sol in w, alc & eth; mod sol in chlf; insol benz & petr eth; its methods of prepn & other props are given in Beil
Ref: Beil **3**, 774, (269) & [476]

Isonitrosocyanoacetyl Azide, $NC.C(:N.OH).CO.N_3$; mw 139.08, N 50.36%; yel crysts, mp - explodes on heating; sol in eth, alc & acet; was prepd from the hydrazine salt of isonitrosoacetyl hydrazide in w by reaction with aq $NaNO_2$
Refs: 1)Beil **3**, (272) 2)A.Darapsky & D. Hillers, JPraktChem **92**, 321 (1915) & CA **10**, 884 (1916)

Isonitrosocyanoacetic Acid Methyl Ester, $NC.C(:N.OH).CO_2.CH_3$; mw 128.09, N 21.87%; pltlts + H_2O (from w), mp ; begins to melt at 60-65°, over H_2SO_4 it loses w of crystn & melts at 121°; ver sol in hot w; was prepd by reaction of nitrocyanoacetic acid methyl ester with $NaNO_2$ & dil H_2SO_4, and by other methods. It forms numerous salts. Its *Hydrazine salt,* $NC.C(:NON_2H_5).CO_2CH_3$; yel crysts; readily sol in w, diffc sol in cold abs alc, and insol in eth; deflgr at 78° and decomp at 100°
Refs: 1)Beil **3**, 774, (269) & {1358} 2)A. Darapsky & D.Hillers, JPraktChem **92**, 316, 323 (1915) & CA **10**, 884 (1916) 3)L.Cambi, Gazz **61**, 9 (1931)

Cyanoacetyl Hydrazide (called Cyanessigsäure -hydrazid or Malonsäure-nitril-hydrazid in Ger), $NC.CH_2.CO.NH.NH_2$; mw 99.09, N 42.41%; col prisms (from alc), mp 110-15°; sol in alc & w; almost insol in eth; was prepd by treating methyl cyanoacetate with hydrazine hydrate in absolute alc. On treating with $NaNO_2$ & HCl, it forms *Cyanoacetyl Azide*
Refs: 1)Beil **2**, 591, (256) & {1636} 2)A. Darapsky & D.Hillers, JPrakt Chem **92**, 313 (1915) & CA **10**, 883 (1916)

Nitrocyanoacetyl Hydrazide, $NC.CH(NO_2).CO.NH.NH_2$; mw 144.09 N 38.89%; col ndls (+ H_2O) or yel anhyd prisms, mp >285°; was obtd by reacting hydrazine hydrate with either the hydrazine salt or K salt of nitrocyanoacetic acid methyl ester. It forms several salts. The *Hydrazine salt,* $C_3H_4N_3O_3 + H_2N.NH_2$, occurs as colorless or orn-red crysts, the orn-red salt sinsters at 185-95° & decomp at 200°; the *Potassium salt,* $KC_3H_3N_4O_3$, col prisms, explodes on heating
Refs: 1)Beil **2**, (258) 2)A.Darapsky & D. Hillers, JPraktChem **92**, 326, 328, 332, 334 (1915) & CA **10**, 884 (1916)

Isonitrosocyanoacetyl Hydrazide (called Isonitrosocyanessigsäurehydrazide or Oximino-

malonsäure-nitril-hydrazid in Ger), NC.C(:NOH)-CO.NH.NH_2; mw 128.09, N 43.74%; brown, lustrous ndls (from alc), mp 166° (dec); was obtd by treating the methyl ester of isonitrosocyanoacetic acid with hydrazine hydrate in abs alc. Its *Hydrochloride salt*, $C_3H_4N_4O_2$ + HCl, lt yel crysts, becomes red on heating, and suddenly decomp at 204°; its *Hydrazine salt*, NC.C(:NON_2H_5)-CONH.NH_2, yel ndls (from dil alc), decomp at 135°; and *Silver & Lead* salts which are brn ppts

Refs: 1)Beil **3**, (272) 2)A.Darapsky & D. Hillers, JPraktChem **92**, 317 (1915) & CA **10**, 884 (1916)

Cyanoacetylene, Acetylene Cyanide or Acetylene Nitrile (called Cyanacetylen or Propiolsäure Nitril in Ger), NC.C:CH; mw 51.05, N 27.44%; col liq, fr p 5°, bp 42.5°, d 0.8159 at 17°, n_D 1.38699 at 17°; was obtd by distilling Propiolamide & P_2O_5 in an atm of CO_2 at reduced pressure. The liq is flammable, and becomes brn on standing, even in the absence of light & air. The vapor is intensely irritating. Cyanoacetylene forms with $AgNO_3$ a wh expl substance; and with ammoniacal Cu_2Cl_2, a grn compd is formed which defgr on heating

Refs: 1)Beil **2**, (208) 2)C.Moureu & J.C. Bongrand, CR **151**, 946 (1910) & JCS **100** I, 22 (1911)

Cyanazide, Cyanoazide or Cyanogen Azide (called Cyanazid or Kohlenstoffpernitrid in Ger), $NC.N_3$; mw 68.04, N 17.65%; col oil which detonates violently by thermal or mechanical shock; can be handled relatively safely in solvs; half-life of a 27% soln in acetonitrile is 15 days at RT, but this soln can be stored indefinitely without change at 0° to -20°. Synthesis of Cyanogen Azide is carried out by suspending NaN_3 in dry acetonitrile, and cyanogen chloride is distilled into the mixt below -12°. The soln is warmed to RT and filtered to remove NaCl. The use of dry solvs is important to avoid formation of expl, solid by-products, and care must be taken to avoid separating $NC.N_3$ from the soln (Ref 5)

This azide is reported to have a versatility & scope of chem reactivity that is very broad & useful (Ref 5)

The early literature (See Refs 1, 2, 3 & 4) describing this compd is reported by Marsh & Hermes (Ref 5) to be incorrect

Refs: 1)Beil **3**, (60) & [102] 2)G.Darzens, CR **154**, 1232 (1912) & CA **6**, 2075 (1912) 3)C. V.Hart, JACS **50**, 1922-30 (1928) & CA **22**, 3138 (1928) 4)P.Walden & L.F.Audrieth, Chem-Revs **5**, 355 (1928) & CA **22**, 4396 (1928) 5)F. D.Marsh & M.E. Hermes, JACS **86**, 4506-07 (1964) and C & EN **42**, 51 (Oct 26, 1964)

Cyanoazidodithiocarbonate. See Cyanogen Azidodithiocarbonate, Vol 1, p A635-R

6-Cyanobenzazimidole [called 6-Cyan-Benzazimidol; Benztriazol-carbonsäure-(6)-nitril -1-oxyd; 6-Cyan-Benztriazol-1-oxyd; or 1-Oxy -benztriazol-carbonsäure-(6)-nitril in Ger], NC.C_6H_3:N.NH (N(:O) ring) or NC.C_6H_3.N:N.NOH (ring); mw 160.13, N 34.99%; col ndls (from alc), mp dec & deflgr at 218-19°; was prepd by gently warming 3-nitro-4-hydrazinobenzonitrile with NaOH and adding dil HCl

Refs: 1)Beil **26**, [157] 2)W.Borsche, Ber **54**, 661 (1921) & CA **15**, 2842 (1921) 3)T.J.F. Mattaar, Rec **41**, 35 (1922) & CA **16**, 1225 (1922)

N-(2-Cyano-4,6-dinitrophenyl)-N-nitroaminoethyl Nitrate or N-2-Hydroxyethyl-N,3,5-trinitro-anthranilonitrile Nitrate (latter name given in CA 5th Decennial Formula Index, p 409F), NC.$C_6H_2(NO_2)_2$.$N(NO_2)$.CH_2.CH_2ONO_2; mw 342.19, N 24.56%; pale yel crysts (from chlf & alc); mp 110°, explodes when heated over a flame; was prepd by reaction of β-ethanolamine with 1-chloro-2-cyano-4-nitrobenzene to give 2-cyano-4-nitro-1-hydroxyethylaminobenzene and nitration with abs HNO_3, or by reaction of ethanolamine with 1-methoxy-2-cyano-4,6-dinitrobenzene in alc to give 2-cyano-4,6-dinitro-1 -(β-hydroxyethylamino)-benzene and nitration with abs HNO_3 (Ref 2)

Refs: 1)Beil - not found 2(H.M.A.Hartmans, Rec **65**, 468-70 (1946) & CA **41**, 734 (1947)

Cyanodiphenylamine. See Anilinobenzonitrile, Vol 1, p A422-L

Cyanoethyl Nitrate, $NC.CH_2.CH_2ONO_2$; mw 116.08, N 24.14%; liq, fr p -27°, d 1.28 at 30°; was prepd by reaction of ethylene chlorohydrin & NaCN, followed by nitration. This compd

absorbed on filter paper is not detonated by impact; it is less sensitive & less powerful than ethylene glycol dinitrate and of adequate stability (Refs 2 & 3)

Bergeim (Ref 2) proposed the use of this compd as an ingredient of dynamite: cyanoethyl nitrate 15, NG 15, $NaNO_3$ 58, woodmeal 11 & chalk 1%

Refs: 1)Beil - not found, but the parent compd, 3-hydroxypropane nitril or hydracrylo nitrile ($NC.CH_2.CH_2OH$) is described in Beil **3**, 298, (113), [213] & {537} 2)F.H.Bergeim, USP 1685771 (1928) & CA **22**, 4821 (1928) 3)Blatt, OSRD **2014** (1944) 4)J.Boileau et al, Bull Fr **1957**, 338-41 & CA **51**, 13740-41 (1957) (Prepn, props & IR spectra of alphatic nitrate-nitrils)

Cyanoformamidine Azide (called Azido-cyanimino -amino-methan; C-Azido-N;cyan-formamidin or "Dycyanamidazid" in Ger), $NC.N:C.(NH_2).N_3$; mw 110.08, N 76.35%; col pltlts (from eth), mp 151-52° (dec); v sol in alc & acet; sl sol in chlf & hot benz; v sl sol in w & eth; and insol in petr eth; can be prepd by passing NH_3 into a cold ethereal soln of dicyandiazide or by adding cyanogen bromide in acet to 5-aminotetrazole nitrate in NaOH at 0°. When heated in concd NaOH, NaN_3, Na_2CO_3, cyanamide & NH_3 are evolved. When heated in a tube, it deflgr leaving a brn residue

Refs: 1)Beil **3**, [102] 2)C.V.Hart, JACS **50**, 1927 (1928) & CA **22**, 3138 (1928)

Cyanogen, Cyan Cyanide, Dicyanogen, Ethane Dinitrile, Oxalonitrile or Prussite (called Cyan, Dicyan, Oxalsäure-dinitril, or Äthan-dinitril in Ger), NC.CN; mw 52.04, N 53.84%; col gas, having a pungent penetrating odor, fr p -34°, liq p -21°, d 0.866 at 17°; sol in w, alc & eth; can be prepd by mixing solns of KCN & $CuSO_4$ or by heating $Hg(CN)_2$ and by other methods (Ref 1). Cyanogen is extremely poisonous and its effect on mammals is identical with that of prussic acid. Cyanogen burns with a violet -colored flame, forming CO_2 & N_2. James & Laffitte (Refs 7 & 8) have reported the expln limits for C_2N_2-air mixts at pressures of 100-760 mm Hg, abs temps of 700-950°, and compns from 7-73% C_2N_2

Cyanogen is used in org synthesis and as a poison gas in warfare

Refs: 1)Beil **2**, 549, (238), [511] & {1587} 2)Sidgwick, OrgChem of N (1937), 299-302 3)Davis (1943), 387 4)Mellor (1946), 369-70 5)H.E.Williams, "Cyanogen Compounds: Their Chemistry, Detection and Estimation", Arnold & Co, London (1947), 417pp 6)V.Migrdichian, "The Chemistry of Organic Cyanogen Compounds" Reinhold, NY (1947), 460 pp (ACS Monograph 105) 7)H.James & P.Laffitte, CR **236**, 811-13 (1953) & CA **47**, 6141 (1953) 8)H.James & P. Lafitte, 5th Symposium on Combustion, Pittsburgh **1954**, 616-19 & CA **49**, 16386-87 (1955) 9)Sax (1957), 522 10)Cond Chem Dict (1961), 323

Cyanogen Bromide, NC.Br, mw 105.93, prisms or acicular transparent crysts, having a penetrating odor, mp 52°, bp 61.6°, d 2.015 at 20°; slowly decompd by cold w; sol in alc, benz & eth; prepd by action of bromine on KCN or by interaction of NaBr, NaCN, $NaClO_3$ & H_2SO_4

Cyanogen bromide reacted with $KC(NO_2)_3$ in dry acetone to give a mixt of solid & liq materials. The solid material (N 25.15-25.35%) could be detonated by impact, and burned rapidly. The liq was a powerful lachrymator (Ref 2). The subj compd is used in org synthesis, as a parasiticide, as an agent for treating cellulose products and as a military poison gas (Ref 5)

Refs: 1)Davis (1943), 376 2)US Rubber Co Progress Rpt **5** (1948-49), pp 39-40 3)Sax (1957), 523 4) Cond Chem Dict (1961), 323 5)P ATR **2700**, Vol 2 (1962), p C 169-L

Cyanogen Chloride. See CK under Chemical Agents or Chemical Warfare Agents, Vol 2, p C167-L

Cyanogen Fluoride, NC.F, mw 45.02; col gas, fr p -72° forming a white mass; insol in w; can be prepd by interaction of Ag fluoride & cyanogen iodide. It is used in org synthesis & as a military lachrymator

Refs: 1)Sax (1957), 523 2)Cond Chem Dict (1961), 323

Cyanogen Iodide, NC.I, mw 152.94; col ndls, having a pungent odor & acrid taste, mp 146.5°, d 2.84; sol in w, alc & eth; prepd by heating a metal cyanide with iodine. It is a very violent poison

Refs: 1)Sax (1957), 523 2)Cond Chem Dict (1961), 323

Cyanoguanidine or Dicyandiamide (called Cyanguanidin, Dicyandiamid or Guanidincarbonsäurenitril in Ger),

$NC.NH.C(NH_2):NH$ or
$NC.NH.C(:NH).NH_2$;

mw 84.08, N 66.64%; wh crysts (from eth + acet), mp 208°, d 1.400 at 25°, Q_{form} -6.9kcal/mol at const vol; Q_{comb} 331kcal/mol at const vol; sol in w & alc; sl sol in eth; prepd commercially by the dimerization of cynamide in the presence of bases. Cyanoguanidine reacts with many reagents to form a number of heterocyclic compds. Such reactions offer a method of prepg 1,3,5-triazines, pyrimidines, triazoles & tetrazoles. It also forms numerous salts

Cyanoguanidine is used in fertilizers; as a stabilizer of NC; in org synthesis of melamine, barbituric acid & guanidine salts; in pharmaceutical products; dyestuffs; and in expls. Some examples of expls contg cyanoguanidine are as follows:

a)Mixts of $NaNO_3$ & cyanoguanidine added to mixts of AN & Amm perchlorate to form an expl molten below 100° (Ref 3)

b) A mixt of cyanoguanidine, dicyandiamidine, NGu & a nitrate, chlorate or perchlorate, which has high expl props (Ref 4)

c) Cyanoguanidine 10, AN 69, Amm perchlorate 17 & paraffin 4% (Ref 2)

d) Cyanoguanidine 10, AN 70 & PETN 20% (Ref 7a). This expl is Designated as **ASN** and is described in Vol 1, p A496-L

Methods of Analysis of Cyanoguanidine. No method has been developed which can be applied to all kinds of mixts contg cyanoguanidine. Methods of analysis have been developed for detg small amts of cyanoguanidine in some mixts. No method is reported to give an accuracy better than ±1% in detg the purity of fairly pure (>95%) cyanoguanidine. The best way to analyze "pure" cyanoguanidine is to determine the amt of probable impurities (moisture, ash, thiourea, iron, insol matter, and compd pptd by PA) and subtract the sum from 100

Garby (Ref 7) detd cyanoguanidine content by hydrating it to guanylurea, then treating with a soln of an ammoniacal Ni salt to ppt Ni guanylurea. Harger (Ref 5) pptd cyanoguanidine as Ag cyanoguanidine picrate. Johnson (Ref 6) modified Harger's method by titrating the excess Ag and calcg the amt of dry cyanoguanidine originally present. Berlin & Zinov'eva (Ref 11) hydrated cyanoguanidine to guanylurea using an excess of std base to calc the amt of cyanoguanide originally present. See also Bourjol & Teindas (Ref 15)

Refs: 1)Beil **3**, 91, (42), [75] & {167} 2)G. Gin, BritP 157046 (1916) & CA **15**, 1815 (1921) 3)Dynamit-AG, GerP 305567 (1917) & CA **14**, 2555 (1920) 4)C.Mannelli & B.Luigi, ItalP 49536 (1919) & CA **14**, 2555 (1920) 5)R.N. Harger, IEC **12**, 1107-11 (1920) 6)E.Johnson, IEC **13**, 533-35 (1921) 7)C.D.Garby, IEC **17**, 266-68 (1925) 7a)M.Tonegutti, FrP 833729 (1938) & CA **33**, 3590 (1939) 8)E.W.Hughes, JACS **62**, 1258-67 (1940) (Cryst structure of cyanoguanidine) 9)L.Galimberti, Boll Sci Fac-Chim Ind Bologna **1941**, 18-21 & CA **37**, 3271 (1943) (Prepn of cyanoguanidine from Ca cyanamide in 90% yeild) 10)J.L.Osborne, USP 2416542 (1947) & CA **41**, 3120 (1947) (Prepn of cyanoguanidine starting with AERO Cyanamide, which is the trade name for coml Ca cyanamide manufd by the American Cyanamide Co) 11)A. A.Berlin & Z.A.Zinov'eva, ZhObshchKhim **17**, 43-50 (1947) & CA **42**, 61-62 (1948) (Rapid detm of cyanoguanidine) 12)P.H.Sykes et al, BIOS Rept 1720 (1947) (PB No 91926) (Cyanoguanidine production in Germany) 13)G.Bourjol, MP **30**, 247-54 (1948) & CA **47**, 3243 (1953) (Prepn of cyanoguanidine by hydrolysis of tech grade Ca cyanamide) 14)D.J.Salley & M.Thomas, JACS**70**, 2650-53 (1948) (Heat of combustion of cyanoguanidine & other compds) 15)G.Bourjol & Mme Teindas, MP **31**, 55-57 (1949) (Analytical methods applicable to manuf of cyanoguanidine & other guanidine derivs) 16)Kirk & Othmer **4** (1949), 673 17)Cyanamid's Nitrogen Chemical Digest, "The Chemistry of Dicyandiamide", American Cyanamid Co, NY, Vol **3** (1949) 18)L. Médard & M.Thomas, MP **34**, 425, 433 (1952) (Heats of combustion of cyanoguanidine and of 11 other org compds used in proplnts & expls) 19)Ullmann **5** (1954), 827 20)Sax (1957), 577 (Toxicity details are unknown) 21)Cond Chem-Dict (1961), 369 22)Conf "Propellant Manual", SPIA/M2 (1962), Unit Nos 623, 624 & 626 (Not used as a source of info)

Cyanoazoguanidine or Diazoguanidine Cyanide [called Diazoguanidincyanid; Triazendicarbonsäure-nitril-amidin; or Triazencarbonsäure-(1)-nitril-carbonsäure-(3)-amidin in Ger], $NC.N:N-NH.C(:NH).NH_2$; mw 112.10, N 74.98%; lt-yel

ndls (from boiling w), mp - becomes brown >200°, melts & decomp completely at higher temps; sol in alkalies & acids; almost insol in cold w; insol in alc & eth; was pptd on mixing aq solns of diazoguanidine nitrate & KCN. Its *Nitrate*, $C_2H_4N_6 + HNO_3$, col crysts, melts at 123° (Refs 1 & 2). Lieber et al (Ref 3) detd its IR absorption spectra

Refs: 1(Beil **3**, 128 2)J.Thiele & W.Osborne, Ann **305**, 69 (1899) & JCS **76I**, 412 (1899) 3)E. Lieber et al, Anal Chem **23**, 1594 (1951) & CA **46**, 3857 (1952)

Cyanoguanidine Nitrate or Dicyandiamide Nitrate, $H_2N.C(:NH).NH.CN.HNO_3$; mw 147.10, N 47.61%; col monoclinic crysts, mp - has no sharp mp or decompn point; was prepd by addg 67% HNO_3 to an aq suspension of cyanoguanidine at 25°. The mixt was cooled in an ice bath, the crysts separated, and washed with methyl ethyl ketone. The product is reported to be suitable for expls, insecticides, resins, chemotherapeutics and as chem intermediates

Refs: 1)Beil - not found 2)J.H.Paden & A.F. MacLean, USP 2537850 (1951) & CA **45**, 4263 (1951)

Nitrocyanoguanidine, $NC.NH.C(:NH).NH.NO_2$; mw 103.08, N 40.77%; reported to be one of several high-N compds suitable for use in mixts with 5-35% of a hypophosphite [NaH_2PO_2, $Ca(H_2PO_2)_2$ or $NH_4H_2PO_2$] to produce a flameless gas-producing charge. When ignited the mixt is converted into permanent gases without flame at a temp insufficient to ignite $CH_4 - O_2$ mixts. The prepn & props of nitrocyanoguanidine where not found in the literature

Refs: 1)Beil - not found 2)A.T.Tyre, USP 2470082 (1949) & CA **43**, 5190 (1949) (See name given in CA Index)

Cyanohydrazine or Carbazonitrile, $NC.NH.NH_2$; mw 57.04, N 73.65%; no props are reported in abstracts; was obtd on refluxing $NH_2NHCSNH_2$ with alkali (Ref 2), and by reaction of $AgNO_3$ with selinosemicarbazone ($H_2NCSeNH.NH_2$) (Ref 4). Its *Nitrate* added in small portions to concd H_2SO_4 gave a fine wh ppt which, after washing with 4 portions of w and air drying at RT, exploded violently. It was also exploded by friction & light impact. When heated, decompd with evolution of gas (Ref 3)

Refs: 1)Beil - not found 2)R.Sahasrabudhey & H.Krall, JIndian Chem Soc **18**, 225-28 (1941) & CA **36**, 752 (1942) 3)I.J.Schnaffer, US Rubber Co Quart Rept **3** (30 April - 31 July 1948) (Contract NOrd 10129) 4)R.Huls & M.Renson, Bull Belg **65**, 696-99 (1956) (in French) & CA **51**, 5727 (1957)

Cyanomethane. See Acetonitril and Derivatives, Vol 1, p A45ff

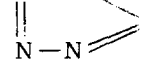
Cyanomethyl Nitrate (called Nitrat des Glykolsäurenitril in Ger), $NC.CH_2.ONO_2$; mw 102.05, N 27.45%; liq, bp 69-70° at 13mm with sl decomp; explodes on heating rapidly; mod sol in w; can be prepd by dry distillation of the addn product from iodoacetonitrile ($ICH_2.CN$) & silver nitrate ($AgNO_3$) or by reaction of formaldehyde & Na cyanide, followed by nitration. This expl is not detonated by impact. It is stable in dry air, but in the presence of moisture develops acidity. It also develops pressure & decomposes when stored in a closed vessel

Refs: 1)Beil **3**, 243 2)R.Scholl & W.Steinkopf, Ber **39**, 4396 (1906) 3)Blatt, OSRD **2014** (1944)

Cyanomethylnitrolic Acid (called Cyanmethylnitrolsäure or Nitrooximinoessigsäurenitril in Ger) $NC.C(:NOH).NO_2$; mw 115.05, N 36.53%; hygr crysts, decomp on standing; v sol in w, alc & eth; mod sol in benz; sl sol in chlf; insol in petr eth; was prepd from the Amm salt of nitroacetonitrile in w by treatment with $NaNO_2$ & H_2SO_4. Its *Silver salt*, carmine-red microsc crysts; explodes feebly when heated

Refs: 1)Beil **2**, 558 & (242) 2)W.Steinkopf, Ber **42**, 621 (1909) & CA **3**, 1155 (1909)

5-Cyano-(α-tetrazole) [called 5-Cyan-tetrazol or Tetrazol-carbonsäure-nitril (5) in Ger],



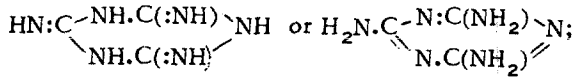
C.CN; mw 95.07, N 73.67%; col ndls (from alc), mp starts to decomp at 70° & melts at 90°; readily sol in w & in most org solvs; insol in CS_2; can be prepd by passing dicyan (NC·CN) into cooled 40% aq hydrazoic acid. Its *Silver salt*, AgC_2N_5, ppt, explodes on heating

Refs: 1)Beil **26**, (183) & [336] 2)E.Oliveri-Mandalà & T.Passalacqua, Gazz **41**, 431 (1911) & **43**, 465 (1913) 3)J.Lifschitz, Ber **48**, 415 (1915) 4)T.Curtius et al, Ber **48**, 1617, 1620, 1624 (1915) 5)E.Oliveri-Mandalà, Gazz **54**, 775 (1924) 6)F. R. Benson, Chem Revs **41**,

6 (1947) 7)W.Friederich, USP 2710297 (1955) & CA **50**, 5768 (Prepn of Cyanotetrazole, other tetrazole derivs & their salts) 8)M.M.Williams et al, JPhys Chem **61**, 264 (1957) (Heat of combn values detd for a number of tetrazoles & related high N compds)

Cyanocompounds (Poly). Syntheses & evaluation as high energy fuels for propellant systems is the subject of a Conf Standford Research Inst Final Report (31 Oct 1963 to 31 Dec 1964)

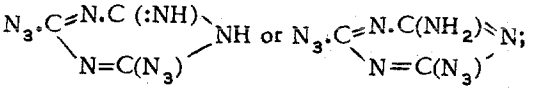
Cyanuramide, Melamine or 2,4,6-Triamine-sym-triazine (called Cyanuramid;Melamin;Isomelamin; 2,4,6 Triimino-- 1,3,5 - Triazin; or 2, 4, 6 - Triimino-hexa-hydro - 1, 3, 5 - Triazin in Ger),



mw 126.13, N66.64%; wh monoclinic crysts (from w), mp 250°, bp-sublimes, d 1.573 at 20°; sl sol in w, glycol, glycerol & pyridine; v sl sol in alc; insol in eth, benz & CCl_4; can be prepd by heating dicyandiamide with ammonia, by heating guanidine carbonate with 6N NH_3 to 160°, by reduction of cyanuric triazide, and by other methods (Ref 1). Cyanuramide may be considered a cyclic trimer of cyanamide (qv). It forms numerous salts, some of which are unstable & expl. Its *Picrate*, lt-yel ndls, dec when heated to 268° & chars at 300°

Refs: 1) Beil **26**, 245, (74) & [132] 2) Sax (1957), 854 3) CondChemDict (1961), 708

Cyanuramide Diazide (called 4,6-Diazido-2-imino -dihydro-1,3,5 - trizin or 4,6 - Diazido -2- amino -1,3,5-triazin in Ger),



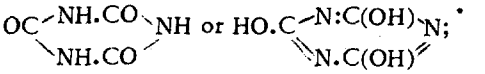
mw 178.13, N 78.64%; col pltlts (from alc), mp-dec on heating above 200°, explodes on heating rapidly to 210°; sol in aq NaOH & repptd when the soln is acidified; insol in most of the common solvs; was prepd by passing NH_3 into an ethereal soln of cyanuric triazide. When heated in a strong NaOH soln, cyanuramide diazide in decompd into NaN_3, cyanamide & CO_2

Refs: 1) Beil **26**, [83] 2) C.V. Hart, JACS **50**, 1929 (1928) & CA **22**, 3138 (1928)

Cyanurea (called Cyanharnstoff, Allophansäure-nitrile, Cyansäureureid, or Cyanaminoformamid in Ger), NC.NH.CO.NH_2; mw 85.07, N49.40%; ndls, mp-softens & dec at 100°; can be prepd by interaction of K cyanate & cyanamide in water soln, by action of Ba $(OH)_2$ on dicyandiamide in w soln, and by action of aq HCl on the Ag salt of dicyanimide (Ref 2). Cyanurea forms a large number of metallic salts (Ref 1)

Refs: 1) Beil **3**, 82 [68] {154} 2) Franklin (1935), 121-23

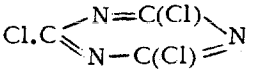
Cyanuric Acid; Tricarbimide; Tricyanic Acid; or sym-Triazine - 2,4,6-triol (called Cyanursäure; Isocyanursäure; 2.4.6 - Trioxo-hexahydro - 1.3.5-Triazin; or 2.4.6 - Trioxy - 1.3.5-Triazin in Ger),



mw 129.08, N32.56%; crysts + $2H_2O$ (from w), odorless but having a bitter taste; anhyd crusts (from concd HCl or concd H_2SO_4), mp - dec on heating; d (hydrate) 2.228 at 24°; is formed in spontaneous polymerization of cyanic acid & cyamelide from which it can be separated by extraction with hot w; is best prepd in a pure state by the action of w on cyanuric bromide. Cyanuric acid is hydrolyzed by hot mineral acids to CO_2 & NH_3, but is stable to caustic alkalies with which it forms numerous salts

Refs: 1) Beil **26**, 239 (73) & [131] 2)Sidgwick, OrgChem of N (1937), 343 3) Davis (1943), 374, 387 & 433 4) E.R. Atkinson, JACS **73**, 4443 (1951) (Prepn of Cyanuric Acid by hydrolysis of nitroammelide) 5) Sax (1957), 524 (Toxicity) 6) CondChemDict (1961), 324

Cyanuric Chloride or 2,4,6 - Trichloro - 1,3,5 - triazine (Called Cyanurchlorid or 2.4.6-Trichlor -1.3.5 - triazin in Ger),



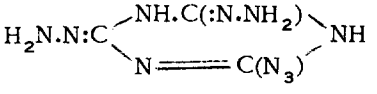
mw 184.44, N22.79%; monoclinic crysts (from eth), having a pungent odor, mp 146°, bp 194° at 764 mm of Hg, d 1.32 at 20°, Q_{comb} 294 k cal/mol at C_v, Q_{form} 107 KCal/mol at C_v, Q_{vapzn} 11.2 kcal/mol; sol in chlf, CCl_4, hot eth, dioxane & ketones; v sl sol in w in which it readily hydrolyzes. Cyanuric chloride is formed by polymerization of cyanogen chloride and also by action of PCl_5 on

cyanuric acid. This compd is irritating to the skin and may cause vesicles & ulceration. It is also a lachrymator & irritant to the nasal mucous membranes. After repeated exposures, allergic manifestations such as asthma & hives may occur

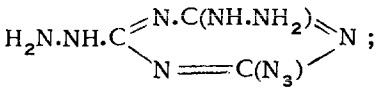
Cyanuric chloride is used in chem synthesis, dyestuffs, pharmaceuticals, surfactants & expls

Refs: 1) Beil **26,** 35 & [7] 2) Franklin (1935) 204 3) Davis (1943), 432-33 4) Cyanamid's Nitrogen Chemicals Digest, "The Chemistry of Cyanuric Chloride", American Cyanamid Co, NY (1951) 5) Sax (1957), 524 6) CondChemDict (1961), 423

Cyanuric Dihydrazide Azide (called Cyanurdihydrazidazid; 6-Azido - 2.4 - dihydrazono-tetrahydro - 1.3.5 - triazin; or 6 - Azido - 2.4 - dihydrazino - 1.3.5 - triazin in Ger),



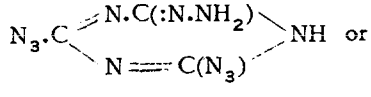
or



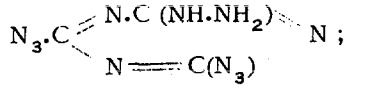
mw 182.16, N76.90%; crysts (from alc), mp 85-87°; can be prepd by reaction of cyanuric hydrazide with $NaNO_2$ in aq soln. This compd is exploded by impact

Refs: 1) Beil **26,** [120] 2) E. Ott, GerP 355926 (1922) & CA **17,** 1242 (1923)

Cyanuric Hydrazide Diazide (called Cyanurhydrazid-diazid; 4.6 - Diazido - 2 - hydrazono - dihydro - 1.3.5 - triazin; or 4.6 - Diazido - 2 - hydrazino - 1.3.5 - triazin in Ger),



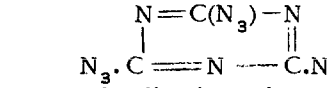
or



mw 193.13, N 79.78%: crysts (from alc), mp 87-88°; insol in w & dil inorg acids; may be prepd by treating either cyanuric trihydrazide or cyanuric dihydrazide monoazide with $NaNO_2$ in HCl soln. This compd explodes on rapid heating or on impact

Refs: 1) Beil **26,** [84] 2) E. Ott, GerP 355926 (1922) & CA **17,** 1242 (1923)

Cyanuric Triazide (called Cyanurtriazid or 2.4.6 -Triazido - 1.3.5 - triazin in Ger),



; mw 204.13, N 82.35%; wh ndls (from alc), mp 94°, bp-dec when heated above 100°, explodes on heating from 150-80° depending on the rate of heating; d 1.54 at 13° (cast) & 1.4 - 1.5 (pressed); readily sol in acet, hot alc, benz, chlf & eth; insol in w; can be prepd by interaction of cyanuric chloride & NaN_3 in aq soln (Refs 1 & 2). Taylor & Rinkenbach (Ref4) prepd the compd in a pure state and described its props

Cyanuric Triazide is exploded by impact & friction, and is more than twice as powerful as MF, but too sensitive to be handled in large quantities. The expl props & other related props of cyanuric triazide have been reported as follows:

Brisance by Sand Test, 32.2g crushed vs 12.2 for MF & 48 for TNT (Ref 25)

Explosion Temperature, 205° when heated at 20°/min and 170° on heating at 5°/min

Friction sensitivity, extremely sensitive (Ref 21)

Heat of formation, -222 kcal/mol at C_v (Ref 12)

Impact sensitivity, with 2 kg wt 0.9 cm and 500g wt 7 cm vs 8 for MF & 32 for LA; and with 1 kg wt 7 cm (all on BM app)

Initiating ability, min amts reqd for initiation of 0.4 g HE are: for TNT 0.10g, PA 0.05, Tetryl 0.04, Exp D 0.15, which values are smaller than corresponding valves for MF

Power by Trauzl test, 135% PA & 140% TNT

Sensitivity to heat, detonates when heated rapidly or when exposed to a flame

Stability, moderately volatile & hygroscopic

Toxicity, details are unknown (Ref 22)

Velocity of detonation, 5550-5600 m/sec for a chge 0.3" in diam pressed to d 1.15 g/cc (Ref 25)

Uses. Cyanuric Triazide has not been used commercially in military ammo, because of its extreme sensitivity to impact, diffc in controlling cryst size, and its volatility & hygroscopicity. Otherwise, it would be a better compd than MF for use in detonators (Refs 3 & 4)

Refs: *1) Beil* **26,** [16] 1a) SA Pour L'Ind-Chim à Bâle, BritP 170359 (1920) & CA **16,** 1154

(1922) (Prepn & props) 2) E. Ott & E. Ohse, Ber **54,** 179-86 (1921) & CA **15,** 2069 (1921) (Prepn & props) 3) E. Ott, USP 1390378 (1921) & CA **16,** 344 (1922) (Prepn, props & proposed use in detonators) 4) C.A. Taylor & W.H. Rinkenbach, US Bur of Mines RI **2513** (1923) & CA **17,** 3607 (1923) (Prepn & detong props of Cyanuric Triazide) 5) H. Kast & A. Haid, ZAngChem **38,** 43-52 (1925) & CA **19,** 1197 (1925) (Expl props of initiating expls) 6) C.A. Taylor & W.H. Rinkenbach, JFrankInst **204,** 369-76 (1927) & CA **21,** 3462 (1927) (Sensitiveness to friction, impact & heat) 7) C.V. Hart, JACS **50,** 1925 (1928) 8) Marshall **3** (1932), 158 9) H. Muraour, BullFr **51,** 1156 (1932) & CA **27,** 603 (1933) 10) Stettbacher (1933), 20, 329, 338 11) W. Friederich, SS **28,** 114 (1933) (Rate of deton by Dautriche method) 12) A. Schmidt, SS **29,** 263 (1934) & CA **29,** 3841 (1935) (Thermochemical props) 13) E.W. Hughes, JChemPhys **3,** 1-5 (1935) & CA **29,** 1304 (1935) (Crystal structure) 14) Peppin Lehalleur (1935), 136 & 368 (Called triazoture de cyanogène or triazide cyanurique in French) 15) Davis (1943), 432ff 16) Blatt, OSRD **2014** (1944) 17) F.P. Bowden & H.T. Williams, Pr RoySoc **208A,** 176-88 (1951) & CA **46,** 5844 (1952) (Initiation & propagation of expln in azides & fulminates) 18) A.D. Yoffe, PrRoySoc **208A,** 188-99 (1951) & CA **46,** 5845 (1952) (Thermal decompn & expln of Azides) 19) Kirk & Othmer **6** (1951), 16 20) F. Moulin, Helv **35,** 175 (1952) & CA **46,** 8651 (1952) (Prepn & warning of expl nature of Cyanuric Triazide, when dry) 21) H. Koenen & K.H. Ide, Explosivst **4,** 4 (1956) (Friction sensitivity tests) 22) Sax (1957), 524 23) H. Ficheroulle & A. Kovache, MP **41,** 18-19 (1959) (Prepn, solubility & other props of Cyanuric Triazide) 24) J. Wingler et al, "Stability of Cyanuric Triazide under Storage Conditions", PATR **FRL-TR-9** (Sept 1960) 25) W.R. Tomlinson, Jr & O.E. Sheffield, AMC Pamphlet **706-177** (1963), pp 63-64 (Props of Cyanuric Triazide)

Cybernetics The comparative study of automatic control system formed by the nervous system & brain of animals & man and by mechanical-electrical communication systems in order to understand & improve communications. Missile guidance systems are composed of elements concerned with sensory perception, communication & control, which are in the general field of cybernetics

Refs: 1) N. Wiener, "Cybernetics", The Technology Press, Wiley & Sons, NY (1948) 2) H. Sh.Tsien, "Engineering Cybernetics", McGraw-Hill, NY (1954) 3) A.S. Locke et al, "Guidance" Van Nostrand, NY (1955), p 708 4) A.W. Ross, "An Introduction to Cybernetics", Wiley, NY (1956) 5) OrdTechTerm (1962), 89

Cycene (Cycène in Fr & Cicene in Ital). A Brit permitted expl patented in 1889 by M. Kitchen. It consisted of KNO_3 38.9, sugar 38.9, K chlorate 16.7 & mineral oil 5.5%. This expl is sensitive to shock & friction

Refs: 1) Daniel (1902), 178 2) Giua, Trattato **6,** (1959), 395

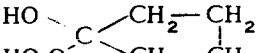
Cyclic Ketone Alcohols or Cycloalkanones. They are prepd by condensation of the corresponding cyclic ketones with formaldehyde in the presence of alkali. They are converted by reduction into cyclic alcohols. Friederich (Ref 1) found that cyclic ketone alcohols (such as tetramethylolcyclopentanone, tetramethylolcyclohexanone & octamethylolcyclohexanedione) and cyclic alcohols (such as tetramethylolcyclopentanol, tertramethylolcyclohexanol, octamethylolcyclohexanediol) on nitration yield expl products. These products are stable when stored at 50°, can be poured at temps below 100°, and have high deton velocities. The expls can be used alone, mixed with one another, or mixed with other expls (Ref 2). Kincaid & McGill (Ref 4) found that the polynitrate esters of poly(hydroxymethyl) cycloalkanones and of poly(hydroxymethyl) cycloalkanols are capable of plasticizing NC. They describe a flashless, nonhygroscopic proplnt (NGu, NC & plasticizer) which is reported superior to proplnts plasticized with a volatile HE. Derivatives of cycloalkanone which have therapeutic value were prepd by Ritzer (Ref 6)

Refs: 1) W. Friederich, BritP 345859 (1929) & CA **26,** 2058 (1932) 2) W. Friederich & K. Flick, GerP 509118(1929) & CA **25,** 819(1931) 3) M.S. Fishbein, VoyennayaKhim (Russia) **1933,** No 6, 3-8 & CA **29,** 7077 (1935) (Review of manuf & props of cyclic keto alcohols & other expls) 4) J.F. Kincaid & R.C. McGill, USP

2698228 (1954) & CA **49**, 5846 (1955) 5) A. Langhans, Explosivst **1/2**, 3-11 (1954) (Reactions of cyclic nitro compds) 6) H. Ritzer, Austrian P 198251 (1958) & CA **52**, 14706 (1958)

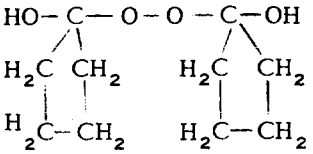
Cycloalkanes, Peroxide Derivatives of. The following peroxides or hydroperoxides were prepd & characterized by Milas (Refs) for possible use as expls:

1 - Hydroxycyclopentyl - 1 - hydroperoxide,



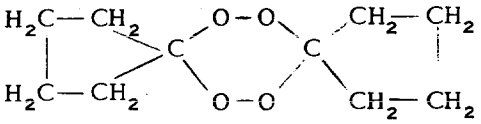
; mw 118.13, O 40.63%, active O_2 13.6%; viscous liq, highly expl

1, 1'-Dihydroxydicyclopentyl - 1, 1' - peroxide or Bis(-hydroxy-cyclopentyl) - peroxide;



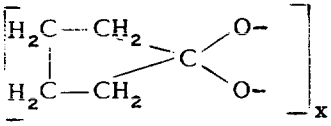
; mw 214.25, O 29.87° active O_2 7.92%, viscous liq; expl props not reported

Dicyclopentylidene Peroxide,



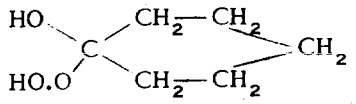
; mw 200.23, O 31.96%, active O_2 17.8%; crysts, mp 73-75°, bp dec at 105°, explodes violently when heated on a spatula

Polycyclopentylidene Peroxide,



; mw $(100.11)_x$; O 31.96%, active O_2 16%; mp 166° (with decompn), explodes violently when heated on a spatula

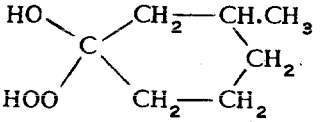
1-Hydroxycyclohexyl - 1 - hydroperoxide,



; mw 132.16, O 36.32%, active O_2 12.13%, crysts, mp 76-78° explodes mildly when heated on a spatula

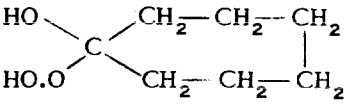
1, 1' - Dihydroxydicyclohexyl - 1, 1' - peroxide. See Bis (1-hydroxycyclohexyl) - peroxide in Vol 2, p B145-R

1 - Hydroxy - 3 - methylcyclohexyl - 1 - hydroperoxide,



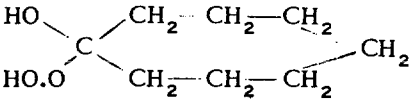
; mw 146.18, O 32.84% obtd as a highly viscous liq which, when allowed to stand at RT for a number of days, began to separate as crysts, which after several recrystns from anhyd ether melted at 120-21°. Analysis gave a compd of formula $C_7H_{12}O_2 \cdot 1/2\ H_2O_2$. It proved to be a highly expl compd which, when heated on a spatula, exploded violently

1 - Hydroxycycloheptyl - 1 - hydroperoxide,



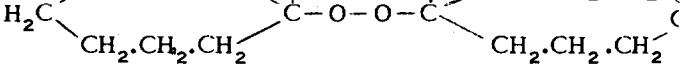
; mw 146.18, O 32.84%, active O_2 10)95% crysts, mp 92-94; expl props not reported

1 - Hydroxycyclooctyl - 1 - hydroperoxide,



; mw 160.21, O 29.96%, active O_2 10.0%; semi-solid, highly viscous product which failed to crystallize even when cooled to -78°; expl props not reported

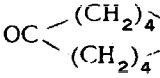
1, 1' - Dihydroxydicyclooctyl - 1, 1' - peroxide or Bis (1-hydroxy - *cyclooctyl) - peroxide,*



mw 286.40, O 22.35%, active O_2 5.6%; highly viscous substance; expl props not reported

Refs: 1) Beil - not found 2) N.A. Milas et al, JACS **61**, 2430-32 (1939) 3) N.A. Milas, USP 2298405 (1942) & CA **37**, 1448 (1943) 4) Hawkins (1961) (Cycloalkyl Hydroperoxides, 46-58)

1, 6 - Cyclodecanedione [called Cycodecandion - (1.6) in Ger],



; mw 168.23, O 19.02%; crysts (from eth), mp 100-02°; was obtd in addn to other products by the ozonization of Δ^9 - octalin in acetic acid. Its *dioxime*, $C_{10}H_{18}N_2O_2$, crysts (from alc), mp 231°, is obtd by treating with hydroxylamine hydrochloride in cold caustic KOH (Refs 1 & 2)

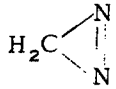
The *dimeric 1,6-cyclodecanedione dioxime*, $C_{20}H_{34}N_2O_6$, mp 220° (dec), explodes when heated in a flame (Ref 4)

1,6-Cyclodecanedione Peroxide or 1,1,6,6-Tetra-hydroperoxy - cyclodecane, $C_{10}H_{20}O_8.H_2O$, mp 116-18°, was prepd by stirring 1,6-Cyclodecanedione with 92% H_2O_2 at 0°. Its *tetra-acetate*, $C_{18}H_{28}O_{12}$, rhmb plates (from alc), dec on heating & detonates at 144-45° and by gentle friction (Ref 3). The *dimeric peroxide*, $C_{20}H_{32}O_6$, mp 166°, was formed by ozonization of octahydronaphthane in petr eth (Ref 4)

Many of the derivs of 1,6 -cyclodecanedione have been reported to be expl. Some of the intermediate compds are treacherous, and all safety precautions should be used in the prepn of cyclodecanedione derivs in order to prevent serious explns. Dietrich was critically injured during his study & investigation of these compds (Ref 3)

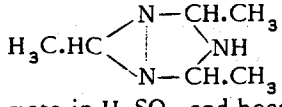
Refs: 1) Beil **7** [540] 2) W. Hückel et al, Ann **474**, 135 (1929) & CA **24**, 1638 (1930) 3) R. Criegee & H. Dietrich, Ann **560**, 140 (1948) & CA **43**, 6189(1949) 4) R. Criegee & G Wenner, Ann **564**, 13(1949) & CA **44**, 1942 (1950)

Cyclodiazo Compounds. *Cyclodiazomethane*,



; mw 42.04, N 66.64%; col, acid- resistant gas, when cooled with liq air it can be condensed to a col liq, explodes spontaneously at -40° but in soln is stable at RT; was prepd by condensing formalin with chloramine & ammonia and dehydrogenating the resulting diaziridine (Refs 1 to 4)

Cyclodiazoethane, $H_3C.HCN_2$; mw 56.07, N 49.97%; gas which can condensed by cooling, explodes on heating; readily sol in eth; was prepd by treating

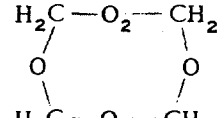


in H_2SO_4 with K dichromate in H_2SO_4 and heating (Ref 3)

Cyclodiazobutane, $H_7C_3.HCN_2$; mw 84.12, N 33.30%; liq, bp 60-78°, attempted distillation from $CaCl_2$ resulted in an expl; was obtd by treating the above compd, contg n-C_3H_7 groups instead of CH_3 groups, with $K_2Cr_2O_7$ in H_2SO_4 and distilled at 40-50° into PhCl (cooled to -78°). The org layer of the distillate was shaken with $NaHSO_4$ & aq $NaHCO_3$, dried & fractionated (Ref 3)

Refs: 1) Beil - not found 2) E. Schmitz & R. Ohme, Tetrahedron Letters **1961**, 612-14 & CA **56**, 12868 (1962) 3) E. Schmitz & R. Ohme, ChemBer **95**, 799-801 (1962) & CA **57**, 2203 (1962) 4) ChemZtg **1962**, 112 & Explosivst **1964**, 78

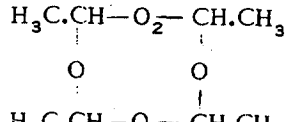
Cyclo-di (diethylether) - oxyperoxide or Cyclo-trioxytetramethylene Peroxide (Called tetraoxymethylenmonoperoxyd in Ger),



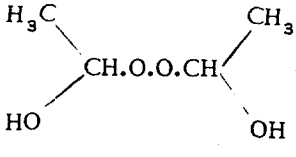
; mw 136.10, O 58.78%; vol oil, bp 35-36° at 12 mm, expl; was obtd in small quantities together with the main product, Cyclodioxytrimethyleneperoxide, by treating an ethereal soln of dimethyloldi(oxymethylene)-peroxide with P_2O_5

Refs: 1) Beil - not found 2) A. Rieche & R. Meister, Ber **66**, 720(1933) & CA **27**, 3448 (1933)

Cyclo - di (α, α - diethyletherperoxide) (called by Rieche & Meister Synthetisches dimeres Butylenozonid or Peroxydisches Acetal in Ger),



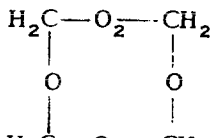
; mw 208.21, O 46.11%; thin oil, decomp violently on heating; practically insol in w but is hydrolyzed on standing; can be prepd by treating an ethereal soln of diethylolperoxide,



with P_2O_5 and evaporating the soln at RT. Some of the monomer is also formed. When the dry product is left standing, it forms a thick oil which is more expl than the original product. On prolonged heating at 90° in a vacuum, the highly expl *Ethylideneperoxide*, $(CH_3CHOO-)_x$ is formed

Refs: 1) Beil - not found 2) A. Rieche & R. Meister, Ber **65**, 1274 (1932) & CA **26**, 5906 (1932)

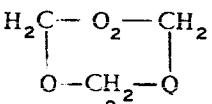
Cyclodioxytetramethylene Diperoxide or Cyclo-di (dimethyletherperoxide) [called Tetraoxymethylen-di-peroxyd in Ger and Pertetraoxymethylene in CA **27**, 3448 (1933)],



$H_2C-O_2-CH_2$; mw 152.10, O 63.11%; crysts (from eth), mp - detonates without melting at 94°; insol in w; diffc sol in org solvs; can be prepd by action of P_2O_5 on an ethereal soln of dimethylol peroxide. This compd is a violent expl, being very sensitive to heat, shock & friction

Refs: 1) Beil - not found 2) A. Rieche & R. Meister, Ber **66**, 721 (1933) & CA **27**, 3448 (1933) 3) Walker (1944), 128

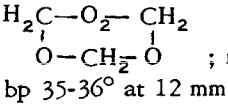
Cyclodioxytrimethylene Peroxide (called Pertrioxymethylen in Ger),



; mw 106.08, O 60.33%; liq, bp 35-36° at 12 mm, detonates when heated in test tube; d 1.2765 at 16.7°, n_D 1.4216; can be obtd by prolonged treatment of dimethylol di-etherperoxide, $(HO.CH_2O.CH_2)_2O_2$, with ether in the presence of P_2O_5. The compd is detonated also by impact & friction

Refs: 1)Beil - not found 2)A.Rieche & R. Meister, Ber **66**, 721 (1933) & CA **27**, 3448 (1933) 3)Walker (1944), 128

Cyclodioxytrimethylene Peroxide (called Pertrioxymethylen in Ger),



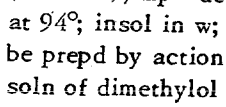
; mw 106.08, O 60.33%; liq, bp 35-36° at 12 mm, detonates when heated in test tube; d 1.2765 at 16.7°, n_D 1.4216; can be obtd by prolonged treatment of dimethylol-dietherperoxide, $(HOCH_2OCH_2)_2O_2$, with either in the presence of P_2O_5. The compd is detonated also by impact and friction

Refs: 1)Beil - not found 2)A.Rieche & R. Meister, Ber **66**, 720 (1933) & CA **27**, 3448 (1933)

Cyclofive. A castable expl consisting of RDX 53 & Fivonite (Tetramethylolcyclopentanone Tetranitrate or Nitropentanone) 47% with 1% pyrocatechol added. Its brisance by Plate Denting Test is 124% TNT and its power by Ballistic Mortar Test in 134% TNT

Refs: 1) ORSD Rept **5744** (1945) 2)OSRD Rept **5745** (1945) 3)OSRD Rept **5746** (1945), pp 8 & 14 and Table

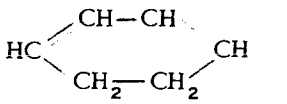
Cycloheptane, Heptamethylene or Suberane (called Cycloheptan or Suberan in Ger),



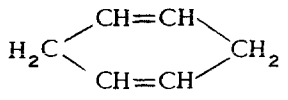
; mw 98.18, H 14.37%; col oil, fr p -12°, bp 118°, fl p <100°F, d 0.8099 at 20° (Refs 1, 3 & 4). Its Azido deriv, *Cycloheptyl Azide*, $C_7H_{13}N_3$; mw 139.20, N 30.19%; liq, bp 83.6° at 19mm, d 0.9866, n_D 1.4793 at 25° (Ref 2)

Refs: 1)Beil **5**, 29, (11), [15] & {63} 2)J.H.Boyer et al, JACS **78**, 326 (1956) & CA **50**, 12855 (1956) 3)Sax (1957), 525 4)Cond Chem Dict (1961), 325

Cyclohexadiene or Dihydrobenzene (called Cyclohexadien or Dihydrobenzol in Ger),



1,3 - Cyclohexadiene

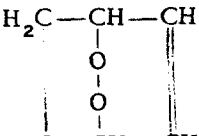


1,4 - Cyclohexadiene

C_6H_8, mw 80.12, H 10.06%. The *1,3 - Deriv*, sweet-smelling liq, fr p -104.8 , bp 80.3°, d 0.8411 at 20°, n_D 1.472 at 20°; can be prepd by cleavage of (±) - *trans* 1,2 - dibromocyclohexane with HBr and by other methods (Ref 1). Vereshchagin & Polyakova (Ref 4) found that 1,3 - Cyclohexadiene detonated, in samples less than 5cc, under the influence of a wave produced by the expln of 1.1 g Tetryl. The expln has an induction period of 5 sec after the critical pressure of 1500 atm/min is reached. It is believed that the decompn is thermal in nature. At high pressure, the rate of polymerization is so great that the heat evolved is sufficient to produce expln (Ref 4). *1,4-Cyclohexadiene*, liq, fr p - 49.2°, bp 88.8° at 764 mm Hg, d 0.8573 at 20°, n_D 1.4725 at 20°; can be prepd by treating a mixt of α- and β- 1,2,4,5 - tetrabromocyclohexane with Zn dust & glacial acetic acid in the cold, and by other methods (Ref 2). Zelinskii & Pavlov (Ref 3) found that 1,4 - cyclohexadiene reacts explosively when brought in con-

tact with Pd or Pt sponge at RT
Refs: 1) Beil **5**, 113, (60) [79] & {310} 2) Beil **5**, 113, [80] & {313} 3) N.D. Zelinskii & G.S. Pavlov, Ber **66**, 1420-22 (1933) & CA **27**, 5725 (1933) 4) L.F. Vereshchagin & A.M. Polyakova, DoklAkad N **47**, No 3, 203-04 (1945) & CA **40**, 4215 (1946)

Cyclohexadiene Peroxide or 1,4-Epidioxy-2-cyclohexene,



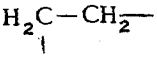
; mw 112.12, O 28.54%; solid, mp 82-83°, bp 40-55° at 0.3 mm Hg, d 1.102 at 22°, n_D 1.453 at 85°; passing O into cyclohexadiene for 100 hrs at 25° gave 45% conversion into peroxides, a portion of which is the simple monomeric peroxide (Refs 4,5 & 6)

The polymeric peroxide, $[C_6H_8O_2]_x$ explodes at 110° (Refs 2 & 3)

Refs: 1) Beil - not found 2) K. Bodendorf, Arch Pharm **271**, 1-35 (1933) & CA **27**, 4472 (1933) 3) E.H. Farmer, TrFaradSoc **42**, 228ff (1946) 4) H. Hock & F. Depke, ChemBer **84**, 355 (1951) & CA **46**, 1452 (1952) 5) Tobolsky & Mesrobian (1954), 32, 167 & 178 6) E.G.E. Hawkins, "Organic Peroxides," Van Nostrand, Princeton (1961) 232

CYCLOHEXANE AND DERIVATIVES

Cyclohexane, Hexamethylene, Hexanaphthene or Hexahydrobenzene (called Cyclohexan or Hexahydrobenzol in Ger),



; mw 84.16, H 14.37%; col mobile liq, having a pungent odor, fr p 6.3°, bp 80.7°, flash p -1°F, d 0.779, n_D 1.4263; insol in w; miscible with most lacquer solvs; can be prepd by the catalytic hydrogenation of benzene or found as a constituent of crude petroleum. The MAC of cyclohexane is 400 mg/cubic meter of air and the expl range is 1.3 - 8.4%. Cyclohexane is used as a solv for cellulose ethers, oils, fats, waxes & rubber, and as a recrystallizing medium in org synthesis
Refs: 1) Beil **5**, 20, (6), [6] & {18} 2) Sax (1957), 525 3) CondChemDict (1961), 325

Cyclohexyl Azide or Azidocyclohexane, $C_6H_{11}N_3$; mw 125.17, N 33.57%; liq, bp 64/65° at 21 mm press, d 0.9855 at 25°, n_D 1.4690 at 25°, Q_{comb} 966 k cal/mol; was prepd by replacing the bromine in cyclohexyl bromide with the azido group by reaction with NaN_3 (Ref 2) and by other methods (Refs 3, 4 & 8). See Refs 5,6,7 & 9 for addnl props
Refs: 1) Beil - not found 2) T.F. Fagley & H.W. Meyers, JACS **76**, 6001 (1954) & CA **49**, 3642 (1955) 3) L. Horner & A. Gross, Ann **591**, 127 (1955) & CA **50**, 2495-96 (1956) 4) J.H. Boyer et al, JACS **78**, 325 (1956) & CA **50**, 12855 (1956) 5) P. Gray et al, PrRoySoc **235A**, 481 (1956) & CA **50**, 15203 (1956) (Dissoc energy 91.7 k cal/mol) 6) R.L. Bennett & J.M. Scott, JPhChem **60**, 1585 1956) & CA **51**, 5485 (1957) (Dipole moment = 2.37 x 10^{18} esu) 7) E. Lieber et al, JSciIndRes-(India) **16B**, 95 (1957) & CA **51**, 13808 (1957) (Physical studies) 8) E. Lieber et al, JOC **22**, 238 (1957) & CA **51**, 16279 (1957) 9) J.H. Boyer & L.R. Morgan, JACS **81**, 3369 (1959) & CA **54**, 1395 (1960) (Acid-catalyzed reactions)

Nitro Derivatives of Cyclohexane

Mononitrocyclohexane, $C_6H_{11}NO_2$; mw 129.16, N 10.85%; straw-colored to clear liq, fr p -36°, bp 206° (dec), flash p 190°F(TOC), d 1.0666 at 25°; can be prepd by reaction of gaseous cyclohexane with N_2O_4 in a CO_2 stream at 30 or 80°.
Refs: 1) Beil **5**, 26, (10) & {54} 2) Sax (1957) 948

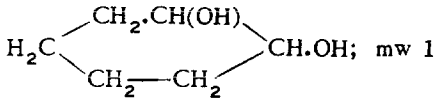
1-Nitro-2-nitrosocyclohexane, $C_6H_{10}N_2O_3$; mw 158.16, N 17.71%; triclinic ndls (from chlf & alc) or monoclinic prisms (from alc + chlf), mp 153° (dec); can be obtd by reacting cyclohexane with NO
Ref: Beil **5**, [40] & {55}

1, 2 - Dinitrocyclohexane, $C_6H_{10}N_2O_4$; mw 174.16, N 16.09%; liq, fr p 46°; was obtd, in addn to other products, by adding N_2O_4 to cyclohexane
Ref: Beil **5**, {55}

Trinitro, $C_6H_9N_3O_6$, *Tetranitro,* $C_6H_8N_4O_8$, and higher nitrated derivs were not found in Beil or in CA thru 1961

Cyclohexanediol and Derivatives

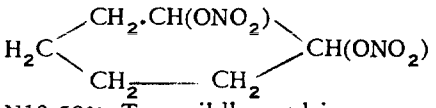
Cyclohexane - 1,2 - diol or 1,2 - Hexahydrocatechol [called Cyclohexandiol - (1.2), Hexahydrobrenzcatechin or Brenzcatechit in Ger],


; mw 116.16, O 27.55%.

Two isomers are known: *Cis,* crysts (from toluene); exists in three enantiotropic forms, the transition point lies between 75.2 - 78.1°, mp 99°, bp 118° at 14 mm Hg; and *Trans,* crysts (from benz, toluene or ether), mp 104°, bp 120° at 14 mm Hg. Other props & methods of prepn are given in Beil

Refs: Beil **6,** 739 & [743]

Cyclohexane - 1,2 - diol Dinitrate,


; mw 206.16, N13.59%. Two mildly expl isomers were prepd by nitrating the corresponding diols:

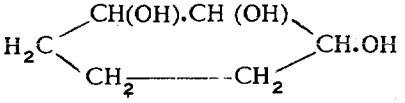
cis-Cyclohexane - 1,2-diol Dinitrate, prismatic rods (from hot ligroin), mp 24.5-25°, n_D 1.4790 at 21°; impact test value with 2 kg wt (Rotter machine) 60 cm vs 100 cm for TNT; fumes at 170°; Bergmann-Junk test 0.67 mg NO evolved/ lg sample in 20 hrs at 106°

Trans-Cyclohexane - 1,2 - diol Dinitrate, pltlts (from hot ligroin), mp 18.5 - 19°; impact test value 72-85 cm; fumes at 165°, ignites at 185°; Bergmann-Junk test 0.67 mg NO evolved/ lg sample

Refs: 1) Beil - not found 2) W.R. Christian & C.B. Purves, CanJChem, **29,** 929-33 (1951) & CA **46,** 5537 (1952)

Cyclohexanetriol and Derivatives

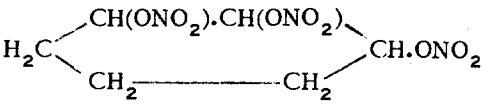
Cyclohexane - 1,2,3 - triol or Pyrogallitol [called Cyclohexantriol - (1.2.3) or Hexahydropyrogallol in Ger],



mw 132.16, O 36.32%. Four isomers of this compd exist: pltlts (from w), mp 95°, bp 225°; cis-trans-cis-(α - Pyrogallitol), ndls (from eth acet), mp 108°, bp dec above mp; cis-cis-trans-(β - Pyrogallitol), pltlts (from eth acet), mp 124°; and cis-cis-cis-(γ - Pyrogallitol), ndls (from alc), mp 145°, bp 290°. Other props & methods of prepn are given in Beil

Refs: 1) Beil **6,** 1068 (533) & [1058] 2) G.J. Gogek et al, CanJChem **29,** 938 - 45 (1951) (Synthesis of cis-cis-trans-cyclohexane - 1,2,3-triol)

Cyclohexane - 1,2,3 - triol Trinitrate,



mw 267.16, N 15.73%. Three isomeric expl compds were obtd by nitration with HNO_3 & P_2O_5 of the corresponding triols:

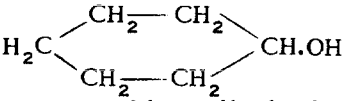
cis, cis, cis - Cyclohexane - 1,2,3 - triol trinitrate, large, thin plates (from hot ligroin), mp 74-74.5°; fumes at 155-70°; ignites at 180-82°; Betgmann-Junk test 0.95 mg NO evolved/ lg sample in 20 hrs at 106°; power by Ballistic Mortar 123% TNT; and impact test value by Rotter machine with 2 kg wt 50-60 cm vs 8 cm for NG & 100 cm for TNT

cis-cis-trans-Cyclohexane - 1,2,3 - triol Trinitrate, faintly yel oil, fr p does not freeze at -5° but becomes a colorless glass at -40°; n_D 1.4895 at 21°; fumes at 155-63°; ignites at 172°; Bergmann-Junk test 3.5 mg NO evolved/lg sample in 20 hrs at 106°; power by Ballistic Mortar 128% TNT; and impact test value with 2 kg wt 34-40 cm

cis-trans-cis-Cyclohexane - 1,2,3 - triol Trinitrate, rodlike prisms (from hot ligroin), mp 88-88.5°; fumes at 155°; Bergmann-Junk test 8.6 mg NO evolved/lg sample in 20 hrs at 106°; impact sensitivity 85 cm with a 2 kg wt

Refs: 1) Beil - not found 2) W.R. Christian & C.B. Purves, CanJChem **29,** 928-34 (1951) & CA **46,** 5538 (1952)

Cyclohexanol or Hexahydrophenol (called Cyclohexanol or Hexahydrophenol in Ger),

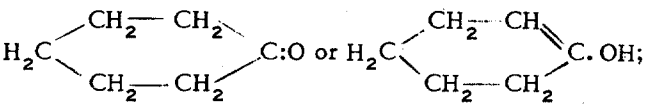

; mw 100.16, O 15.98%; col hygro liq, having a camphor-like odor; fr p 23°, bp 161°, flash p 68°, d 0.937 at 37°, n_D 1.465 at 22°; miscible with most org solvs & oils; sparingly sol in w; derived by reducing phenol with H_2 over active Ni at 160-70°F, cyclohexanone is removed by condensing with benzaldehyde in the presence of alkali. The MAC is 409 mg/cubic meter of air or 100 parts/million in air. Cyclohexanol is used in the manuf of celluloid, as a solv for rubber, NC, resins, and in a variety of ways in solv mixts

Refs: 1) Beil **6,** 5, (4) & [5] 2) Sax (1957), 525 3) CondChemDict (1961), 325

2-Nitrocyclohexanol, $C_6H_{11}NO_3$; mw 145.16, N 9.65%; col liq (crysts from chlf), mp 47-47°, bp 92° at less than 1 mm pressure; was obtd, together with other products, including the 2-Nitrocyclohexyl Nitrate, when cyclohexene was added to N_2O_4 in ether at 0°, the solvent removed, and the product poured into ice (Ref 2). IR spectra are reported by Brown (Ref 4). See also Ref 3. These compds may be employed as additives for liq hydrocarbon fuels such as diesel fuel & jet propellants

Refs: 1) Beil - not found 2) H. Baldock et al, JChem Soc **1949**, 2627ff & CA **44**, 2935 (1950) 3) T.P. Doumani et al, USP 2621205 (1952) & CA **47**, 10552 (1953) 4) J.F. Brown, Jr, JACS **77**, 6344 (1955) & CA **50**, 2297 (1956)

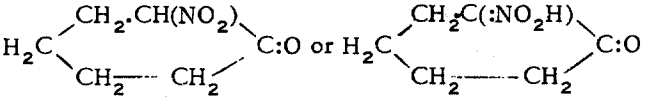
Cyclohexanone, Ketohexamethylene or Pimelic Ketone [called Cyclohexanon or Cyclohexen - (1)-ol - (1) in Ger]



mw 98.14, O 16.30%; col liq, fr p -47°, bp 157°, flash p 129°F (OC), d 0.9478 at 20°, n_D 1.4507 at 20°; miscible with most solvs; derived by passing cyclohexanol over Cu with air at 280°F, and also by oxidn of cyclohexanol with chromic acid or chromic oxide. Its MAC is 100 parts/million in air or 401 mg/cubic meter of air; 50 ppm can cause irritation of the eyes & throat. Its expl range is 1.1% at 212°F. Cyclohexanone is used in org synthesis, as a solv for expls and other materials, and in degreasing of metals, etc

Refs: 1) Beil **7**, 8, (6) & [5] 2) Davis (1943) 278 & 285 3) Sax (1957), 526 4) Cond Chem-Dict (1961), 325-26

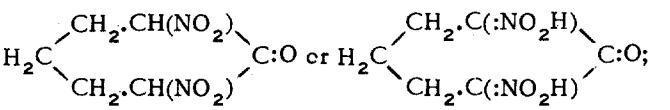
2-Nitrocyclohexanone,



mw 143.14, N 9.79%; oil or almost col prisms (from eth + petr eth), mp 37°, bp 54-60° at 1 mm; readily sol in org solvs, petr eth & caustic soda soln; diffc sol in w; was obtd by treating the K salt with H_2SO_4 in ice water. The *Potassium salt,* $KC_6H_8NO_3$, lt yel ndls (from dil acet), mp -dec at 215-20°, explodes on rapid heating; is obtd as the principal product by reaction of ethyl nitrate & K ethylate with cyclohexanone in alc + eth at -10 to -15°

Refs: 1) Beil **7**, [12] 2) H. Wieland et al, Ann **461**, 298 (1928) & CA **22**, 2553 (1928) 3) J.F. Brown, Jr, JACS **77**, 6344 (1955) (IR spectra) 4) T.E. Stevens, Chem & Ind (London) **1960**, 449 & CA **54**, 24471 (1960) (Prepn) 5) D.S. Zhuk, DoklAkadN **136**, 346 (1961) & CA **55**, 17529 (1961) (Prepn, and other products obtd by nitration of cyclohexene with HNO_3 at 100°)

2,6-Dinitrocyclohexanone,



mw 188.14, N 14.89%; ndls (from alc), mp 110)5°, v sl sol in w, diffc sol in MeOH, alc & eth giving a yel col, the alc soln colors the skin intensely yel; obtd by treating the mono K salt with an excess of dil H_2SO_4. The di K-salt, $K_2C_6H_6N_2O_5$, yel, is obtd as the main product by using twice the quantity of K & ethyl nitrate with cyclohexanone in alc + eth at -10 to -15°; with 4N H_2SO_4, the aq soln of the di K-salt gives a mono-K salt, $KC_6H_7N_2O_5$, red crysts (from w), exploding at 221°

Refs: 1)Beil **7**, [12] 2)H.Wieland et al, Ann **461**, 302 (1928) & CA **22**, 2553 (1928) 3)T. Severin, Chem Ber **92**, 1520 (1959) & CA **54**, 341 (1960) (IR spectra)

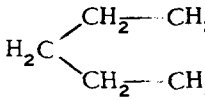
Cyclohexanone Oxime (called Cyclohexanon -oxim in Ger), C_6H_{10}:N.OH; mw 113.16, N 12.39%; hexag prisms (from ligroin), mp 88-90°, bp 204° (almost w/o decompn); readily sol in w; mod sol in alc & eth; sl sol in ligroin; obtd from reaction of cyclohexanone & hydroxylamine sulfate in alkaline soln (Ref 1). Yamamoto (Ref 2) reported that a mixt of AN (60%), Amm sulfate & cyclohexanone oxime when placed in a steel pipe & heated showed a borderline temp of 140° for its explosivity. Mixts contg 50 & 40% AN showed a temp of 150°. With less than 33% AN or below 130°, no explns were observed. The low temp of expln is accounted for by the high vap pressure of fused cyclohexanone oxime which reacts with AN, resulting in a local temp rise up to 300°

Refs: 1)Beil **7**, 10 (7) & [10] 2)S.Yamamoto, JIndExpls Soc Japan **14**, 230-32 (1953) & CA **49**, 11282 (1955) 3)G.Schulze, GerP 1042575

(1958) & CA **55**, 3474 (1961) (Prepn)

Cyclohexanone Peroxide. The compn of peroxides obtd from the interaction of cyclohexanone & other cyclic ketones with hydrogen peroxides has been a controversial matter. Milas (Ref 3) claimed to have obtd from an ethereal soln of H_2O_2 & cyclohexanone:

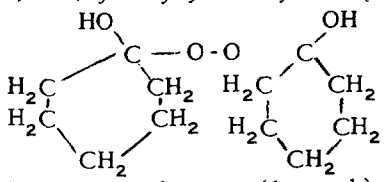
1-Hydroxycyclohexyl 1-hydroperoxide,



(Compd I)

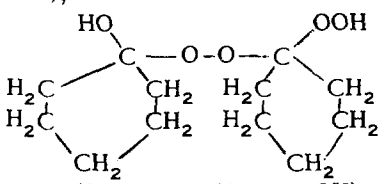
OOH; mw 116.16, O 27.55%; crysts (from eth), mp 76-78°, explodes mildly when heated on a steel spatula; and

1,1'-Di(hydroxycyclohexyl)-1,1'-peroxide,



(Compd II)

; mw 230.30, O 27.79%; col crysts (from eth), mp 68-70°, burns with a smoky flame when heated on a spatula but does not explode. The behavior of these peroxides on benzolation led Criegee et al (Ref 4) to suggest that the peroxides were actually compds II and *1-Hydroxy-1'-hydroperoxycyclohexyl Peroxide* (and not Compds I & II),

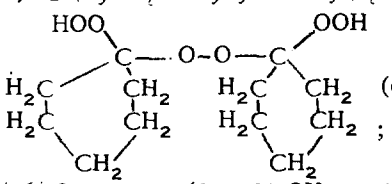


(Compd III)

; mw 246.30, O 32.48%; crysts (from AcOH), mp 78°

He also obtd, by using larger amts of 30% H_2O_2 in a reaction with cyclohexanone in ether:

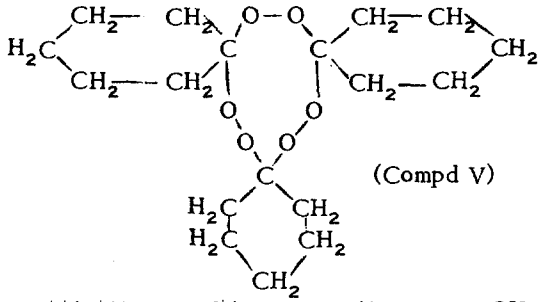
1,1'-Di(hydroperoxycyclohexyl) peroxide,



(Compd IV)

; mw 262.30, O 36.30%; crysts (from MeOH, aq AcOH or H_2O), mp 82-3°, explodes when heated in a flame. Compd IV can also be obtd from either compd II or III & H_2O_2

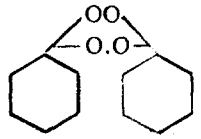
Finally, Criege (Ref 4) found that treatment of compd IV with cyclohexanone gave *Trimeric Cyclohexanone Peroxide*:



(Compd V)

mw 342.42, O 28.04%; crysts (from aq AcOH, alc or acet), mp 93°, explodes when heated in a flame

Cooper (Ref 6) described a compd as I, above, which had a mp similar to that of Criegee's compd III. By treating his compd with dehydrating agents, Cooper obtd the *Cylic Diperoxide,*

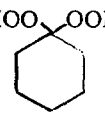


(Compd VI)

; mw 228.28, O 28.04%; crysts, mp 129°, explodes mildly at 190-200°. Cooper (Ref 7) later agreed that the compd thought to be I was in fact compd III

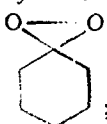
Kharasch & Sosnovsky (Ref 10) reinvestigated the reaction and concluded that, using aq H_2O_2, cyclohexanone always gave compd II in the absence of an acid catalyst. In the presence of an acid, compd III was always formed; treatment of III with perchloric acid gave the peroxide VI, in addn to high-boiling acids

According to Hawkins (Ref 12) the free *dihydroperoxide,*



has never been isolated, although its esters have been obtd. Also the monocyclic monohydroperoxide (compd I) has not been isolated (compare with Ref 8)

The synthesis of compd VI, already mentioned, was described earlier by Stoll & Scherrer (Ref 2) and Dilthey et al (Ref 4). The synthesis of *Cyclohexanone Monoperoxide,*



; mw 114.14, O 28.04%; solid (from petr eth), mp 131-33°, explodes violently when heated on a Ni spatula; prepd by treating Compd II, above, with concd H_2SO_4 (Ref 2) was also claimed recently by L'Air Liquide (Ref 11) and called *Cycohexyl 1,1-peroxide*

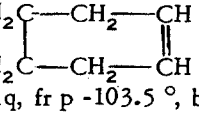
Although some measure of agreement has

been reached concerning cyclohexanone peroxides, it appears that mixts of products are often obtd, and the main product depends largely on the reaction conditions. Halbig (Ref 9) found that a mixt of cryst peroxides, with varying amts of active O, is formed by the addn of cyclohexanone to acidified aq H_2O_2. These peroxides are reported to be of interest as bleaching agents, germicides, and accelerators for combustion & explosion processes

Refs: 1)Beil - not found 2)M.Stoll & W. Scherrer, Helv **13**, 149 (1930) & CA **24**, 2733 (1930) 3)N.A.Milas et al, JACS **61**, 2431 (1939) 4)W.Dilthey et al, JPrakt Chem **154**, 229-30 (1940) & CA **34**, 2339-40 (1940) 4)R.Criegee et al, Ann **565**, 7, 11, 19 (1949) & CA **44**, 1916 (1950) 5)Lucidol Div, Novadel-Agene Corp, Buffalo, NY, Organic Peroxides Data Sheets **28** & **33** (1950) (Coml cyclohexane peroxides of which compd II, above, is the principal constituent) 6)W.Cooper, JCS **1951**, 1342 7)W. Cooper and W.H.T.Davidson, JCS **1952**, 1180 8)Tobolsky & Mesrobian (1954), 43 9)P. Halbig, USP 2750421 (1956) & CA **51**, 1260 (1957) 10)M.S.Kharasch & G.Sosnovsky, JOC **23**, 1322 (1958) 11)L'Air Liquide, FrP 1163205 (1958) & CA **55**, 3474 (1961) 12)E. G.E.Hawkins, "Organic Peroxides", Van Nostrand, Princeton, NJ (1961), 152

Cyclohexene and Derivatives

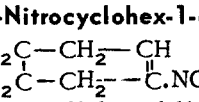
Cyclohexene or 1,2,3,4-Tetrahydrobenzene (called Cyclohexen or Tetrahydrobenzol in Ger),



; mw 82.14, H 12.27%; col liq, fr p -103.5 °, bp 82.98°, flash p 11°F, d 0.8056 at 25°, n_D 1.44377 at 25°; sol in alc; insol in w; forms azeotropes with many solvs; can be obtd by reaction of 1,3-butadiene & ethylene at 200-300° under pressure and by other methods. The MAC of Cyclohexene is 400mg/cubic meter of air (Ref 2)

Refs: 1)Beil **5**, 63, (31), [37] & {178} 2)Sax (1957), 526-27 3)Cond Chem Dict (1961), 326

1-Nitrocyclohex-1-ene,



; mw 127.14, N 11.02%; very heavy, light-yel liq, having an odor resembling NB & irritating to the mucus membranes, bp 64° at 1mm; can be prepd by heating 2-nitrocyclohexylacetate with K_2CO_3 at 105-110° or by reacting 2-nitrocyclohexylnitrate with dil NaOH and by other methods

Refs: 1)Beil **5**, [41] & {192} 2)H.Baldock et al, JCS **1949**, 2627 & 2631

Cyclohexene Nitrosite or Cyclohexene Pseudonitrosite, $C_6H_{10}N_2O_3$. See 1-Nitro-2-nitrosocyclohexane under Cyclohexane and Derivatives

Cyclohexene Nitrosate, $C_6H_{10}N_2O_4$; mw 174.16, N 16.09%; greenish oil, dec on heating above 100° and is an expl substance even on long storage in an ice box; obtd in addn to cyclohexene nitrosite by gradual introduction of a petr eth soln of N_2O_4 into a similar soln of cyclohexene below 0°

Refs: 1)Beil **5**, [40] 2)A.Schaarschmidt, Z-AngChem **36**, 565 (1923) & CA **18**, 588 (1924)

Cyclohexene Ozonide (called Cyclohexenozonid in Ger), $C_6H_{10}O_3$; mw 130.14, O 36.88%; liq having a pungent camphor-like odor, bp 47-52° at 10mm and 59-60° at 12mm; easily sol in w with partial decompn; obtd by treating cyclohexen in hexane with O_3 and concentrating the mother liquors. The solid *polymeric ozonide*, $(C_6H_{10}O_3)_x$ is obtd if cyclohexene is ozonized in dil solns (5ps in 100ps cyclohexene) with washed 9% O_3. This ozonide, mp 60-65°, explodes violently at 140-50° and is sol in hot alc, absol eth & chlf (Refs 1 & 2)

Cyclohexene ozonide is considered to be too sensitive, unstable and of insufficient power to be of military interest. Its FI (figure of insensitivity) in 56% PA and its power by Pb block is 8% PA (Refs 3 & 4)

Refs: 1)Beil **5**, (32) & [40] 2)C.Harries & R.Seitz, Ber **45**, 941 (1912); Ann **410**, 25 (1915) & CA **9**, 2877 (1915) 3)A.H.Blatt & F.C.Whitmore, OSRD **1085** (1942), 70 4)Blatt, OSRD **2014** (1944)

N-Cyclohexylacetanilide (called N-Cyclohexyl-acetanilid or Essigsäure-cyclohexylanilid in Ger), $C_6H_5.N(C_6H_{11}).CO.CH_3$; mw 217.30, N 6.45%; crysts (from eth), mp 69-70°; Q_c^v 1895 kcal/mol; Q_f^v 69.8kcal/mol (Ref 3); readily sol in alc, eth & chlf; diffc sol in hot w; can be prepd from cyclohexanol & aniline followed by acetylation. This compd may be suitable as an ingredient of proplnts

Refs: 1)Beil **12**, [144] 2)A.Guyot & M.Fournier, BullFr **47**, 207 (1930); CR **189**, 927 (1929) & CA **24**, 2992 (1930) 3)P.Tavernier & M.

Lamouroux, MP **38**, 76, 84 (1956) & CA **51**, 14404 (1957)

Cyclohexylaminoethanol or Cyclohexylethanolamine [called β-Cyclohexylamino-äthylalkohol or (β-Oxy-äthyl)-cyclohexyl-amin in Ger], $C_6H_{11}.NH.CH_2.CH_2OH$; mw 143.22, N 9.78%; oil, bp 119° at 11mm & 234-36° at 752mm; obtd by heating cyclohexylamine & glycolchlorohydrin (ClC_2H_4OH). It forms a *Picrate*, $C_8H_{17}NO + C_6H_3N_3O_7$, crysts (from alc), melting at 129°
Refs: 1)Beil **12**, [8] 2)E.Wedekind & E. Bruch, Ann **471**, 92 (1929)

Cyclohexyl-NENA or N-(2-Nitroxyethyl)-cyclohexylnitramine, $C_6H_{11}.N(NO_2).CH_2.CH_2.ONO_2$; mw 233.22, N 18.02%; col crysts (from acet & ligroin), mp 37.8-38.4°; can be prepd from cyclohexanolamine, 98% HNO_3 & acetic anhydride in the presence of a chloride catalyst. This compd was prepd as a possible NC plasticizer for use in double-base proplnts
Refs: 1)Beil - not found 2)A.T.Bloomquist & F.T.Fiedorek, USP 2485855 (1949), pp 6. 14 & CA **44**, 3516-17 (1950)

Cyclohexylaminotoluene. See Cyclohexyltoluidine

CYCLOHEXYLANILINE AND DERIVATIVES

N-Cyclohexylaniline or Cyclohexylaminobenzene (called Cyclohexylanilin, Anilinocyclohexan or N-Phenyl-cyclohexylamin in Ger), $C_6H_5.NH.C_6H_{11}$; mw 175.26, N 7.99%; prisms or pltlts on strong cooling, mp 14-16°, bp 279° at 764mm, d 0.996 at 21.5°, n_D 1.562 at 21.5°; can be prepd by heating chlorocyclohexane & aniline for 2 hrs at 125° and by other methods. Cyclohexylaniline forms numerous salts & addn compounds
Ref: Beil **12**, 172, (163) & [98]

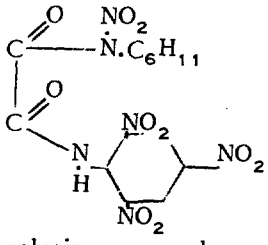
N-Cyclohexyl-2-nitroaniline or 2-Nitro-1-cyclohexylaminobenzene, $C_6H_4NO_2.NH.C_6H_{11}$; mw 220.26, N 12.72%; orn crysts (dil alc), tasteless but having an odor similar to DPhA, mp 105°; readily sol in alc, benz, chlf & acet; insol in w; was prepd by heating a soln of 1-chloro-1-nitrobenzene in alc with aq cyclohexylamine for 7 hrs at 150°. Similarly, cyclohexylamine with 1-chloro-4-nitrobenzene gave **N-Cyclohexyl-4-nitroaniline**, yel crysts, mp 100°; readily sol in benz & acet; mod sol in alc; and sl sol in w & petr eth. Nitration of either compd yields an expl
Refs: 1)Beil - not found 2)J.J.Blanksma & G.F.Wilmink, Rec **66**, 447 (1947) (in English) & CA **42**, 874 (1948)

N-Cyclohexyl-2,4-dinitroaniline or 2,4-Dinitro-1-cyclohexylaminobenzene, $C_6H_3(NO_2)_2.NH.C_6H_{11}$; mw 265.26, N 15.84%; yel crysts, mp 155°; readily sol in benz & acet; sl sol in light petroleum & alc; insol in w; was prepd by heating for 3 hrs on a w bath a soln of 1-chloro-2,4-dinitrobenzene & 45% aq cyclohexylamine. Nitration yields an expl compd
Refs: 1)Beil **12**, [407] 2)M.Busch & F.Gebelein, JPrakt Chem **115**, 111 (1927) & CA **21**, 1102 (1927) 3)J.J.Blanksma & G.F.Wilmink, Rec **66**, 448 (1947) & CA **42**, 874 (1948)

N-Cyclohexyl-2,4,6,-Trinitroaniline; N-Cyclohexyl Picramide or 2,4,6-Trinitro 1-cyclohexylaminobenzene, $C_6H_2(NO_2)_3NH.C_6H_{11}$; mw 310.26, N 18.06%; yel ndls, mp 108°; readily sol in benz, chlf & acet; mod sol in alc; insol in w; was prepd by boiling picryl chloride in alc with 45% aq cyclohexylamine. Nitration yields a more expl deriv
Refs: 1)Beil - not found 2)J.J.Blanksma & G.F.Wilmink, Rec **66**, 448 (1947) & CA **42**, 874 (1948)

N-Cyclohexyl-N-nitro-2,4,6-trinitroaniline; N-Cyclohexyl-N-nitropicramide or 2,4,6-Trinitro 1-cyclohexylnitraminobenzene, $C_6H_2(NO_2)_3N(NO_2)C_6H_{11}$; mw 355.26, N 19.71%; col, tasteless crysts, mp 165°, ignites at 330°, explodes with a loud report when heated in a sealed tube; was prepd by nitrating the above trinitro deriv with anhyd HNO_3, or by nitrating one of nitro derivs of cyclohexylaminobenzene containing either 2 or 3 nitro groups in positions 2,4 or 6. This new expl compd which has not previously been described in the literature (Ref 2), is similar to Tetryl, a std military expl
Refs: 1)Beil - not found 2)J.J.Blanksma & G.F.Wilmink, Rec **66**, 448 (1947) (in English) & CA **42**, 874 (1948)

N-Cyclohexyl-N-nitro-N'-picryloxamide or N-Cyclohexyl-N-nitro-N'-(2,4,6-trinitrophenyl)-oxamide,



; mw 426.30, N 19.72%; gelatinous compd contg H_2O (from alc), very bitter taste; mp - explodes on heating in a sealed tube; sol in acet, alc & acetic acid; sl sol in w, eth, benz & chlf; was obtd by nitrating N-cyclohexyl-N'-(2,4,6-trinitrophenyl)-oxamide (itself, a weak expl) with equal parts of anhyd HNO_3 & concd H_2SO_4 (Ref 2). Its lower nitrated products are also described
Refs: 1)Beil - not found 2)K.A.de Vries, Rec **61**, 235 (1942) & CA **38**, 2313 (1944)

Cycohexyltoluidine and Derivatives

Cyclohexyltoluidine or Cyclohexylaminotoluene (called Cyclohexyl-toluidin in Ger), $CH_3.C_6H_4.NH.C_6H_{11}$; mw 189.29, N 7.40%. Three isomers are known: *Cyclohexyl-o-toluidine,* yel oil having a sharp taste, fr p 300° (dec); *Cyclohexyl-m-toluidine,* yel oil, darkens on long storage in the light, fr p 280° (dec); and *Cyclohexyl-p-toluidine,* col lfts (from dil alc), mp 41-42°, bp 180° at 30mm of Hg. All derivs form salts. See Ref for methods of prepn
Ref: Beil **12**, [436, 467 & 493]

Mononitrocyclohexyltoluidine, $C_{13}H_{18}N_2O_2$; mw 234.29, N 11.96%. Two isomers are described in the literature: *4-Nitro-N-cyclohexyl-o-toluidine or 2-Cyclohexylamino-5-nitrotoluene,* $CH_3.C_6H_3(NO_2).NH.C_6H_{11}$, yel ndls (from alc), mp 93°, was prepd by Suschitzky (Ref 2); and *N-2-Nitrocyclohexyl-p-toluidine,* $CH_3.C_6H_4.NH-C_6H_{10}NO_2$, ppt, mp 71°, and its *Hydrochloride,* crysts (from benz & alc), mp 150-50.5°, were prepd by Topchiev & Fantalova (Ref 3)
Refs: 1)Beil - not found 2)H.Suschitzsky, JCS **1955**, 4026 & CA **50**, 9313 (1956) 3)A.V. Topchiev & E.L.Fantalova, DoklAkadN **132**, 629 (1960) & CA **54**, 24476 (1960)

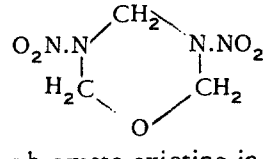
Dinitrocyclohexyltoluidine, $C_{13}H_{17}N_3O_4$, mw 279.29, N 15.05%. The following isomers are described in the literature: *3,5-Dinitro-2-cyclohexyltoluidine* $CH_3.C_6H_2(NO_2)_2.NH-C_6H_{11}$, orn-yel prisms (from alc), mp 90° (Refs 1 & 2); *4,6(?)-Dinitro-3-cyclohexyltoluidine,* dk-yel ndls (from alc), mp 137° (Refs 1 & 2); and *3,5-Dinitro-4-cyclohexyltoluidine,* orn-colored prisms (from alc), mp 78° (Refs 1 & 2)
Refs: 1)Beil **12**, [462, 481 & 538] 2)M. Busch & F.Gebelein, JPraktChem **115**, 118, 121 & 122 (1927) & CA **21**, 1102 (1927)

x-Trinitro-2-cyclohexyltoluidine, $CH_3.C_6H-(NO_2)_3.NH.C_6H_{11}$; mw 324.29, N 17.28%; lt-yel pltlts (from alc), mp 125°; was prepd by heating cyclohexyl-o-toluidine hydrochloride with concd H_2SO_4. Its expl props were not reported
Refs: 1)Beil **12**, [463] 2)M.Busch & F. Gebelein, JPraktChem **115**, 123 (1927) & CA **21**, 1102 (1927)

Cyclonite. See Cyclotrimethylenetrinitramine

Cyclonite - Nitromethane Mixture. A mixt of Cyclonite 86 & Nitromethane 14% was studied at the Univ of Toronto, Canada in 1942. This mixt has the following props:
Density 1.56-1.68, depending on pressure used
Friction Test by Sliding Shot 50% point at >240 cm vs 90 cm for RDX
Impact Sensitivity less sensitive than TNT
Influence Sensitivity shoots at 3" and fails at 4" with 60% Dynamite; cryst TNT shoots at 10" and fails at 12"
Power by Ballistic Mortar ca 146% TNT vs 148% TNT for RDX
Sensitivity to Flame burns slowly when ignited by a match
Velocity of Detonation 7720 m/sec for a self-primed 1¼" cartridge of d 1.56 g/cc
Ref: Blatt OSRD 2014 (1944)

Cyclonite Oxide or 3,5-Dinitro-dihydro 1,3,5-oxadiazine (names given by Blatt); **1-Oxa-3,5-dinitro-3,5-diazacyclohexane** (name given by Jones & Thorne); and **3,5-Dinitro-tetrahydro-2H-1,3,5-oxadiazine** (name given in CA) (called Oxyhexogène in Fr)



; mw 178.11, N 31.46%; wh crysts existing in at least 2 polymorphic forms: plates & massive form, mp 97-102°; sl sol in w; sol in HNO_3; decompd sl by concd HNO_3 while aq NH_3 has no effect; obtd

as an impurity or by-product in the prepn of RDX (Ref 2)

Its impact sensitivity is 46 cm vs 70 cm for TNT and power by Trauzl test is 397 cc at d 0.84 vs 256 cc for TNT (155% of TNT) (Ref 2)

Several investigators have studied other props of this compd (Refs 3 to 7)

Refs: 1)Beil - not found 2)Blatt OSRD **2014** (1944) 2a)L.Pauling, OSRD **5953** (1945) (Absorption spectra) 3)R.N.Jones & G.D.Thorn, CanJRes **27B**, 830, 834 & 851 (1949) & CA **44**, 2848 (1950) (UV absorption spectra) 4)W.A.Shroeder et al, Anal Chem **23**, 1741-42 (1951) & CA **46**, 5434 (1952) (UV & visible absorption spectra in alc) 5)F.Tabouis et al, MP **33**, 62-63 (1951) & CA **47**, 5684 (1953) (Impurity in RDX) 6)E.W.Malmberg et al, Anal Chem **25**, 901-07 (1953) & CA **47**, 12095 (1953) (Detection & estimation of impurities in RDX) 7)M.V. George & G.F.Wright, JACS **80**, 1200-04 (1958) & CA **52**, 18455 (1958) (Elec polarization data in dioxane)

Cycloparaffins. These are hydrocarbons in which 3 or more C atoms in each molecule are united in a ring structure, and each C atom is also joined to 2 H atoms or alkyl groups. Examples of cycloparaffins are cyclopropane, cyclobutane, cyclopentane, cyclohexane and their derivs (Refs 2 & 5)

Both the saturated & unsaturated members of the cycloparaffin series are narcotic, and may cause death thru respiratory paralysis. For most members there appears to be little range between the concns causing deep narcosis & those causing death. There is little info regarding the chronic effectd resulting from exposure of humans to cycloparaffins (Ref 4)

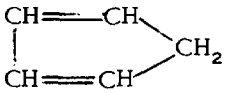
The occurrence of cycloparaffins in nature is limited to petroleums, although unsaturated cycloalkenes and cycloparaffin alcohol & ketone derivs are found widely distributed in essential oils. Naphthenes is the term more commonly used than cycloparaffins or cycloalkanes (Ref 3)

Cycloparaffins may be nitrated to produce expl derivs. See also Cycloalkanes, Peroxide Derivatives

Refs: 1)Davis (1943), 129 2)Hackh's (1944), 248 & 559 (Naphthene) 3)B.T.Brooks, "The Chemistry of the Nonbenzoid Hydrocarbons", Reinhold, NY (1950), pp 482-580 4)Sax (1957), 529 5)Cond Chem Dict (1961), 327

Cyclopentadiene and Derivatives

1,3-Cyclopentadiene (called Cyclopentadien in Ger),



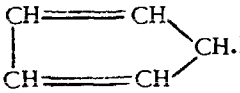
; mw 66.10, H 9.15%; liq, fr p -85°, bp 42.5°, d 0.8048 at 19°, n_D 1.4446 at 19°; miscible with alc, eth & benz; insol in w; may be obtd from the products of distillation of coal tar, and by other methods (Ref 1)

Samples of cyclopentadiene of 5g or less can be completely exploded under the influence of a deton wave produced by the expln of 1.1g of Tetryl (Ref 2)

On treating a chloroformic soln of cyclopentadiene with $SnCl_4$ for a short time and cooling to -5 to -10°, there is obtd a sol polymer, $(C_5H_6)x$, called polycyclopentadiene. By heating dicyclopentadiene ($C_{10}H_{12}$) to 200°, there is obtd an insol polymer, $(C_6H_5)x$, a col or lt-gray powd dec at 373°. Cyclopentadiene gives on nitration a nitro deriv. Dicyclopentadiene in CCl_4 on treating with ozone gives Polyclopentadiene-ozonide, $(C_6H_5O_3)x$, a very expl amorphous powd (Ref 1)

Refs: 1)Beil **5**, 112, (60), [77] & {304} 2)L.F.Vereshchagin & A.M.Polyakova, Dokl AkadN **47**, 197 (1945) & CA **40**, 4215 (1946) 3)Sax (1957), 529 4)Cond Chem Dict (1961), 327

5-Nitro-1,3-cyclopentadiene,



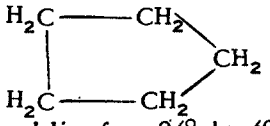
; mw 111.10, N 12.61%; yel ndls, having a penetrating odor (from petr eth), mp - very volatile & decomp on heating; was prepd by treating cyclopentadiene with $NaOCH_3$ & ethyl nitrate. Its *Sodium salt*, $NaC_5H_4N.O_2$, consists of red-brn lfts, sol in w & alc, and exploding mildly when touched with a hot wire

Refs: 1)Beil **5**, 113 2)J.Thiele, Ber **33**, 670 (1900)

Note: Higher nitrated derivs were not found in Beil or in CA thru 1961

Cyclopentane and Derivatives

Cyclopentane or Pentamethylene (called Cyclopentan in Ger),



; mw 70.13, H 14.37%; col liq, fr p -94°, bp 49°, flash p -37°, d 0.7445 at 20°, n_D 1.4067 at 20°; sol in alc; insol in w; derived from petroleum. Cyclopentane is a highly flammable compd. It is used as a solv for cellulose ethers

Refs: 1)Beil **5**, 19, (4), [4] & {10} 2)Sax (1957), 529 3)Cond Chem Dict (1961), 327

Cyclopentyl Azide or Aziodcyclopentane, $C_5H_9N_3$; mw 111.15, N 37)81%; liq, bp 51-52° at 20mm, d 0.9789 at 25°, n_D 1.4615 at 25°, Q_{comb} 820kcal/mol; was prepd by replacing the bromine in cyclopentyl bromide with the azide group by reaction with NaN_3 (Ref 2) and by other methods (Refs 3 & 8)

See also Refs 4, 5, 6 & 7 for addnl props

Refs: 1)Beil - not found 2)T.F.Fagley & H. W.Meyers, JACS **76**, 6001 (1954) & CA **49**, 3642 (1955) 3)J.H.Boyer et al, JACS **78**, 325 (1956) & CA **50**, 12855 (1956) 4)P.Gray et al, PrRoy Soc **235A**, 481 (1956) & CA **50**, 15203 (1956) (Dissoc energy 86.7 kcal/mol) 5)R.L.Bennett & J.M.Scott, JPHChem **60**, 1585 (1956) & CA **51**, 5485 (1957) (Dipole moment = 2.27 × 10^{18} esu) 6)E.Leiber et al, Anal Chem **29**, 916 (1957) & CA **51**, 12658 (1957) (IR spectra) 7)E.Lieber et al, JSciIndRes (India) **16B**, 95 (1957) & CA **51**, 13808 (1957) (Physical studies) 8)R.Lieber et al, JOC **22**, 238 (1957) & CA **51**, 16279 (1957)

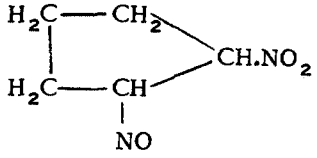
Nitrocyclopentane, $C_5H_9NO_2$p mw 115.13, N 12.17%; liq, bp 90-91° at 40mm press, d 1.0776 at 23°, n_D 1,4518 at 23°; prepd by heating cyclopentane with Al nitrate at 110-15°

Ref: Beil **5**, (5)

1-Nitro-1-nitroso-cyclopentane or Cyclopentyl Pseudonitrole, $C_5H_8(NO)(NO_2)$; mw 144.13, N 19.44%; col crysts (from eth), mp 90-98°, giving a deep-colored liq; was obtd in addn to other products by reacting $AgNO_3$ with iodocyclopentane in eth (Ref 1) and by treating cyclopentyl bromide with $NaNO_2$ (Ref 2)

Refs: 1) Beil **5**, (5) 2) N. Kornblum et al, JACS **78**, 1504 (1956) & CA **50**, 14506 (1956

1-Nitro.2-nitroso-cyclopentane or Cyclopentyl Pseudonitrosite,



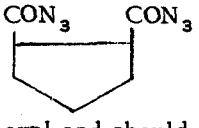
; mw 144.13, N 19.44%

Exists in two forms: crysts (from acet), mp 104-05° (dec); readily sol in acet; sl sol in chlf, alc & eth acetate; insol in eth, CS_2, Benz & gasoline; was prepd from cyclopentene & N_2O_3 in eth & petr eth under strong cooling; and crysts, mp 69-70°, of low stability; was prepd by passing nitrous gas in an ethereal soln of cyclopentane with strong cooling (Ref 1)

Refs: 1) Beil **5**, {16} 2) F. Boberg & A. Kiese, Ann **626**, 71-82 (1959) & CA **54**, 3365 (1960) (Obtd as by-product)

Note: Higher nitrated derivs of cyclopentane were not found in Beil or in CA thru 1961

Cyclopentane - 1,2 - dicarbonyl Azide,

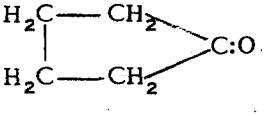


; mw 208.18, N40.37%; liq, highly expl and should not be kept free of solv; was prepd by adding PCl_5 to 1-cyclopentene - 1,2 - dicarboxylic acid in anhyd ether at ice-bath temp, the resulting acid chloride was dissolved in ether and stirred vigorously with an aq soln of NaN_3. This compd when decompd in methanolic soln gave a compd identified as the azide of 2-carbomethoxyamino - 1 - cyclopentene - 1 - carboxylic acid, $C_8H_{10}N_4O_3$, solid (from alc), mp 75-76° (dec). A soln of aniline in dry benz added to an ethereal soln of the diazide yielded the azide of 2-phenyl-ureido-1-cyclopentenecarboxylic acid, $C_{13}H_{13}N_5O_2$, crysts (from alc), mp 128-29° (dec)

Refs: 1) Beil - not found 2) J.H. Müller et al, JACS **73**, 2488-90 (1951) & CA **46**, 1457 (1952) (The subject compd is listed by authors as 1-Cyclopentene-1,2-dicarbonyl Azide and in CA 5th Decennial Index, Formula, p 244F and Subject, p 3932s as 1,2-Cyclopentanedicarbonyl Azide)

Cyclopentanone and Derivatives

Cyclopentanone, Adipic Ketone or Ketocyclopentane (called Cyclopentanon in Ger),



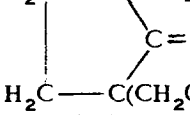
; mw 84.11, O 19.02%; water

white mobile liq, having a distinctive ethereal odor, bp 125-26° at 630 mm, fr p -52.8°, flash p (cc) 87°F, d 0.9502 at 20°, n_D 1.4370 at 20°; methods of prepn & other props are given in Beil (Ref 1). Cyclopentanone condenses with formaldehyde to form a substance, contg 4 $-CH_2OH$ groups, which can be converted directly into expl tetranitrates (Ref 2)

A residue exploded several weeks after an attempted condensation of 1,3-di(biscyclopentadienyliron)-2-propen-1-one with cyclopentanone at Eastman Kodak res labs, Rochester, NY (Ref 5)

Refs: 1) Beil **7**, 5, (3) & [3] 2) Davis (1943), 278 & 285 3) Sax (1957), 529 4) CondChemDict (1961), 327-28 5) C&EN **44**, No 49, 50 (28 Nov 1966)

Nitrocyclopentanone or 2,2,5,5-Tetrakis(hydroxymethyl)-cyclopentanone Tetranitrate (called Tetramethylolcyclopentanone Tetranitrate by Davis)



(Trivial name FIVONITE) ; mw 384.22, N 14.58%; monoclinic crysts, mp 74°, explodes 115°, d 1.62 n_D 1.568, activation energy 12.0 kcal/mol; was prepd by condensing cyclopentanone with formaldehyde and nitrating the 2,2,5,5-tetra-methylolcyclopentanone (Ref 2). Other methods of prepn are also known (Ref 4)

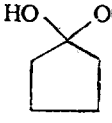
Nitrocyclopentanone possesses satisfactory stability, it is between RDX & Tetryl in impact sensitivity, and its power is about the same as that of Tetryl (Ref 4). The rate of deton is 7940 m/sec at a loading density of 1.59 g/cc (Ref 5). Many investigators have studied its props such as brisance (Ref 3), x-ray diffraction (Ref 6), UV absorption spectra (Ref 7), rate of decompn (Ref 8), rate of deton (Ref 9) and analysis by IR spectroscopy (Ref 10)

Mixts of Nitrocyclopentanone with RDX, Haleite & PETN are more powerful & more brisant than corresponding mixts with TNT, but they are also more sensitive. The compd is also a successful plasticizer for NC in double-base propellants, but a competitive material, DINA, has proved to be more satisfactory

Refs: 1) Beil - not found 2) W. Friederich & K. Flick, GerP 509118 (1929) & CA **25**, 819 (1931) 3) W. Friederich, SS **28**, 2, 51, 80 & 113 (1933) & CA **27**, 3079 (1933) 4) NDRC Summary Rept of Div 8, Vol 1 (1946), 24-25 5) Davis (1943), 285, 6) A.M. Soldate & R.M. Noyes, Anal Chem **19**, 443 (1947) & CA **41**, 6105 (1947) 7) W.A. Schroeder et al, AnalChem **23**, 1742 (1951) & CA **46**, 5434 (1952) 8) H. Henkin & R. McGill, IEC **44**, 1393 (1952) & CA **46**, 8857 (1952) 9) A. R. Martin & H.J. Yallop, TrFaradSoc **54**, 257 (1958) & CA **52**, 17715 (1958) 10) F. Pristera et al, AnalChem **32**, 506 (1960) & CA **54**, 12586 (1960)

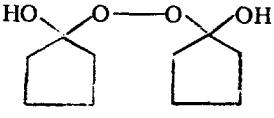
Cyclopentanone Peroxides. Milas et al (Ref 2) found that the reaction of cyclopentanone & H_2O_2 yielded a variety of peroxides:

1-Hydroxycyclopentyl Hydroperoxide - 1,



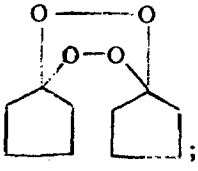
, viscous residue, highly expl;

1,1¹-Dihydrodicyclopentyl Peroxide - 1,1¹



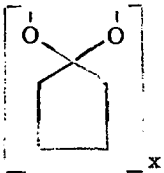
, viscous residue;

Dicyclopentylidene Peroxide,



; crysts, mp 73-75°, gas evolved at 105, explodes violently when heated on a spatula;

Polycyclopentylidene Peroxide,



, solid (from dioxane), mp 166° with effervescence, explodes violently when heated on a spatula

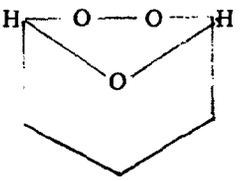
Dilthey et al (Ref 3), by dropwise addn of cyclopentanone to a mixt of acet, acetic anhydride, H_2SO_4 & H_2O_2 at 15° and stirring, obtd a *Trimeric Peroxide*, prisms (from alc), mp 172° (decomp); when the filtrate was poured into water, a *Dimeric Peroxide*, small ndls (from alc or benz), mp 105°. Both peroxides are powerful expls & sensitive to impact

Hawkins (Ref 5) feels that the structures of the above peroxides appear to be based solely on active oxygen content and that further structural proof should be provided (See also Ref 4)

Refs: 1) Beil - not found 2) N.A. Milas et al, JACS **61**, 2430-31 (1939) 3) W. Dilthey et al,

JPraktChem **154**, 231-32 (1940) & CA **34**, 2340 (1940) 4) G. Lohaus, Ann **583**, 6-12 (1953) & CA **49**, 1588 (1955) 5) E.G.E. Hawkins, "Organic Peroxides", Van Nostrand, Princeton, NJ (1961), 152 & 160

Cyclopentene Monomeric Ozonide,

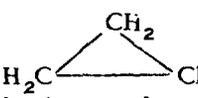


; mw 84.11, O 19.02%; col ndls (from hexane, followed by sublimation), having a pungent odor, mp 76°, bp 60° at 11mm, defgr mildly when heated in a free flame; sol in org solvs insol in w; was obtd when cyclopentene in hexane was ozonized at 12-14° with O_3

Refs: 1) Beil - not found 2) G. Blust & G. Lohaus, Ann **583**, 5(1953) & CA **49**, 1588 (1955)

Cyclopropane and Derivatives

Cyclopropane or Trimethylene (called Cyclopropan in Ger),



; mw 42.08, H 14.37%; col gas having an odor resembling that of solv naphtha & having a pungent taste; fr p -127°, bp -32.9°, d 1.7352 at -80° to 0.6769 at -30°; sol in alc; MAC 400 ppm in air or 688 mg/cu meter of air; expln range 2.4 to 10.4%. Cyclopropane is a highly flammable gas, forming expl mixts with air & O. Other props & methods of prepn are given in Beil (Ref 1)

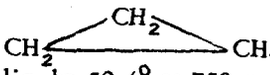
Refs: 1) Beil **5**, 15, (3), [3] & {3} 2) Sax (1957), 530 3) CondChemDict (1961), 328

Mononitrocyclopropane, $C_3H_5NO_2$; mw 87.08, N 16.09%; liq, having a mild, sweet odor, bp 65-67°, bp 142-43.5° at 742 mm, d 1.346 at 20°, n_D 1.4398 at 20°; was prepd by nitrating cyclopropane with HNO_3 and/or N_2O_4 in the vapor phase. Nitrocyclopropane is stable on storage at RT for months; can be distilled at atm press with only sl decompn; and is not decompd or polymerized by continued exposure to light, air, dil HCl, aq $NaHCO_3$ or concd aq NaOH

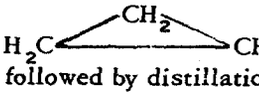
Refs: 1) Beil - not found 2) H.B. Hass & H. Schechter, JACS **75**, 1382-84 (1953) & CA **48**, 3269-70 (1954)

Note: Higher nitrated derivs of cyclopropane were not found in Beil or in CA thru 1961

Cyclopropylacetylene or Ethenylcyclopropane,



, mw 66.10, H 9.15%; liq, bp 52.4° at 750 mm, d 0.7828 at 20°, n_D 1.4284 at 20°; was prepd from vinylcyclopropane,

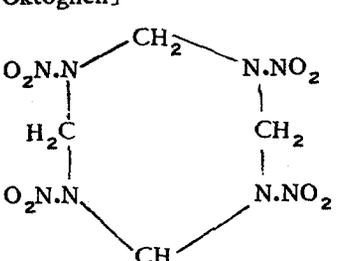


, thru bromination followed by distillation with KOH dissolved in ethyl Cellosolve. Its *Silver salt* explodes on heating

Refs: 1) Beil - not found 2) Ya. M. Slobodin & I.N. Shokhor, ZhObshchKhim **22**, 195-99 (1922) & CA **46**, 10112 (1952); JGenChem, USSR **22**, 243-47 (1952) & CA **47**, 5899 (1953) (English translation)

"Cyclops". A high-energy proplnt described in conf rept of Callery Chemical Company, "Preparation and Characterization of High Energy Monopropellant "Cyclops", Tech Rept **SSD-TR-61-42**, Final Report (Conf) (Dec 1961), Contract AF04 (611)-7040

CYCLOTETRAMETHYLENETETRANITRAMINE; 1,3,5,7-TETRANITRO-1,3,5,7-TETRAZACYCLOOCTANE; OCTAHYDRO-1,3,5,7-TETRANITRO-1,3,5,7-TETRAZINE; HOMOCYCLONITE; OCTOGEN or HMX (called in CA Octahydro - 1,3,5,7-Tetranitro-1,3,5,7-tetrazocine) [called in Gr Britain HMX (High Melting Explosive of His Majesty's Explosive), in France Cyclotetraméthylènetetranitramine or Octogène, in Germany Octogen and in Russia Oktoghen]



; mw 296.17, N 37.84; wh crysts (from nitromethane), mp 276-80 with decompn), d 1.90, sol in solvs which dissolve RDX (qv); insol in w & nonhygroscopic. HMX is formed under some conditions, in the nitration of hexamethylene-tetramine, hence it is

present in Type B RDX

Preparation (Ref 7). In the first stage, hexamine, glacial acetic acid, acetic anhydride, AN & HNO_3 are added simultaneously, continuously & equivalently and the temp is maintained at 45°. The reaction mixt is aged 15 minutes. The second stage reactants: AN/HNO_3/ acetic anhydride are added over a 25 minute period and aged 65 minutes. The mixt is poured into water and simmered on a steam bath for 12 hrs. The ppt of RDX-HMX contains 73% of HMX. The RDX is destroyed, leaving HMX, by placing the crude product in a soln of Na tetraborate decahydrate in water, heating to boiling, and addg 5N NaOH. When the pH increases from 8.7 to over 9.7, complete destruction of the RDX has taken place. The HMX is filtered from the hot mixt & recrystd from nitromethane. This is the beta form of HMX

The present method of prepg HMX is essentially that of earlier workers (Refs 2,6,7,10,11, 12,13,14,16 & 23), however, it is somewhat refined and paraformaldehyde is added to increase the yield (Ref 18)

Procedure: To a 6 to 10-liter flask equipped with stirrer, thermometer & 3 dropping funnels, add 785g of acetic acid, 13g of acetic anhydride & 17 g of paraformaldehyde while maintaining the temp at 44 ± 1° with a water bath. Add over a 15 minute period, with constant stirring, a soln of 101g of hexamine in 165g of acetic acid, 320g of acetic anhydride & 180g of HNO_3-AN soln (prepd by dissolving 840g of AN in 900g of HNO_3 99% or stronger). The hexamine & HNO_3 are added continously and proportionally. The mixt is aged for 15 mins. Then add 320g of acetic anhydride & 271 of HNO_3-AN soln proportionately, and then 160g of acetic anhydride in bulk. Allow the mixt to age for 60 mins and reflux for 30 mins after addg 350g of hot water. Cool to 20° by addg ice, collect the water-insoluble ppt and wash with 3 portions of cold water. The yield of 200g HMX is 95% of theory and the purity is higher than 90%. This product is alpha HMX which is converted to beta form by crystn from boiling acetone, acetonitrile or cyclohexanone using equal parts by wt of solid & solvent. The recrystd HMX has a mp of 278-79°

Polymorphs. HMX exists in 4 polymorphic modifications between room temps & its mp (280°). These polymorphs are known as alpha, beta, gamma & delta forms. With each polymorph, there is associated a stability range, and differences in physical props such as density, solubility & refractive indices. These polymorphs can also undergo transformations

The common modification of HMX is the beta form. Therefore, when the unqualified term HMX is used, it is understood to be the beta form. This modification is prepd by the very slow cooling of solns of HMX in acetic acid, acetone, nitric acid or nitrometrane. The crysts formed are monoclinic with a density of 1.90 g/ml

Alpha HMX can be prepd by crystg from the same solvs as used in the prepn of beta HMX, except that the solns must be cooled rapidly. Its cryst form is orthorhombic with a d of 1.82 g/ml

Gamma HMX is prepd from the same solvs as alpha & beta forms but with much more rapid cooling. The crysts are monoclinic with a d of 1.76 g ml

Delta HMX can be crystd most readily from solvs in which it is only sl soluble, such as acetic acid or tris(β-chloroethyl) phosphate, and rapid chilling, preferably in small amts poured over ice. The crysts are hexagonal with a d of 1.80g/ml

The polymorphs of HMX may be obtd also by heating, for example, beta HMX is converted to alpha above 102-104.5°; alpha to gamma above 160-64°, gamma is metastable to 160-64°; and above 160-64° to melting point delta is formed. However, these values are controversial (See Refs 15 & 28). More detailed info on the prepn of HMX polymorphs is given in Refs 5, 29 & 30.

The order of sensitivity of the polymorphs of HMX may be summarized as follows: delta > gamma > alpha > beta. The range of values obtd using an Explosives Research Laboratory, Bruceton, Pa Apparatus (Type 12) with a 2.5 kg wt is as follows: delta 19.2, gamma 13.8 to 33.9, alpha 15.6 to 26.4, and beta 21.2 to 24.9 cm. A value of 12 cm for PETN was obtd under the same conditions (Ref 28)

Explosive and Other Properties of beta HMX

(Refs 29 & 31). *Brisance by Sand Test:* 54.4 to 60.4 g sand crushed vs 48g for TNT

Chemical reactivity: comparable with RDX except that HMX is not easily decompd by alkaline hydroxide, and concd H_2SO_4 liberates a little more than 1/3 of HNO_3 while with RDX a little more than 2/3 is liberated

Confined "cook-off" temperature: 210°

Explosion tempetature: 306° in 10 secs, 327°

in 5 secs & 380° in 0.1 sec with no cap used
Friction pendulum test: explodes with steel shoe, unaffected by fiber shoe
Hardness by Mob's scale: 2.3
Heat of combustion: 667.4 kcal/mole and 2255 to 2362 cal/g
Heat of explosion: calcd with H_2O liq 1356 and calcd with H_2O gas 1222 cal/g
Heat of formation : -17.1 kcal/mol and -60.5 cal/g
Heat test at 100°: % loss 1st 48 hrs 0.05, % loss 2nd 48 hrs 0.03, and no expln in 100 hrs
Hygroscopicity at 30° & 95% RH: 0.0%
Impact sensitivity: Bur Mines App, 2 kg wt - 32cm for a 20mg sample; PA App, 2 kg wt 9 inches for a 23 mg sample vs 95^+ & 14^+ as comparable values for TNT
Power: by Ballistic Mortar 150% TNT; by Trauzl Test 145% TNT
Sensitivity to initiation: Minimum detong chge reqd for max brisance 0.30g of LA
Solubility: grams HMX/100g of soln - 4.4 in dimethylformamide, 0.129 in nitrobenzene at 25°, 0.125 in 1,2-dichlorethane at 70°, 0.0375 in acetic acid at 25°, 0.96 in acetone at 25°, 1.98 in acetonitrile at 25°, 2.11 in cyclohexanone at 25°, 0.02 in ethyl acetate at 25°, 0.02 in ethyl bromide at 25°, 0.46 in methylethylketone at 25°, 0.172 in nitroethane at 25°, 0.778 in nitromethane at 25°, and 1.75 in triethylphosphate at 25°;
gms HMX/100 ml of soln - 21 in gamma-butyrolactone, 1.3 in cyclopentanone, 5.2 in cyclohexanone, 2.2 in acetone, 2.0 in acetonitrile, 1.1 in nitromethane, 0.03 in nitroethane, and 1.8 in methylisobutylketone (hexone);
gms HMX/100 ml of solv - 0.003 in chloroform, 0.002 in carbon tetrachloride, 0.144 in dioxane, and insol in carbon disulfide
Specific heat:

Temp °C	cal/gm/°C
-75	0.153
0	0.228
25	0.248
50	0.266
75	0.282
85	0.288
90	0.290
100	0.295
125	0.307
150	0.315

Thermal stability: HMX is more stable than RDX as indicated by vacuum stability & expln temp tests. The 150° Vacuum Stability test shows HMX to be comparable to TNT & Ammonium Picrate in thermal stability
Vacuum stability test: cc gas evolved/40 hrs/ 5g sample - 0.37 at 100°, 0.45 at 120°, and 0.62 at 150°
Velocity of detonation: 9124 meters/sec at d 1.84
Uses. HMX is used as an expl chge alone when desensitized, as a booster chge, in admixtures with TNT *(Octols)*, and as an oxidizer in solid rocket & gun propellants

US military requirements & tests of HMX as covered by Specification MIL-H-45444A(3) (Ref 30) are as follows:
Purity - Grade 1, min 93% and Grade 2, minimum 98%, as determined by treating an accurately weighed 0.2g sample with 100ml of 1,2-dichloroethane saturated with HMX. Shake the mixture for 1 hr and filter, using a fritted glass crucible, and vacuum. Wash the residue in crucible with two 100ml portions of diethylether. Weigh the insoluble HMX and calculate %
Material - shall consist of the beta polymorph only as determined by the examination of a 5% mull of the sample in hexachlorobutadiene or tetrachloroethylene by means of a Perkin Elmer Double Beam Infrared Spectrophotometer, Model 21. The mull is prepd by grinding the sample one min in dry state and then adding hexachlorobutadiene and again grinding for another min. The spectrophotometer has a drum setting for the slit of 980. This gives a slit of about 460 microns at 13.5 microns and a slit of 580 microns at 14.5 microns. A gain setting of 7 is found satisfactory. The prepared 5% mull is transferred to a 0.5 mm sodium chloride cell (sample cell). A 5% mull of pure beta HMX (the beta HMX in the compensating beam as fine as obtainable, preferably less than 200 mesh) is transferred to another 0.5 mm sodium chloride cell (compensating cell). The cells are placed in the instrument. It is recommended that the compensating cell be placed in the instrument about a minute before the sample cell. This will allow the mull in the compensating cell to settle a little so that the starting transmittance reading of the sample will be about 60% at 13.5 microns. If the mulls settle out on standing or run they can be remixed by inverting the cell a few times. A small bubble of air will aid in the mixing. The spectrogram shall be examined for the possible presence of an absorption band at about 14.0 to 14.3 microns. If alpha and/or gamma HMX is

present in amounts of 5% or over in the sample, a band or some definite absorption will be observed between 14.0 and 14.3 microns. The sample is run in duplicate. One or two "blank" spectrograms are prepared using a 5% mull of pure HMX in both cells, the cell with the greater absorption is used as the sample cell. If there is any doubt, prepare one standard containing 5% alpha HMX in beta HMX, and one standard containing 5% gamma HMX in beta HMX. Run these standards as described for the sample and use the obtained spectra to evaluate the sample (Ref 26)

Melting Point min 270°C, as determined by means of a Fischer-Johns hot stage melting point apparatus

Total Acetone Insoluble - shall not exceed 0.05% as determined by dissolving 10 gm of sample in 400 ml of acetone with the aid of heat and filtering, using a filtering crucible. The increase in weight of crucible is the weight of insoluble

Inorganic Insoluble - shall not exceed 0.03% as determined by igniting the total acetone insoluble and weighing

Insoluble Particles - no insoluble particles shall remain on a US Standard No 40 Sieve and not more than 5 insoluble particles on a No 60 as determined by extracting 50g of sample in a Soxhlet extractor and brushing insoluble material thru a No 40 and a No 60 Sieve

Granulation - the uses of HMX are such, that 6 classes are required: Class A to Class F, inclusive - the coarsest crystals being retained on a US Std Sieve No 12 and finest crystals passing thru a a No 325. The granulation is determined by aid of a mechanical sieve washer and pressure tank, using ethanol sat with HMX or a spray nozzle under water tap pressure, and a 2% solution of a wetting agent such as dioctyl sodium sulfosuccinate (Aerosol-OT)

Acidity - 0.02% max, calculated to acetic acid, as determined by dissolving HMX in acetone, adding water until the ratio of acetone to water is 4 to 1 and titrating with 0.05N NaOH using phpht indicator

Shipping HMX is shipped in water - tight bags (rubber, rubberized cloth, plastic) in form of a slurry or cake containing not less than 10% by weight of a solution consisting of 40% by weight of isopropyl alcohol and 60% water

Analytical Procedures:

Detection. HMX can be detected in the presence of nitrates & nitramines (RDX) by a sulfuric acid soln of DPhA (diphenylamine). The test sample, on a white spot plate, is treated with a drop of soln of DPhA in 88% H_2SO_4. A blue color in 1 min is obtd with nitrates & other nitramines (RDX). HMX requires 5 mins or more. The test is made with a cold reagent. How cold is not specified by the authors (Ref 22). Diphenylbenzidine instead of DPhA gives more reproducible results. HMX may also be detected by the following test (differentiation from RDX). Add an excess of dimethylformamide to the sample. Evaporate the excess and hydrolyze to dimethylammonium chloride by warming with HCl. Add successively: $CuSO_4$, NH_4OH, CS_2 & Benz. The presence of HMX is indicated by the brown color of the Cu dimethyldithiocarbamate in the benz (Ref 32)

HMX when dissolved in concd sulfuric acid, produces a red-violet color in the presence of ferrous ions (ferrous ammonium sulfate) (Refs 25 & 27). HMX may also be detected by its infrared spectrogram and x-ray diffraction pattern

Quantitative Determination:

HMX may be detd by the Chromous Chloride Method of Gutmacher & Selig (Ref 26b). The method, essentially, consists of the reduction of HMX with $CrCl_3$ in a dimethylformamide & HCl solution. Excess $CrCl_3$ is back titrated with ferric ammonium sulfate using phenosafranine as indicator. The method is reported to give a 99.5-100% recovery. The method which is a modification of Jamison's method (See Ref 26a) is applicable to HMX on a micro scale. The procedure is as follows:

Prepare ferric ammonium sulfate (Fe alum) (0.05N) by dissolving 50g in 800 ml of water in a 2 liter volumetric flask. Add 50 ml concd H_2SO_4 and dilute to volume. Prepare chromous chloride (0.05N) by dissolving 27.5g $CrCl_3 \cdot 6H_2O$ in 500 ml water in a 2 liter vol flask. Add 50 ml concd HCl and dil to vol. Pour into a Machlett buret containing 1 lb of amalgamated zinc. Allow to stand overnite in a nitrogen atmosphere. Maintain this atmosphere at all times. A bright blue color indicates complete reduction of chromic (green) to chromous ion. Prepare amalgamated zinc by allowing 1-2 lbs mossy Zn to stand for 20 mins in a liter of 15-18% HCl contg 10-15g mercuric chloride. Decant and wash the amalgamated zinc with dist w. To det the HMX, dissolve the sample contg not more than 1.9 mg of nitro groups (3 mg sample) in 10 ml of di-

methylformamide. This weight of sample insures a 500% excess of $CrCl_3$. Add 15 ml concd HCl. Sweep the air from the titrating flask with nitrogen for 10 mins while stirring with a magnetic stirrer. Maintain a nitrogen atmosphere and a 70° temp thruout titration. Add 25ml $CrCl_3$ and stir for another minute. Add 8 drops of phenosafranine. Titrate with ferric alum to a color change from light green to a grey-dark green. Test a blank and calc % HMX as follows:

$$\% \text{ HMX} = [1.234\ N(V_1 - V_2)]/W$$

where V_1 = ml of Fe^{+++} alum soln used to titrate blank

V_2 = ml of Fe^{+++} alum soln used to titrate excess chromous chloride

N = Normality of Fe^{+++} alum soln

1.234 = the factor obtd by dividing the mw of of HMX (296) by the total electron change of 4 nitro groups undergoing reduction (6 x 4 = 24)

W = weight of sample in grams

Quantitative Determination of HMX in the Presence of RDX. A résumé of the analytical methods for RDX & HMX, reported to June 1965, is given in Ref 32. For the control analysis of HMX, two methods merit consideration (Refs 30 & 32). The method of Scullion et al (Ref 32) depends upon the formation of a stable 1:1 molecular complex of HMX & dimethylformamide (DMeFA) which can be filtered & washed with CCl_4 w/o appreciable loss. The addn of DMeFA to a simple mixt of RDX & HMX and the removal of excess DMeFA in a vacuum oven at 40° affords a gravimetric method of detg HMX from the increase in wt of sample. The HMX/DMeFA complex also has characteristic optical crystallographic, absorption spectrum & colorimetric properties which are of considerable distinctiveness and consequent diagnostic value. For example, the "spot" test for HMX involves treating the sample with excess DMeFA, evaporating the excess DMeFA, hydrolyzing to dimethylammonium chloride by warming with HCl, and then treating successively with copper sulfate soln, ammonia, carbon disulfide & benzene. HMX is indicated by the brown color of the Cu dimethyldithiocarbamate in the benz

The Spec method (Ref 30) requires a special reagent prepared by stirring 1,2-dichloroethane at RT for 4 hrs in contact with HMX crysts. The solubility of HMX in 1,2-dichloroethane at 24° is 0.02/100 ml of solvent. Weigh a dried 0.02g/100ml sample of HMX, to the nearest milligram, in a 125 ml glass-stoppered conical flask. Add 100 ml of the 1,2-dichloroethane that has been saturated with HMX. Shake the flask and contents on a wrist-action shaker for 1 hr. Transfer the sample residue from the flask into an accurately weighed, fine porosity 30 ml fritted glass crucible attached to a filtering flask connected to vacuum. Rinse the flask and wash the residue in the crucible 2 times with 100 ml portions of diethylether. Suck dry under vacuum for 15 mins. Allow the crucible to come to RT and weigh crucible & insol residue. The insoluble residue is HMX. The percentage of RDX in the sample is given by:

$$\% \text{ RDX} = (Ws - Wr)/(Ws \times 100)$$

where Ws = original wt of sample in grams

Wr = insoluble residue wt in grams

An instrumental method (Ref 18) also is indicated to be satisfactory. In this method, the HMX content is detd by the measurement of the difference in the refractive indices of these compds in nitromethane soln with the aid of a Brice-Phoenix Differential Refractometer. The method is reported to have an accuracy and precision of ± 0.2% and ± 0.5%, respectively, and appears to be sufficiently rapid for a routine control analysis after the prepn of graphs

For a method to be used to follow the course of a chemical research, the method given in Ref 33 might be used to advantage. In this method RDX is detd in HMX in concns of 0.05 to 5% by Thin-Layer Chromatography on alumina coated plates. The RDX is recovered from the substrate by extraction with acetonitrile and is measured by spectrophotometry at 228 millimicrons. See also Analytical Procedures under RDX in this Volume

Note: This Section was written in collaboration with E.F. Reese of PicnArsn)

Refs for HMX: 1) Beil - not found 2) F.C. Whitmore, "The Preparation of HMX", OSRD Rept **652** (June 1942) (PB 30776) 3) J.R. Johnson, "Crystallographic Studies of RDX, HMX, and Related Compounds", OSRD Rept **694** (July 1942) 4) W. E. Bachmann, "Studies on the Preparation and Properties of RDX", OSRD Rept **820** (Aug 1942) (PB 34798) (Some work is reported on the structure of HMX & BSX) 5) A.T. Blomquist, "The Polymorphism of HMX", OSRD Rept **1227** (Feb 1943) 6) R. Connor et al, "The Analysis of RDX-HMX Mixtures", OSRD Rept **1711** (Aug 1943) 7) W.E. Bachmann, "The Preparation of HMX", OSRD Rept **1981** (Nov 1943) 7a) Blatt, OSRD **2014** (1944) 8) A.T. Bloomquist, "Microscopic

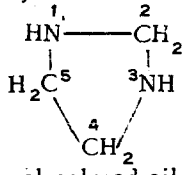
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Cyclotols See under COMPOSITION B TYPE EXPLOSIVES AND CYCLOTOLS

Cyclotols, Analytical Procedures. See Composition B Type Explosives and Cyclotols, Analytical Procedures

Cyclo - 2,4,5 - trimethylene - 1,3 - diamine and Derivatives

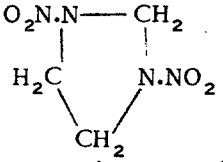
Cyclo - 2,4,5 - Trimethylene - 1,3 - diamine; 1,3-Diazapentane or Imidazolidine (called Tetrahydroimidazol in Ger),



; mw 72.11, N 38.85%; obtd as a yel-colored oil, which was not isolated as such, by reaction of ethylenediamine in HCl with HCHO and cooling. Its props were not reported although

some derivs were prepd (Ref 2)
Refs: 1) Beil - not found 2) H. Krässig, Makromol Chem **17**, 82, 105-06 (1956) & CA **50**, 13953-54 (1956) 3) J.A. Blanchette, USP 2959572 (1960) & CA **55**, 5028 (1961) (Mentioned as useful in coatings)

1,3-Dinitro-cyclo-2,4,5-trimethylene - 1,3 - diamine; 1,3 - Dinitro - 1,3 - diazacyclopentane or 1,3 - Dinitroimidazolidine



; mw 162.11, N 34.56%; wh crysts (from 95% alc), mp 132.5 - 134°; was prepd by treating paraformaldehyde in 87% H_2SO_4 with EDNA (Ethylenedinitramine) and addg the solid product to chilled 100% HNO_3, or by addg N-methylol EDNA to 89% H_2SO_4 and pouring the mixt onto ice (Ref 2). This and other related condensation products are useful in the synthesis of substituted hydrazines

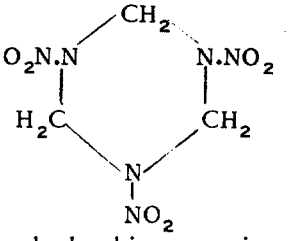
The kinetics of the alkaline decompn of the subject compd was studied by Jones (Ref 3)

Simkins & Wright (Ref 4) prepd the compd by different methods and detd its expl props, which are as follows: 1) comparable to Tetryl in expl props 2) FI - 78% of Picric Acid 3) power by Trauzl test 136% of PA 4) Decomp w/o ignition at 205°, but ignites at once at 350° 5) decompd rapidly by hot 50% H_2SO_4 and 6) forms a eutectic, with 14% TNT, melting at 70.5°
Refs: 1) Beil - not found 2) L. Goodman, JACS **75**, 3019-20 (1953) & CA **49**, 4629-30 (1955) 3) W.H. Jones, JACS **76**, 928-29 (1954) & CA **49**, 10932 (1955) 4) R.J.J. Simkins & G.F. Wright, JACS **77**, 3157-59 (1955) & CA **50**, 3469 (1956) 5) ADL, Synthesis HE's **4** (1956) p 94 (Conf, not used as a source of info)

Cyclotrimethyleneimine. See Azetidine, Vol 1, pp A518-19

CYCLOTRIMETHYLENETRINITRAMINE, CYCLONITE OR RDX

(also called Hexahydro- 1,3,5 - trinitro- s-triazine; sym-Trimethylenetrinitramine; Trinitrotrimethylene-triamine; 1,3,5 - Trinitro - 1,3,5 - triazacyclohexane; and in French Hexogène, T4 & Cyclotriméthylène-trinitramine; in German Hexogen, E-Salz, K-Salz, KA-Salz, SH-salz & W-Salz; in Italian Exogene, T4 & Trimetilenetrinitroamina; in Japanese Shouyaku & Tanayaku; in Russian Gheksoghen; in Spanish Ciclonita, Exógeno & T4; and in Swedish Hexogen),



;mw 222.13, N37.84%; wh orthorhombic crysts in a wide variety of habits from ndls (from HNO_3), plates (from acetic acid) to massive form (from nitromethane or acet); mp sublimes readily before melting at 205° (with decompn). An unstable polymorph of RDX can be obtd by recrystn on a microscope slide from high boiling solvs such as thymol, nitrobenz, aroclor & TNT. This modification has not been characterized because of the difficulty of obtg & preserving well formed crysts. Because of its physical instability, there is no possibility of obtg this polymorph even in lab recrystallizations (Ref 102). See also Refs 35 & 46

Pure RDX has a cryst d 1.816, and when subjected to pressure its loading density is 1.52 at 5000, 1.60 at 10000, 1.68 at 20000, 1.70 at 25000 and 1.72 at 30000 psi respectively. Other props of RDX are given below

Inhaling of RDX dust may cause intoxication characterized by fits simulating epilepsy with loss of counsciousness. The fits are sometimes preceded by sleeplessness & irritability, and are followed by coma lasting several hours often ending in vomiting & subsequent amnesia (Ref 98). Studies of the toxicity of RDX are also reported by Sunderman (Ref 47), von Oettingen et al (Ref 99), Sax (Ref 136) and others. Adherence to conventional health precautions & preventive measures should assure a healthful enviornment to plant workers and others who handle RDX. The health hazards in the manuf of RDX are considered to be no greater than in the manuf of other expls such as TNT, DNT, Tetryl & NG (Ref 47)
Historical RDX was first synthesized by Henning (GerP 104280 of 1899) for medical use. He failed to nitrate hexamine directly with strong HNO_3 but first prepd hexamine dinitrate, $C_6H_{12}N_4 \cdot 2HNO_3$, by treating an aq soln of hexamine with nitric acid. The resulting wh crysts were collected, dried and added in small portions to HNO_3 (d 1.52)

which was cooled to -5°. The mixt, after standing for 1/2 hr, was poured into ice water and RDX separated. The water-washed and dried product melted at 200°

Brunswig (GerP 299028 of 1916) prepd, examined & reported RDX to be stable to heat and not losing wt during 152 hrs heating at 110° (cited in Ref 9)

It was von Herz (Ref 2) who first recognized its value as an expl and succeeded in prepg RDX by direct nitration of hexamine, but the yields were poor. In his method powdered hexamine was added slowly to HNO_3 (d 1.52) at 20 - 30°. The soln was heated to 55° and allowed to stand, then cooled to 20° and diluted with a large amt of water. The pptd RDX melted at 200°. Because of the poor yield the manuf of RDX was expensive and not very attractive for large scale production

Hale (Ref 3) further studied the nitration and secured a yield of 68% by addg hexamethylenetetramine to 100% HNO_3 at a temp of 30° or less. The mixt was cooled to 0°, held for 20 minutes and drowned

No further substantial improvements were made in the manuf of Cyclonite for several years, following the work of Hale at Picatinny Arsenal, although many countries were working on the problem prior to WWII. The greatest improvements came during WWII due to research & development studies, made by several countries, in the manuf, props & uses of RDX in military expls

WWII Developments. **Canada & USA.** Meissner (Ref 20) by 1940 had improved the process of manufg Cyclonite in two ways: a) he made it continuous and b) he pptd the RDX from the spent acid, not by diluting with w to 15-20% strength, but by combination of dilution to 40-60% strength followed by introduction of this diluted acid into concd H_2SO_4. This procedure completely removed the RDX, leaving a spent acid which was stable in storage and which permitted simple recovery of the HNO_3. The process of direct nitration of hexamine is known as the "Nitrolysis Method"

In 1940 Drs Ross & Schiessler (Ref 76 & cited in Ref 77, p 37) of McGill Univ developed a process which did not require the use of hexamine to manuf RDX. In their method formaldehyde & AN are allowed to react in the presence of acetic anhydride:

$$3HCHO + 3NH_4NO_3 + 6(CH_3CO)_2O \longrightarrow (CH_2NNO_2)_3 + 12\,CH_3COOH$$

This reaction is nearly quantitative because it avoids the waste of formaldehyde inherent in the nitrolysis method and does not require such large amts of HNO_3. However, it does req large amts of acetic anhydride and the recovery of acetic acid produced in the reaction. The method of Ross & Schiessler (Ref 76) closely resembles that of the German "E-Process"

Dr Bachmann (Refs 23, 26, 33, 50 & 91 and cited in Ref 77, p 37) while investigating both the "Nitrolysis" and the Ross & Schiessler methods developed, in 1941 and later, a process based upon the following reaction:

$$C_6H_{12}N_4 + 4HNO_3 + 2NH_4NO_3 + 6(CH_3CO)_2O \rightarrow 2(CH_2NNO_2)_3 + 12CH_3COOH$$

Hexamine is nitrated to Cyclonite, and the 3 mols of formaldehyde liberated react with the AN in the presence of acet anhyd to form a second mole of Cyclonite. According to Bachmann, this reaction appears to be combination of the "nitrolysis" reaction and the procedure of Ross & Schiessler. However, this reaction is conducted under conditions which are different from both of the other reactions. Bachmann's process doubled the yield of RDX obtainable by the direct nitration of hexamine with HNO_3 alone. The resulting product, known as **Type B RDX,** contains as a constant impurity 8-12% HMX (see under Cyclotetramethylenetetranitramine)

As a group, many other researchers were working on new processes for prepg RDX both in the USA (Refs 24, 27,28,30,32,34,36,37, 38, 42 & 60) and in Canada (Refs 78,86,87,88,89,90,92 and others). The Bachmann process was applied industrially during WWII at one plant in Canada and later in the US by the Tennessee-Eastman Co (Holston Ordnance Works, Kingsport, Tenn). It not only made the manuf of RDX more economical but also led to the discovery of several new expl nitramines and to a more thorough knowledge of the chemistry of hexamine. The many by-products which may be obtd during the nitrolysis of hexamine, but all not necessarily present in crude RDX, include the following which have to be isolated:

1) **HMX** or Octogen, or Cyclotetramethylenetetranitramine

2) **DPT** or 1,5-endo-Methylene-3,7-dinitro-1,3,5,7 tetrazacycloöctane or Dinitropentamethylene tetramine

3) **BSX** or 1,7-Diacetoxy - 2,4,6-trinitro-2,4,6-

triazaheptane or 1,7-Diacetoxy-tetramethylene-2,4,6-trinitramine

4) Homo-DPT or 1,5-endo-Ethylene-3,7-dinitro-1,3,5,7-tetrazaclycloöctane

5) 1,5-endo-Ethylidine-3,7-dinitro-1,3,5,7-tetraza-cycloöctane

6) 1-Nitro-3,5-dicyclohexyl-1,3,5-triazacyclo-hexane

7) N, N'-Dimethylol-N-N'-dicyclohexyldiamino-ethane

8) 3,7-Dinitro-3,7-diaza-1,5-dioxacycloöctane

9) Dinitroxydimethylnitramide

10) Diacetoxydimethylnitramide

11) 1,3,5-Trinitroso-1,3,5-triazacyclohexane

Aristoff (Ref 90) considered the Bachmann reaction to be actually a combination of the direct nitrolysis of hexamine with HNO_3 concurrent with a resynthesis of hexamine with fragments produced by the nitrolysis

A lab method of prepg pure RDX, known as **Type A RDX** and not contg HMX is given by Brock-man et al (Ref 88). This method consists of oxidg the compd 1,3,5-trinitroso-1,3,5-triazacyclohexane obtd from hexamine, HCl & $NaNO_2$. The oxidn with H_2O_2 - HNO_3 yields as an intermediate 1-nitroso-3,5-dinitro-1,3,5-triazacyclohexane which is con-verted on further treatment with H_2O_2 - HNO_3 into Cyclonite having a mp of 205°

Other methods of prepg Cyclonite in the USA are described by Wyler (Ref 45) who patented a method of nitrating hexamine by HNO_3 in the presence of phosphorous pentoxide. The yield of Cyclonite was 90% of theoretical. Wyler (Ref 63) also patented a method of nitrating hexamine by HNO_3 in the presence of ammonium salts , such as NH_4NO_3 or $(NH_4)_2SO_4$. The yield of Cyclo-nite was 80% & 91% respectively using Amm salts vs 65% yield when no Amm salt was used. Caesar & Goldfrank (Ref 65) proposed a method of nitrat-ing hexamine using nitrogen pentoxide dissolved in inert, non-aqueous solvs, such as hydrocarbons or chlorinated hydrocarbons, according to the following equations:

$$C_6H_{12}N_4 + 3N_2O_5 \longrightarrow (CH_2.NNO_2)_3 + 3HCHO + 2\,N_2O_3$$

The authors claim yields of Cyclonite up to 89% of theoretical

Great Britain (Ref 83). After publication of von Herz's patent in 1920 (Ref 2), the Armament Res Dept at Woolwich began its investigation of RDX (British code name for ResearchDepartment Explo-sive or Royal Demolition Explosive). As a result of this research, it was found that the batch process was dangerous due to the formation of large amts of by-products which attack RDX to a certain ex-tent. Therefore, a process was developed in which hexamine was continously added to cooled 98% HNO_3. The resulting soln overflowed into a vessel wherein a controlled hot dilution with water took place. This allowed the RDX to crystallize and it also broke down the above-mentioned by-products, preventing their reaction with RDX. The residual weak acid, which was separated from the RDX by continuous filtration, was mixed with the weak nitric acid from the absorption towers and sent to a concentration plant

A small-scale pilot plant producing 75 lbs of RDX per day was installed in 1933 and operated until 1939. Another plant was installed in 1939 at Waltham Abbey. A full-scale plant was erected in 1941 near Bridgewater, with a capacity of 90 tons per week. This was increased later to 180 tons of RDX per week. During the period in which the Bridgewater plant was operating, investigative work to improve its efficiency was carried out con-tinuously. A detailed description of the Bridge-water plant process is given in Ref 3, pp 593-601. The Bridgewater plant operated throughout WWII

Germany (Refs 40, 45b, 54, 55, 56, 57, 62, 80 & 82). This expl called Hexogen in also known as *E-Salz, K-Salz, KA-Salz, SH-Salz or W-Salz* depend-ing upon its method of manuf. Although Hexogen was known in Germany since 1899 (Henning, Ger-man P 104280), it was not used until 1935 when its manuf was started using the W-Verfahren (W-Process) listed below. Four other methods of manufg Hexagen were later introduced. Product-ion reached its peak in June 1945 when almost 8 million pounds were manufd during the month. The KA-Verfahren proved to be the best process be-cause it was most economical, reqd less space & equipment, and used readily available materials

The German WWII methods of manufg Hexogen, in approx chronological order, are as follows:

W-Verfahren (W-Process). Developed in 1935 by Dr Wolfram of IG Farbenindustrie, this process is based upon the following reactions:

a) sulfur trioxide & ammonia to yield Amm amino-sulfonate & amm iminosulfonate

b) Product from a) is treated with a soln of $Ca(OH)_2$ to give sol Ca aminosulfonate & a ppt of $CaSO_4$

c) Liberated ammonia from b) is used in reaction a); Ca sulfate is removed by filtration, and the Ca aminosulfonate is treated with K_2SO_4

d) The resulting K aminosulfonate is separated & treated with formaldehyde at 30°
e) The condensation product, K methyleneaminosulfonate, is nitrated with mixed nitric sulfuric acid at 30°
This process gave yields up to 80% based on the formaldehyde used. It was used at the Krümmel plant of Dynamit AG until an expln in Jan 1943 completely destroyed the plant

E-Verfahren (E-Process). This process, developed during 1935-38 by Drs Erbele & Fischer of Dynamit AG, is based on the reaction of paraformaldehyde with AN, dissolved in acetic anhydride. The resulting Hexogen is separated, washed with water, stabilized & dried. The product has a mp of only 190-95° and is obtd in yields of 60-75% based on paraformaldehyde. It contains the same impurities, but in larger amts, as found in the KA-Verfahren (process) described below

SH-Verfahren (SH-Process). Dr Schnurr developed this process in 1937-38, based on the original method of Henning. It involves direct nitration (at -5°) of hexamethylenetetramine (also called hexamine & urotropin) with absolute HNO_3 (99%). The improvement introduced by Dr Schnurr consists in carefully controlling the heating of the contents of the nitrator after reaction is completed. The unstable products formed during the reaction are thereby partly decompd & partly nitrated to Hexogen. The purified product (yield 72%) has a mp of 200-202°. This process is considered to be more economical than the W-, E- & K- Processes, but inferior to the KA Process. It was used in at least 3 plants of the Dynamit AG: Döberitz with a capacity of 500 tons per month, Christianstadt with a capacity up to 300 tons per month, and Ueckermünde bei Stettin which produced 250 tons per month

K-Verfahren (K-Process). This process was developed by Dr Knöffler of Westfälisch-Anhaltische Sprengstoff Aktiengesellschaft somewhat later than the E-Verfahren (about 1938). It is based on the fact that the deficiency of two NH_2- groups in the hexamine molecule, ($C_6H_{12}N_4$), which are reqd for the production of each two moles of RDX can be remedied by introducing into the reaction two moles of AN. The reactants become, therefore, 1 mole of hexamine, 4 moles of 99% HNO_3 & 2 moles of AN to produce 2 moles of RDX:

$$C_6H_{12}N_4 + 4HNO_3 + 2NH_4NO_3 \longrightarrow 2(CH_2{\cdot}NNO_2)_3 + 6H_2O$$

One plant, Elsnig of WASAG, utilized the K-Process and produced 200 tons of Hexogen per month, even though the process was not economical

KA-Verfahren (KA-Process). This process, also developed by Dr Knöffler, is actually a combination of parts of the K- & E- Processes and was the most efficient developed by the Germans. It is based on the reactions:

$$C_6H_{12}N_4 + 2HNO_3 \longrightarrow C_6H_{12}N_4{\cdot}2HNO_3$$

$$C_6H_{12}N_4{\cdot}2HNO_3 + 2NH_4NO_3{\cdot}HNO_3 + 6(CH_3CO)_2O \longrightarrow 2(CH_2N{\cdot}NO_2)_3 + 12CH_3COOH$$

which involve treating hexamethylenetetramine dinitrate with acid AN in acetic anhydride. The resulting product is separated from acetic acid, washed with water & dried. It contains as impurities 1-2% HMX & a small amt of a cyclotrimethylene deriv. The advantage of the KA-Process over the E-Process is that by using hexamine dinitrate in lieu of formaldehyde only half the amt of water is produced, thus requiring one half the usual amt of acetic anhydride. Hence, it was possible without increasing the size or amt of equipment, to increase the production of the Bobingen DAG plant from 125 to 250 tons per month when the method was changed in 1944 from the E-Process to the KA-Process. Yields of the product, called KA-Salz in Germany, when calculated on the basis of formaldehyde from which hexamine is prepd, were 80-82% vs 73-75% yields in the E-Process. See also Stettbacher (Refs 9 & 84) and PATR **2510** (1958), pp Ger 88 & 90 (Hexogen or H)

Italy. According to Stettbacher (Ref 84, p 68) the manuf of T4 began in Italy before WWII. The process of manufg T4 as employed by "La Società Dinamita Nobel de Avigliana " is described by Pérez Pardo (Ref 22). In this process:
a) methanol was oxidized to formaldehyde and this was added to either ammonia or ammonium carbonate:

$$6HCHO + 4NH_3 \longrightarrow C_6H_{12}N_4 + 6H_2O$$

b) the resulting soln of hexamine was evapd and the crude hexamine purified by crystn from alc
c) to the concd soln of hexamine, dil nitric acid (d 1.4) in sl excess of that reqd by the equation:

$$C_6H_{12}N_4 + 2HNO_3 \longrightarrow C_6H_{12}N_4{\cdot}2HNO_3$$

was added at 15-20° slowly & with stirring. The resulting hexamine dinitrate was vacuum filtered, washed several times with water and finally with 90% alc, and then dried at 30-40°
d) the dried hexamine dinitrate was nitrated with 7 parts of concd HNO_3 (d1.51) below 15° and

raised to 30° for 1 hr when the reaction slowed down

e) the mixt was cooled to 0° and filtered, thus removing the bulk of the RDX. The mother liquor was heated in vacuo at a temp below 40° and most of the nitric acid was distilled & recovered. The concd mother liquor was cooled, which caused the pptn of some addnl RDX which was separated by filtration. The residual mother liquor was diluted with water in order to ppt the last traces of RDX. The remaining diluted acid was discarded.

f) all of the fractions were collected, washed with w, and dried. This method produced RDX having a mp between 200-202°

The props of Italian T4, of T4 desensitized by paraffin, and of Tritolo (TNT) are given by Belgrano (Ref 122)

Belgium. There is little information in regard to the manuf or extensive use of RDX in expl compns in Belgium

France. The lab prepn and knowledge of the props of RDX were known as early as 1931. Several refs to such studies appear in the French literature. The study of RDX in France was interrupted by the German occupation in 1940, but these studies were resumed after WWII. For example, studies of RDX & its phlegmatized mixts were reported by Desseigne (Ref 108), LeRoux (Refs 109 & 119), Tabouis et al (Ref 118) and others

RDX was also known and used alone and in composite expls in **Japan, Russia, Spain and Sweden.** It has been prepd & studied in **Switzerland** only on a lab basis for research purposes

Properties of RDX. As described above, there are two types of US technical grade RDX, depending on the method of manuf: *Type A,* mp 202-203° prepd by the nitric acid-process and *Type B,* mp 192-93° (decomp with sl evolution of fumes), prepd by the acetic anhydride, combination or Bachmann processes

There is considerable divergence in the solubility data for RDX taken from different sources in the literature. This is, undoubtedly, due to the different methods and conditions used in making the determinations, and the purity & physical state of the RDX tested. Therefore, it would be safer to assume that the values given in the table on p C 616 are only approximate

Other props of RDX, which are not expl props, include

Behavior towards other expls: a) does not blend with NC and does not gelatinize it (Ref 9, p 181) b) forms a plastic mass with NG which is as powerful as Stettbacher's Pentrinite (Ref 9, p 181) and c) dissolves in molten TNT as follows:

Temp, °C	gms RDX/100 g TNT
81	4.5
85	4.7
90	5.0
95	5.8
100	6.5
105	7.3
110	8.2

and the eutectic is 4.16% RDX at 79°

Chemical properties (Ref 19 and others). RDX is a neutral salt. It dissolves in concd H_2SO_4 with the evolution of gases. When boiled with dil H_2SO_4, it is hydrolyzed as follows (Ref 49): $(CH_2NNO_2)_3 + 6H_2O \longrightarrow 3HNO_3 + 3NH_3 + 3HCHO$. RDX dissolves in HNO_3 of concns up to 70% without decompg and may be recovered from soln by dilution with water. It is not attacked by either cold or hot concd HCl. It decomp completely by heating with an equal part of $Ca(OH)_2$ for 4 hrs at 60°. RDX decomposes slowly when heated at 60° with N/10 NaOH for 5 hrs, and rapidly when heated with N/1NaOH. The decompn products of RDX in alkaline solution which have been isolated are: N_2, NH_3, nitrates, nitrites, formaldehyde, hexamine and org acids (such as formic). When mixed with oxides of heavy metals (such as Fe or Cu), RDX forms unstable compds, which may undergo decompn and cause ignition of mixts at temps as low as 100°

Coefficient of cubical expansion - between 20 & 100°, 0.00025 cc/g/°C

Hardness - by Moh's scale, 2.5

Hygroscopicity - practically nil

Specific heat - cal/g/°C, at 20° 0.298; 40° 0.331; 60° 0.360; 88° 0.384; 100° 0.406; 120° 0.427; and 140° 0.446

Thermal conductivity - (cal/sec/cm/°C) 6.91×10^{-4} at d 1.263 and 6.98×10^{-4} at d 1.533

Volatility - practically nil

Explosive Properties of Cyclonite

Behavior towards flame. When ignited in the open, it burns rather slowly with a hissing sound & bright yel flame. No expln takes place when RDX is tested under these conditions unconfined

Brisance. 1) Kasts' method (calcd), B = 179.9 x 10^6 vs 86.1 x 10^6 for TNT, 107.5 x 10^6 for PA and 172.8 x 10^6 for PETN (Ref 64, p 283). Com-

Solubility of Cyclonite in Various Solvents
(Grams RDX/100g of solution)
(Refs 4, 8, 24a, 49, 64 and others)

Solvent	Temperature, °C						
	0	20	30	40	60	80	100
Acetic Acid (50%)	-	-	0.12	-	0.50	1.25	-
Acetic Acid (100%)	-	-	0.41	-	1.35	2.60	-
Acetic Anhydride	-	4.0	4.80	6.0	9.30	-	-
Acetone	4.2	6.8	8.40	10.3	15.3	(at 58°)	-
Acetonitrile	-	-	12.0	16.2	24.6	33.0	-
Alcohol, Ethyl	0.04	0.10	-	0.24	0.60	1.20	-
Benzene	-	0.05	0.06	0.09	0.20	0.40	-
Carbon Tetrachloride	-	0.0013	0.0022	0.0034	0.007	-	-
Chlorobenzene	0.20	0.33	0.44	0.56	-	-	-
Chloroform	-	0.015	-	-	-	-	-
Cyclohexanone	-	12.7 (at 25°)	-	-	-	-	25 (at 97°
Cyclopentanone	-	-	11.5 (at 28°)	-	-	-	37 (at 90°
Dimethylformamide	-	25.5	27.3	29.1	33.3	37.7	42.6
Ethanol	See Alcohol, Ethyl						
Ether, Dethyl	-	0.055	0.075	-	-	-	-
β- Ethoxylethyl Acetate	-	1.48	1.55	1.9	3.4	-	-
Isoamyl Alcohol	0.02	0.026	0.04	0.06	0.21	0.50	1.33
Methanol	0.14	0.235	0.325	0.48	1.06	-	-
Methyl Acetate	-	2.95	3.30	4.10	6.05 (at 52°)	-	-
Toluene	0.016	0.02	0.025	0.05	0.125	0.295	0.64
Trichloroethylene	-	0.20	0.21	0.22	0.23 (at 52°)	-	-
Trinitrotoluene (TNT)	-	-	-	-	-	4.4	7.0
Water	-	0.005	-	0.025 (at 50°)	-	-	0.28

parison with TNT as 100% gives 209% for RDX, 125% for PA and 200% for PETN, values considerably higher than those obtd experimentally
2) Crusher or Compression test (using Kast's apparatus) with 13x7 mm crusher & 35g of expl in the form of a cylinder having 21mm diam & 70mm height for d = 1.40, crushing was 5.30 mm vs 4.05 mm for TNT and 5.27 mm for PETN. Thus, for TNT = 100% RDX = 132% (Ref 16, p 96)
3) Iron plate dent test (using iron plate 10mm thick and a 35g chge in the shape of a cylinder, 21mm diam & 70mm long) gave 5.5 mm for RDX, 5.0mm for PETN and 4.0mm for TNT, which means RDX = 137% TNT
4) Plate dent test (Method A) for RDX pressed to d 1.50 gave a brisance value of 135% TNT (Ref 150)
5) Majrich & Šorm (Ref 14, p 338) give brisance values based on the lead plate cutting test & on other brisance tests. They compare these values with those calcd by Kast's formula. Their lead plate cutting test data showed sl higher brisance values for RDX than for PETN
6) Sand crushing test (using 200g bomb): 59.0 g of sand were crushed by RDX vs 48.0g for TNT and 62.9g for PETN. This gives for RDX 123% of TNT and for PETN 131% of TNT (Ref 150). Other values reported in the literature based on sand crushing tests range from 125 to 145% of TNT
7) Vivas, Feigenspan & Ladreda (Ref 64, p 286) give the results of a qualitative brisance test in which 100g sticks of expl were laid on a narrow steel plate 28mm thick and then exploded. RDX & PETN broke the plate in two pieces, while TNT produced only an impression

Energy, specific, $f = \frac{pvT}{273} = 14410$ vs 8080 for TNT and 13510 for PETN. Taking the value for TNT as 100%, RDX = 178% & PETN = 167% of TNT (Ref 64, p 285)

Explosion pressure (p). The value for RDX is 12600 kg/cm^2 vs 8080 for TNT, 8950 for PA, and 12700 for PETN (Ref 64, p 284). Taking the value for TNT as 100%, the equiv percentages are RDX 156, PA 111 & PETN 157% of TNT

Explosion temperature. RDX does not explode on heating when unconfined as do some other HE's. It either fumes off and ignites or deflagrates. Some results reported in the literature are as follows:
1) 255-260° in 5 secs, 316° in 1 sec and 405° in 0.1 sec (Ref 150)
2) 290° - fumes off w/o expln (Ref 49, p 625)
3) 280° in 5 secs for RDX Type B (mp 191°) (Ref 48)
4) Pérez Pardo (Ref 22, p 340) states that when RDX is heated rapidly to 215°, it explodes and if touched with an incandescent wire, it burns with a bright flame.

Flammability index, 278 vs TNT = 100 and Tetryl = 244 (Ref 150)

Fragmentation test 1) Tonegutti (Ref 16, p 98) gives two series of tests for RDX, TNT & PETN (51 g of expl at d 1.50 loaded into 40mm shells). His average values show that the number of fragments for RDX is sl lower than those for PETN with equal wts of fragments. Taking the value for TNT as 100%, the brisance by fragmentation test is RDX 141% and PETN 148%
2) Majrich & Šorm (Ref 14, p 298) give values for fragmentation tests conducted in steel pipes of 20 & 30 mm diam. When calculating brisance based only on the number of fragment produced, the value for RDX is about 200% TNT, which is higher than values obtd by other investigators
3) Stettbacher (Ref 84, p 118) gives fragmentation values for RDX & PETN pressed at 3000 atm into 20 mm shells, which indicate RDX to be much less brisant than PETN

Friction pendulum test. Steel shoe - RDX explodes; fibre shoe - unaffected (Ref 150)

Friction test in unglazed porcelain mortar. RDX in 0.03 g samples rubbed in a mortar produced partial deflagration, while PETN under the same conditions detonated completely (Ref 22, 341)

Gases formed on explosion. It was formerly assumed (Avogadro) that RDX decompd on expln as follows:

$$(CH_2.NNO_2)_3 \longrightarrow 3CO + 3H_2O + 3N_2,$$

but Kast (cited in Ref 64, 283 and in Ref 16, 93) gives the following equation, based on the actual analysis of the gases produced:

$$(CH_2.NNO_2)_3 \longrightarrow 2CO + CO_2 + 2H_2O + H_2 + 3N_2$$

(See also Ref 149)

Heat of combustion: 2285 cal/g or 507.3 kcal/mol at const vol, H_2O liq (Ref 11, p 262) and 2281 cal/g or 506.4 kcal/mol under the same conditions (Ref 19)

Heat of explosion. Tonegutti (Ref 16, p 95) reports 1370 cal/g at const vol with w liq as compared with 875 cal/g for TNT and 1446 cal/g for PETN. Earlier values reported were 1390 cal/g by L. Avogadro (Refs 3a, 5 & 6) and 1370 cal/g

by H. Kast at const vol, with water in the vapor phase (cited in Ref 16)

Heat of formation: Minus 96 cal/g or minus 21.3 kcal/mol (Ref 11)

Impact sensitivity test (fall of 2 kg weight);

1) Picatinny Arsenal app - 8 to 9 inches (18 mg sample) vs 14" for TNT, 7 to 8" for Tetryl and 5-7" for PETN

2) Bureau of Mines app - values obtd at PA range from 32 to 42 cm for 20 mg samples from various sources. A value of 34 to 40 cm was obtd for RDX Type B (mp 191-92°) vs 100 cm for TNT (Ref 48)

3) Pérez Ara (Ref 49) gives 9" for RDX

4) European impact machines. Guia (Ref 142, p 229) and Vivas, Feigenspan & Ladreda (Ref 64, p 283) both give 42 cm for RDX vs 38 cm for PETN and >60cm for PA & TNT. Stettbacher (Ref 9, p 182) gives 29 cm for RDX vs 27 for PETN & 30 cm for Tetryl

Summarizing the above impact sensitivity results one can conclude that RDX is sl more sensitive than Tetryl and sl less sensitive than PETN

5) Effect of temp on impact sensitivity (PA apparatus): 9" at 20°, 8" at 30° and 5" at 100°

6) Influence of particle size on sensitivity. The height of fall increases (sensitivity decreases) with decreasing particle size

Initiation sensitivity. For the initiation of 0.4 g of base chge of RDX, at least 0.15g of 80/20 MF/ $KclO_3$ mixt, or 0.19g MF alone, or 0.05g LA is reqd

Power 1) by Ballistic Mortar test, 150-161% of TNT

2) by Trauzl test (Lead Block expansion test), 157-168% of TNT (Ref 150); Tonegutti (Ref 16, p 95) gives 475-490 cc expansion for RDX, 290cc for TNT and 515-520 cc for PETN, which is equivalent to 166% of TNT for RDX and 178% of TNT for PETN; Vivas, Feigenspan & Ladreda (Ref 64, p 283) give 520 cc for RDX, 310cc for TNT, 540 cc for PETN and 350 cc for PA; or taking the value for TNT as 100%, RDX is 168% of TNT, PETN 174% of TNT and PA is 113% of TNT. These same authors report that when RDX is used against earthworks, masonry, or in underwater explns, its power is practically double that of TNT. Stettbacher (Ref 9, p 182) states that the value of 520 cc for RDX reported by some investigators is too high, on the other hand, the values of 450 cc reported by Izzo (Ref 5, p 379) and of 470 cc reported by Naoúm (Ref cited by Stettbacher) are too low. Majrich & Šorm (Ref 14, p 299) give the following Trauzl values, calcd per gram of expl: 32.1cc for RDX, 35.2cc for PETN 29.6 for Tetryl, and 39.0 cc for Hexanitromannitol

3) by other tests. Vivas et al (Ref 64, p 286) describe a test in which shells charged with 25 kg of expl were exploded against consolidated (packed) earth. The shell charged with RDX produced a crater 1.27 meters deep, 4.20 m in diam, and 12.60 cubic meters in volume vs 1.25, 4.00, and 6.75 values for TNT. Taking the volume of 6.75 cubic meters as 100% power for TNT, then RDX is 186.5% of TNT

Rifle bullet impact sensitivity. Picatinny Arsenal data (Ref 150) shows 100% explosions (total or partial) in 5 trials. According to Vivas et al (Ref 64, p 286) and Pérez Pardo (Ref 22, p 341), RDX does not actually explode from the impact of bullets, but rather partially deflagrates, while PETN under the same conditions detonates completely

Sensitivity classification (for quantity-distance storage). Class 7, formerly Class 9 (Ref: AMC Safety Manual AMCR 385-224, change 12, p 17-63, 9 Nov 1964)

Sensitivity to initiation by electrostatic discharge. Minimum equiv voltages at 0.0003 mfd: local ignition 14950 (RDX pulverized or crystalline), no ignition at 12300 for RDX either pulverized or crystalline

Stability tests. 1) 75° International heat test, 0.03% loss in wt in 48 hrs (Ref 150)

2) 82.2° KI test, 60 minutes (PicArsn)

3) 100° Heat test, 0.04% loss 1st 48 hrs, 0% 2nd 48 hrs, and no expln in 100 hrs (PicArsn)

4) 132° Heat test, no change occurred during 8 hrs of heating while PETN produced nitrous fumes (Ref 22, p 341)

5) 135° Thermal stability test, not acid and no explosion in 300 min (PicArsn)

6) Vacuum stability tests: at 120° 0.9 cc gas evolved/5g sample in 40 hrs; at 150° 2.5 cc gas evolved/5g sample in 40 hrs;and at 150° 3.95 cc gas evolved/ 5g sample of RDX Type B (mp 192°) in 40 hrs (PicArsn)

7) According to Vivas et al (Ref 64), stability tests by Taliani, Metz and German 135° heat tests all show that RDX is more stable than Tetryl, but less stable than TNT

Storage of Cyclonite. Wet with water in the USA and Great Britain; dry in Germany & Japan

Surveillance test. No decompn of RDX occurred during storage for 1 year at temps of either 65°

or 85° (PicArsn). When heated at 100° for two months, it did not loss weight and did not decompose (Ref 64, p 287)

Temperature developed on explosion. 3380° for RDX vs 2800° for TNT, 4050° for PETN, and 3230° for PA (Ref 64, p 284). See also Ref 149

Velocity of detonation. 1) The general formula for pressed, unconfined charge of 1.0 diam:

VD (meters/sec) = 2219 + 3613 d

where d = density of chge in g/cc

2) Kistiakowsky gives 5830 at d 1.0 and 8000 at d 1.60 g/cc

3) Kast (cited in Ref 16, p 96) reported 8370 meters/sec at d 1.70 using a cylindrical chge 13.6 mm in diam and 75mm long. He also reported 8360 for d 1.67

4) Tonegutti (Ref 16, p 97) reports 7890 for d 1.56 and chge diam 25 mm. The same investigator (Ref 21) gives 8210 to 8225 meters/sec at d 1.60

5) Evans (Ref 103) reports 8250 m/sec at d 1.60

6) Vivas et al (Ref 64, p 285) give 8380 m/sec at d 1.70 vs 8400 for PETN at d 1.70 and 6700 for TNT at d 1.60

7) According to Pérez Ara (Ref 49, p 625), the max velocity of deton value for RDX is 8500 m/sec

Volume of gases developed on explosion: 908 liters/kg at 0° & 760 mm vs 690 for TNT and 828 for PETN under the same conditions (Ref 64, p 285) (See also Ref 149)

Uses of RDX. The extreme high mp, with accompanying decompn, makes the use of Cyclonite by itself unsuitable for cast loading in projectiles, but it is used in press loading operations. Since Cyclonite is more sensitive to friction than some other std HE's (such as Tetryl, TNT, PA & Explosive D) and only slightly less sensitive than PETN, it is not generally suitable for use as a bursting chge in shells unless it is desensitized (phlegmatized)

In order to use RDX for cast loading of projectiles, it must be blended with a relatively low-melting substance, preferably an expl which functions as a liquid matrix at a suitable casting temp. If Cyclonite is blended with a weak-expl compd (such as mononitronaphalene) in which 20% of the RDX is replaced, its power is reduced considerably and approaches that of straight TNT. It is much better to use mixts of RDX & TNT, in which 70% of RDX is blended with 30% of TNT as in some German expls or in British & American Cyclotols. The following are examples of applications for RDX in expls used in various countries:

USA & Great Britain. Straight (compressed) RDX is used as a base chge in Detonators and in some Blasting Caps (See Vol 2, p B185ff). Compositions of RDX in which it is coated with wax (See Composition A) or a non-expl plasticizer (See Composition C) and in admixtures with TNT (See Composition B) are used for filling shells & bombs. Some ternary mixts contg RDX & AN (such as DBX: AN 21, RDX 21, TNT 40 & Al 18%) are also used in shells & bombs

Germany Straight (compressed) was used in Detonators and straight or sl desensitized (by 3 to 5% wax) in boosters, sub-boosters, and as a bursting chge in rifle grenades, small caliber shells & in small shaped charges. It was used with 3% wax to replace PETN/wax mixts in sub-boosters, grenades & shaped charges. RDX with larger amts of wax (10%) was used as a press-loaded bursting chge in 75 mm shells. For cast-loading mixts, the Germans used compn similar to US Cyclotols & Composition B. For demolition purposes, plastic expls similar to US Composition C were used. For example *Plastit* developed during WWII contained RDX 64, Collodion Cotton 3.5 & nitrohydrocarbons 32.5%. This expl was less efficient than US Composition C2 because it contd less RDX

Italy. RDX was used by itself but graphited (1%) in Detonators & Exploders. RDX slightly desensitized by wax (5%) was used as a sub-booster for all types of ammunition, as a booster for shells, and as a bursting charge for Land Mines. RDX with larger amts of wax was used as a press-loaded bursting chge in shells; such compns were similar to those used by the Germans. One compn, called *Duxita,* contd 3% castor oil as a desensitizer for RDX. Cast-loaded compns for shells were similar to US Cyclotols & Composition B. These were called *Tritolita* and when Al was added *Tritolital.* Another Italian mixt consisting of AN 73.4%, T4 22% & wax 4.6% was used during WWII as a bursting chge for armor-piercing, long nose shells. For demolition purposes, plastic expls similar to German Plastit were used

Japan. RDX was used by itself in boosters for small arms ammunition and in sub-boosters for Bomb fuzes. They also used RDX highly pressed as bursting charges for 20 & 37mm shells, and in Army 7.7mm & Caliber 0.50 Bullets, and in some Land Mines. RDX desensitized by large amts of wax (beeswax 5 to 15%) was used as a bursting chge in 20mm & 75mm Shells. In some shells, Al was added as a separate ingredient for its incendiary effect. For cast-loading of shells, com-

positions similar to US Cyclotols were used. Plastic expls, such as RDX 80-85 & oil 20-15%, were used for demolition purposes

Russia. RDX by itself was used in some Detonators, and in Detonating Fuses & Boosters. RDX phlegmatized with wax was used for filling some small caliber projectiles. A more extensive use or RDX was made in admixtures with other expls. For example, a mixt of RDX/TNT/Tetryl (71.4/16.4/11.7%) was used for filling 76.2 mm Armor-Piercing HE Shells. A mixt of RDX/TNT (50/50, 40/60 & 20/80%) was used in Shaped Charges. A mixt of RDX/Al/Binder (75/21/4) was used as a bursting chge in 25mm HE Shells, in 45mm Armor Piercing HE Tracer Shells, and in 85 mm Antiaircraft Shells

Specification Requirements for Cyclonite

Argentina. Dr M.M. Kostevitch of Argentina, in a private communication, stated the following were the requirements of his laboratory for military grade Cyclonite:

d 1.80-1.82g/cc, mp 203.5 to 205°, ignition temp 230°, moisture (max) 0.01%, ash 0.0, AN 0.0, dinitroso products 0.010, acidity or alkalinity 0.0%, stability - no decompn after heating for 4 days at 100°

Germany (Ref 55). The Hexogen supplied to the Armed Forces had to be crystalline, dry, colorless & screened. It should not contain any intentionally added admixtures or any hard alien matter such as metal, glass or sand. Hexogen intended for use in the prepn of ignition charges & percussion caps had to be purified by recrystallization (for example, from nitrobenzene). Hexogen intended for phlegmatization and used in boosters & bursting charges could be supplied in a water-wet condition

Melting point. a) for W-, K- and SH-Salz above 200° b) for E-Salz above 190°

Granulation. RDX for boosters or bursting charges had to pass thru a 0.75mm opening sieve (DIN #8) and RDX for ignition charges thru a 0.600mm sieve (DIN #10 ⇒ US Std Sieve #30)

Loss of Weight. Should not exceed 0.1% after heating a 10g sample for 5 hrs at 100°

Chlorine. When tested as prescribed, chlorine shall be absent

Ammonia content. When tested as prescribed, the sample shall produce a pH below 7.5

Formaldehyde content. When tested as prescribed, the sample shall not give more than a trace

Acidity. When tested as prescribed, the acid in a 10g sample shall not consume more than:

3.0cc of N/20 NaOH for E-Salz (Tech pure)
2.0 cc of N/20 NaOH for SH- & K-Salz (Tech pure)
0.3cc of N/20 NaOH for W- & K-Salz (recrytd)
0.3 cc of N/20 NaOH for any recrytd salt intended for use in ignition charges or percussion caps

Sulfate content. None

Acetone insoluble residue. Maximum 0.1%. The residue should be free of coarse particles

120° KI test. When tested as prescribed, the KI-starch paper should not show any discoloration after 10 min and but a sl one after 20 min

Packing. The dry Hexogen is to be contained in special paper bags of 12.5kg capacity. Each bag is to be packed in a cardboard container, provided with a cover, the seams to be sealed with a wide adhesive tape. Four of these filled cardboard containers are placed on a wide strip of cardboard about 100cm long & 0.5mm thick and the ensemble packed into a special transport box. The ends of the cardboard strip can later be grasped when lifting the cardboard containers from the transport box. The boxes, each contg 50kg of RDX, are sealed and then shipped

USA. The requirements of RDX used by the Armed Forces are set forth in US Specification MIL-R-398C(1) dated 30 October 1963. This specification covers two types of RDX and 8 classes based on uses & advisory granulations

Type A - Made by the nitric acid process

Type B - made by the acetic anhydride process

Properties	Type A	Type B
Melting point, °C (min)	200.0	190.0
Acetone insol (max)	0.05	0.05
Inorganic insol (max)	0.03	0.03
Insoluble particles retained on US Std Sieve No 60, particles (max)	5	5
Acidity, % (max)	0.05 as HNO_3	0.02 as CH_3COOH

Classes. The suggested uses of RDX based on Class and granulation are as follows:

Class A, for use in Composition A3, Composition B, Composition C4, and Cyclotol

Class B, for use in Composition A3 and Composition C4

Class C, for use in the prepn of RDX booster & detonator pellets

Class D, for use in Cyclotols

Class E, for use in Composition C4 and Plastic

Bonded Explosives (PBX's)
Class F, for use in Composition B3
Class G, for use in PBX's
Class H, for use in Composition C4
Granulation requirements of RDX

Thru US Std Sieve No	Class (% of material passing Sieve) A	B	C	D	E	F	G	H
8	-	-	-	100	-	-	-	-
12	-	-	99*	-	-	-	-	-
20	98	-	-	-	-	-	-	-
35	-	99	-	20	-	-	-	100
50	90	95	40	-	-	-	98	98*
60	-	-	-	-	-	99	-	-
80	-	-	-	-	-	97	-	-
100	60	65	20	-	-	-	90	90*
120	-	-	-	-	-	83	-	-
170	-	-	-	-	-	65	-	-
200	25	33	10	-	-	-	46	70
230	-	-	-	-	-	36	-	-
325	-	-	-	-	97*	22	-	50

*Minimum Percent

Packing of US RDX. Crystalline RDX shall be thoroughly mixed to form a slurry or cake contg not less than 10% by wt of a soln made as follows: 40% by wt of isopropyl alcohol & 60% by wt of water. The RDX shall be packed in bags made of double filled, gray cotton duck large enough to hold a max of 50 lbs (dry weight). Not more than 6 of these bags shall be placed in a rubberized bag or other suitable water-tight material. The large bags, contg a max of 300 lbs dry wt of RDX, shall be placed in a keg or drum complying with Interstate Commerce Commission Specifications

Disposal of Waste Cyclonite (Refs 58 & 131, pp 315-18). If it is inconvenient to destroy RDX by burning or by detonation in a pit, the following procedure for decomposing it may be used:
a) Transfer to an iron container 300 gals of a 5% NaOH soln for each 100 lbs of RDX to be destroyed
b) Heat the soln to boiling (by steam coil or by injection of steam) and add the RDX in small portions while maintaining the soln at the boiling point
c) Continue the boiling for ½ hr after adding all the RDX and then allow the soln to cool & settle. Discharge the supernatant liq into a sump
d) Clean the container with NaOH soln and then with water, discharging all into a sump

Recovery of Cyclonite
Crater (Ref 106) patented a method whereby RDX, in cryst form, may be recovered from expl compns contg it & TNT, NC or Tetryl by use of a solv, (such as acet or chlf) to dissolve the chge, except theRDX
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Cyclotrimethylenetrinitramine (RDX), Analytical Procedures. Qualitative Tests:

a) Diphenylamine (DPhA) Test

This test differentiates RDX from HMX, but nitrates and nitroso compds interfere. However, the absence of nitrates and nitroso compds can be shown by the failure to obtain a blue color with a 0.2% soln of DPhA in 85% phosphoric acid (Ref 2, pp 178-79)

To test for RDX, a few crystals of the material for test are placed in the indenture of a white spot plate and 1 drop of test solution (1mg of DPhA in 10ml of 85% sulfuric acid) is added. A strong blue color in 1 minute indicates RDX. A blue color is obtd with HMX only after 5 mins or longer has elapsed (Ref 1)

b) Hydrolytic Decomposition Test

This test is based on the formation of AN on hydrolysis with a mineral acid. HMX and other nitramines, such as Tetryl and ammonium salts interfere. However, Tetryl may be removed before the test with benzene (solubility of RDX is only 0.05%) and ammonium salts with water

A drop of an acetonic solution of RDX or a small amt of the solid is evaporated with one drop of concd HCl in a micro test tube. After cooling, a drop of Nessler soln is added. A positive test is indicated by the appearance of a brown or yellow ppt or turbidity (Ref 2, p 489)

c) Sodium Nitroferric Cyanide (SNFC) Test. (Sodium Nitroprusside) (RDX differentiation from HMX)

Place 3-5 mg of the material to be tested in the indenture of a white spot plate. Add 5 drops of either acetone or dimethylformade (DMeFA). Stir to dissolve the sample. Add 3 drops of a freshly prepd soln of SNFC (40mg to 100ml of water). Add 2 drops of a 1% soln of NaOH. If acetone is used as the solvent, a pink-red color is obtd which gives place to a blue-green color within 2 mins and if DMeFA is used, a blue color is obtd immediately. Nitro compounds such as TNT, DNT, PA, etc interfere and so do the nitramines, such as Tetryl. However these compds can be removed before testing by benzene, because RDX is practically insol (soly is only 0.05%). Organic nitrates such as PETN do not interfere. Interfering common secondary expls can be separated by thin-layer Chromatography (Ref 4)

d) Determination of Purity.

The purity of RDX may be detd by the Chromous Chloride Method of Jamison (Ref 3). This method consists in the reduction of RDX in an acetic-HCl soln with chromous chloride and back-titrating excess of chromous chloride with ferric alum using phenosafranime as an indicator. The method is reported to give a recovery of 99.82± 0.30% The procedure is as follows:

Dissolve approx 0.1g of RDX, in a 500 ml titration flask, with a mixt of 25 ml of glac AcOH and 25 ml of concd HCl. Stir the contents of the flask for at least 10 mins using a magnetic stirrer while the air is being replaced with CO_2. Maintain a CO_2 atm throughout the determination. Add exactly 100 ml of 0.2N chromous chloride soln and stir for another 15 mins. Add 15 drops of phenosafranine indicator and titrate with 0.15N ferric alum soln. The end-point is a sharp color change from green to blood-red. Determine a blank and caluclate as follows:

$$\% \text{ RDX} = [1.234\ N\ (V_1 - V_2)]\ /W$$

where

V_1 = ml of Fe^{+++} alum soln used for blank

V_2 = ml of Fe^{+++} alum soln used to titrate the excess chromous chloride

N = normality of Fe^{+++} alum soln

W = wt of sample in grams

1.234 = the factor obtd by dividing the mw of RDX (222) by the total electron change of 3 nitro groups undergoing reduction (6 x 3 = 18). See also COLOR REACTIONS AND COLOR REAGENTS in this Volume, p C405-L

Quantitative Determination of RDX in the Presence of HMX:

There existed up to June 1965, twenty-seven methods for the determination of RDX & HMX (Ref 6). One of the methods recommended for consideration is the nonaqueous titration method given in Ref 5. By this method RDX can be detd in the presence of as much as 80% HMX

Procedure: Approx 60 mg of the mixt is dissolved in 40 ml of a methyl isobutyl ketone-isopropanol mixt (4:1) and titrated with sodium methoxide added from a microburet. After each addition of the titrant, the soln is thoroughly mixed by a magnetic stirrer for 1 min and the potentials recorded after allowing the soln to settle for another min. The titration is conducted in an atm of nitrogen. An average recovery of 99.75% with a standard deviation of 0.38 is to to be expected. See also Analytical Procedures under HMX in this Volume

Note: This section was written by E.F.Reese of PicArsn

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CYCLOTRIMETHYLENETRINITRAMINE (RDX OR CYCLONITE) BINARY, TERNARY AND OTHER EXPLOSIVE MIXTURES

In order to safely use Cyclonite for loading projectiles, the expl has to be desensitized to such an extent that its impact test value would be comparable to that of either PA or TNT. One of the ways to achieve desensitization of RDX is to coat the particles with wax or a less sensitive expl such as TNT. A major development during WWII was the development of binary, ternary and other expl mixts using the sensitive & very powerful explosive RDX. Some of the compositions developed for military and industrial applications include:

USA.

Composition A Explosives (See this Vol)
Composition B Explosives (See this Vol)
Composition C Explosives (See this Vol)
Cyclotols (See this Vol)
DBX (Depth Bomb Explosive, AN/RDX/TNT/Al 21/21/40/18)
Dentex (RDX/DNT/Al 48/34/18)
H-6 (RDX/TNT/Al/D-2 wax/$CaCl_2$ 45/30/20/-5/0.5 added)
HBX-1 (RDX/TNT/Al/D-2 wax/$CaCl_2$ 40/38/17/5/0.5 added)
HBX-3 (RDX/TNT/Al/D-2 wax/$CaCl_2$ 31/29/35/5/0.5 added)
PTX-1 (RDX/Tetryl/TNT 30/50/20)
PTX-2 (RDX/PETN/TNT 42.5/27/30.5)
RIPE (RDX/Gulf Crown E Oil 85/15)
Torpex (RDX/TNT/Al 42/40/18)

Many foreign countries have also developed explosive compositions based on RDX

France:Le Roux (Ref 17) prepd & studied the following mixts:

99.5/0.5%	RDX/graphite
95/5	RDX/paraffin
95/5	RDX/mononitronaphthale

The latter mixt decompd at 180°. Le Roux (Ref 18) continued the study of phlegmatized RDX and developed in 1949 a mixt:

RDX/Al/mononitronaphalene 80/15/5

which had a bulk density of 1.05g/cc and a pressed density of 1.70g/cc at 2100 kg/sqcm. Its CUP or modified Trauzl test value is 156% of PA, sensitivity to shock with a 2kg weight 200cm is 44% explns, and velocity of detonation 7990 meters/sec

The French pressed expl consisting of RDX/-beeswax (or paraffin)/0.5-2.0% graphite is similar to US Composition A. It is called in French Hexogène/cire d'abeille. The castable composition consisting of RDX 59.5, TNT 39.5 & beeswax 1% is similar to US Composition B. It is called in French *Hexolite* or *HT* (Hexogène-Tolite)

Since WWII a number of plastic expls using RDX have been developed and/or patented in France. Some examples of these are the following:
Médard (Ref 15) developed a plastic expl contg no NG. It was prepd by mixing RDX (or PETN) 45, $NaClO_3$ 31, & plasticizer (0.5-1kg polyvinyl acetate in 1 liter of liq DNT) 24%. This compn is fairly water resistant and suitable for use in mines
Sevranite No 1 (Ref 16). RDX (or PETN) 48, NH_4ClO_4 31, powd Al 3 & plasticizer (1 p polyvinyl chloride in 17 parts liq DNT) 18%. This compn at a pressed density of 1.70 g/cc has a velocity of deton of 7000 meters/sec. It can be kept under water for several weeks without any effect on its expl power. The CUP value is 142% of PA
Explosif plastique PE 1. Consists of RDX/mineral jelly/ liq paraffin/rubber. This compn is similar to US Composition C4
Explosif plastique PE 3A. Consists of RDX/Shell oil/liq paraffin/lecithin/carbon black
Explosif plastique TG. Consists of RDX 78.5, Collodion Cotton 17.5 & agglomerant 4%
Explosif plastique TN. Consists of RDX 88%, plasticizer 11.9 & lampblack 0.1%
The specific uses of French plastic expls are

not reported

Germany: *C6*. A mixt developed during WWII as a substitute for TNT. It consisted of Methylamine nitrate 50%, $NaNO_3$ 35 & RDX 15%. This mixt was used as a castable bursting chge for shells & bombs

Füllpulver Nr 17a. Consists of a matrix of 54/32/14 Dinitroanisole/AN/RDX and a biscuit of 46/21/20/9/4 AN/$Ca(No_3)_2$/RDX/PETN/hydrated water

Füllpulver Nr 18. Consists of TNT 80, RDX 19 & Montan wax 1%. It was used as a bursting chge in shells

Füllpulver Nr 45. Consists of 50/50 PETN/RDX 70 & wax 30%. It was used as a bursting chge in special projectiles. Another mixt by the same name consisted of RDX 50, PETN 35 & wax 15%

Füllpulver Nr 52. A yel castable compn consisting of DNB 50, AN 35 & RDX 15%. It was used in 50 kg GP Bombs & in SAP Bombs

Füllpulver Nr 52a A pressed expl compn consisting of AN 55, $Ca(NO_3)_2$ 30 & RDX 15%. It was used as a biscuit filling in the nose of parachute & robot Bombs, with a surround of Füllpulver Nr 52

Füllpulver Nr 86 Consists of a mixt of Ethylenediamine dinitrate 46, RDX 18 & wax 36%. It was pressed in blocks, wrapped in wax paper & placed in Al containers

Füllpulver Nr 91-H5. A pressed mixt of RDX 95 & Montan wax 5% was used in boosters & sub-boosters

Füllpulver Nr 92-HID. A pressed mixt of RDX 90 & Montan wax 10% was used in boosters

Füllpulver Nr 95. A pressed mixt of RDX 60 & TNT 40% was used as a bursting chge in shells. This is the same composition as US 60/40 Cyclotol which is cast

Füllpulver Nr 105. A compn consisting of TNT 70, RDX 15 & Al 15%. It was cast loaded into GP Bombs & Torpedoes

Füllpulver Nr 106. A cast mixt consisting of TNT 50, RDX 25 & Al 25% was used as a bursting chge in bombs

Füllpulver Nr 107. A castable compn consisting of TNT 50, RDX 20 & Al 30% was used in underwater ammunition

Füllpulver Nr 108. A castable compn consisting of TNT 60, RDX 20 & Al 20% was used as the bursting chge for underwater ammunition

Füllpulver Nr 109. A pressed mixt of RDX 70, Al 25 & Montan wax 5%. It was used as a biscuit filling, with a Nitroguanidine nose chge, and with a surround of Füllpulver Nr 106, in the 500 kg GP & 1800kg Armor-Piercing Bombs and in Pilotless Aircraft Missiles. This filler is more sensitive than RDX alone, and cannot be used w/o the protective nose chge of Nitroguanidine

Two German plastic expls developed during WWII are the following:

Hexoplast 75. Consists of RDX 75, NC 1.3, liq DNT 20 & TNT 3.7%. It was developed to replace the RDX/US vaseline plastic mixt when US vaseline was no longer available in Germany

Plastit. Consists of RDX 64, NC 3.5 & liquid or semi-liquid nitrohydrocarbons 32.5%. This expl is less efficient than US Composition C2 because it contains less RDX

See also PATR **2510** (1958), pp Ger 23, 47, 90 & 134)

Italy : Among the proposed military expl mixts of interest, those listed in Refs 11, 12, 13 & 14 should be noted:

Idrolita. A high expl consisting of AN 70, RDX 20, paraffin 3 & water 7%. This expl is claimed to stable & insensitive to shock

Incendiare. RDX (PETN or TNT) 15-60, oxidizer (chlorate, nitrate, oxide, perchlorate or peroxide) 20-40 & finely pulverized metal (Al, Mg, Zn or Fe) 20-40% (Ref 10a)

T4 Plastico: RDX 78.5, DEGN (+ 0.3 - 0.4 NC) 17.5 & vaseline 4%. Another mixt consists of RDX 89 & petrolatum 11%. Its d = 1.58g/cc and velocity of detonation = 8000 meters/sec. This expl was used in Land Mines & in Antitank Bombs

Tritolital. TNT 60, RDX 20 & Al 20%. It is similar to US Torpex

Tritolite. Mixt of T4 (RDX) & TNT (Tritolo) was proposed in 1930 for use in underwater weapons. Some of the mixts used extensively during WWII in shaped-charge projectiles & in land mines are:

a) 50/50 RDX/TNT (Cyclotol)
b) 60/38/2 RDX/TNT/wax (dyed red) (Composition B)
c) 25/50/25 RDX/TNT/Al (Trialen 105)
d) 20/40/20 RDX/TNT/Al (Torpex)

Two Italian unknown name expls which were press-loaded bursting charges are:

a) AN 73.4, RDX 22 & wax 4.6%. It was used as a bursting chge for 47 mm AP Shells

b) RDX 95 & wax (dyed red) 5%. It was used as a booster chge in ammunition

Japan: Several compns known as **Angayaku** consist of:

a) AN 75 & RDX 25% press-loaded filler for Bombs

b) AN 78 & RDX 22% press-loaded filler for

Bombs

c) AN 84 & RDX 16%,press-loaded filler for Bombs

d) AN 51, RDX 15 & Guanidine Nitrate 34%, cast-loaded filler for shells & Bombs

e) AN 48, RDX 20 & Guanidine Nitrate 32%, cast-loaded filler for shells & Bombs

f) RDX 85 & wax 15%,pressed-loaded filler for AP Shells

g) RDX 42, PETN 50 & wax 8%,press-loaded filler for machine-gun Bullets

h) AN 43.2, Guanidine Nitrate 28.8, RDX 8 & Al 20%,cast-loaded filler used in underwater ammunition

Chauyaku (Cyclotol). A castable mixt of RDX 50 & TNT 50% used in various artillery projectiles

Haishokuyaku. A dark-gray pressed expl compn No 1: Amm Perchlorate 77, RDX 17, silicon carbide 1.5 & paraffin 4.5%, used in std Demolition Blocks; No 2: Amm Perchlorate 48, Guanidine Nitrate 20, RDX 25, graphite 2 & paraffin 5%, used as a coml mining expl

Koshitsu A brown, putty-like plastic expl, consisting of RDX 80-85 & oil 20-25%; was used in shaped Demolition Charges

Nigotanoyaku Mk 2. Castable mixts of RDX & TNT in various proportions (60/40 - 30/70),used in Bombs, Projectiles, Mines & Demolition Blocks

Tanōyaku: No 1:RDX 60, TNT 30 & Tetryl 10%; No 2: RDX 55, TNT 38 & Tetryl 7. These are cast loaded chges for various Projectiles

Type 94M. A cream-yellow expl compn consisting of Trinitroanisole 60 & RDX 40%. It was used in shaped charge Grenades and as a Booster surround.

Several pressed expl mixts of unknown name are:

a) PETN 58 & RDX 42% used as a filler for Projectiles

b) RDX 83 & PETN 17% used as a filler for Projectiles

c) RDX 92-95 & wax 8-5% used in Antiaircraft Shells

d) RDX 85 & wax 15% used in AP Projectiles

e) RDX & Al powder used as incendiary chge

Russia: *A-IX-2.* An expl mixt of RDX 73, Al powder 23 & wax 4%, was used as a bursting chge in Projectiles

Flegmatizirovannyi Gheksogben (Phlegmatized RDX). RDX was mixed with a small quantity of wax, paraffin or ceresin and pressed to a d = 1.65g/cc for use as a filler in Shells

Skal'nyi Ammonit. Press-loaded industrial expls used for rock blasting: No 1: RDX/Al powder/Ca stearate; No 2: RDX coated with AN

Tetritol-Gheksogben Brizantnoye VV. (PTX-1) A press-loaded (or cast-loaded) ternary expl mixt consisting of RDX 71.9, TNT 16.4 & Tetryl 11.7% It was used in AP HE Shells. See also PATR **2145** (1955)

Spain: *Exógeno plastico or PE* (Composition C). A yellow colored plastic expl prepd by coating granular RDX with 12% vaseline & other oily substances. It was used w/o casing in demolition charges

Hexonita. Consists of RDX 80 & NG 20%

Hexonita Gelatinizada. A plastic expl consisting of RDX 50, NG 46 & NC 4%

Minolex. A castable expl compn, consisting of TNT 40, AN 20 & Al powd 20%, used as a bursting chge m ammunition

Tritolita (Cyclotol). Castable compn of 50/50 RDX/TNT used as a bursting chge in ammunition

Tritolital (Torpex). Castable compn consisting of TNT 60, RDX 20 & Al 20% used as a bursting chg e in ammunition

Sweden: *Bonit* (Cyclotol).A name applied to military expls developed by AB Bofors Nobelkrut for underwater bursting charges. Bonit 1: RDX 50 & TNT 50%; Bonit 2: RDX 70 & TNT 30%; and Bonit 3. RDX 60 & TNT 40%

Hexatol. Same as Bonit 3 used as a bursting chge in Shells, Mines, Boosters & Demolition Charges

Hexotonal (Torpex). Castable expls developed by AB Bofors Nobelkrut as military underwater & bursting charges for many types of ammunition:

a) RDX 40, TNT 44, Al 15 & wax 1%

b) RDX 40, TNT 40, Al 15 & wax 5%

c) RDX 30, TNT 50 & Al 20% plus wax 1 & carbon black 1.5%. See also Addnl Ref 43, p C630

Switzerland: *Hoxonit.* Plast expl developed by Dr A. Stettbacher:

a) RDX 20-50 & NG 80-50%

b) RDX 50, NG 46 & NC 4%

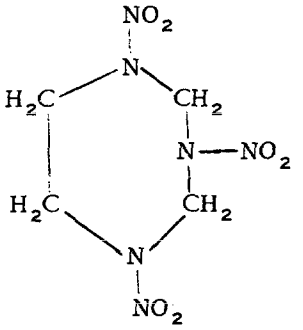
The use of RDX in solventless - extruded double-base proplnts & in rocket proplnts (Ref 33) has been studied. The refs listed should be consulted for addl info on the uses & props of RDX in expl mixts

Refs for RDX Compositions: 1) L. Pauling, OSRD **5945** (1945) (Stability of exptl gun propellants contg RDX) 2) DuPont Co, OSRD **6216** (1945) (Development of propellants contg RDX) [See CC (Cyclonite Cannon) Type Propellants, Vol 2, p C92-R and CR (Cyclonite

Rifle) Type Propellants, Vol 3, p C 563-R] 3) E.G. Cockbain et al, BritP 574271 (1945) & CA **42**, 7531 (1948) (Desensitization of PETN & RDX by adding to a hot aq paste of the expl a small amt of a surface active agent and molten wax as a coating) 4) Anon, Summary Tech Rept of Div 8, NDRC, Vol 1 (1946), 30-33 5) All & EnExpls (1946), 121ff 6) S. Fleischnick, "Semi-Plastic RDX Compositions", PATR **1614** (1946) 7) H. Aaronson, "Semi-Plastic RDX Compositions" PATR **1634** (1946) 8) Dinamite Nobel Società Anonima, ItalP 427535 (1947) & CA **43**, 7688 (1949) [Plastic explosive: RDX 85-89, petrolatum 12-10, thiokol plastic binder (Tioplasto molle) 2-0.5 & glycerophthalic resin 1-0.5%] 9) A.N. Campbell & H.A. Kushnarov, CanJRes **25B**, No 3, 216-27 (1947) & CA **41**, 7218 (1947) (Study of binary mixts of RDX/TNT) 10) Dinamite Nobel Società Anonima, ItalP 439931 (1948) & CA **44**, 6130 (1950) (Plastic explosive: RDX 64-75, petrolatum 10-12, plastic binder 0.5 2.0, glycerophthalic resin 0.5-1.0 & Al powder 25-10%) 10a) Bombrini Parodi-Delfino, ItalP 43093 (1948) & CA **43**, 8682 (1949) 11) "Montecatini" Società Generale per l'Industria Mineraria e Chimica, ItalP 433633 (1948) & CA **44**, 1709 (1950) (Bursting expl: RDX (or PETN) 75 & nitroisobutylglycerol triacetate 25% is melted at 80°) 12) Polverifici Giovanni Stacchini SA, ItalP 433636 (1948) & CA **44**, 1709 (1950) [Bursting expl: RDX (or PETN) 5-65, TNT 30-90 & powd metal (such as Si, B, Mg, Cu, Fe, Al or Zn) 5-30 parts] 13) Ibid, ItalP 445601 & 445603 (1949) & CA **45**, 3160 (1951) (Bursting expls: RDX 5-65, TNT 40-90 & Al 5-30 parts and RDX 5-65, TNT 15-45, Dinitronaphthalene 15-45 & Al 5-30 parts) 14) Direzione Superiore del Servizio Tecnico di Artiglieria, Ital P 450103 (1949) & CA **44**, 11098 (1950) (Bursting expl: RDX 92-86 & castor oil 8-14% 15) L. Médard & État Française, FrP 947052 (1949) & MP **31**, 443 (1949) 16) L. Médard & État Française, FrP 947053 (1949) and MP **31**, 445 (1949) & **32**, 219 (1950) 17) A. Le Roux, MP **32**, 199-200 (1950) 18) A. LeRoux, MP **33**, 95-111 (1951) 19) H.H. Fassnacht & C. A. Woodbury, USP 2548880 (1951) & CA **45**, 10590 (1951) (Gelatinous expl consisting of RDX 77, MNT 5, DNT 10, TNT 4, Tetryl 3 & NC 1% has a d = 1.58g/cc and velocity of detonation = 7500 meters/sec) 20) G.B. Kistiakowsky et al, USP 2606109 (1952) & CA **47**, 4083-84 (1953) [Plastic nonhardening exp: RDX 88 & bodied or polymerized oil (voltolized sperm, rape seed, or paraffin) 12%] 20a) E. James et al, US Atomic Energy Comm **LA-1448** (1952) & CA **54**, 21762 (1960) (Plastic-bonded RDX) 21) L. Avogadro di Cerrione, AnnChim(Rome) **43**, 525-34 (1953) & CA **48**, 9063-64 (1954) (Stability of mixts based on Pentrite & T4) 22) T. Tsukii & S. Kikuchi, Jap P 4443 (1954) & CA **49**, 10628 (1955) (Cyclonite in expl compn for riveting) 23) D.T. Lewis, Analyst **79**, 644-48 (1954) & CA **49**, 1329 (1955) (Polarographic detn of TNT & RDX in expl mixts) 24) H.W. Adam et al, "Development of an RDX Explosive of the C3 Type", PATR **2028** (1954) 25) C.E. Jacobson, "Characteristics of 97/3 RDX/Wax, "PATR **2204** (1955) 26) A. Ya Apin et al, ZhFizKhim **32**, 819-23 (1958) & CA **52**, 21108 (1958) (Expln of Hexogen in the presence of Al & Be) 27) H.W. Koch & H. Freiwald, Explosivst **1958**, 279-84 (Viscosity of Cyclonite - TNT mixts) 28) L. Andrussov, Explosivst **1949**, 89-91 (Discussion on the viscosity of Cyclonite-TNT mixts and the article of Koch & Freiwald) 29) W.O. Williamson, JApplChem (London) **9**, 120-26 (1959) & CA **53**, 10763 (1959) (Fused mixts of RDX & TNT) 30) J. Wilby, "Thermal Decomposition of RDX/TNT Mixtures", ARDE Rept **(MX) 16/59**, Part 1 (1959) 31) F. Spring & C. Glauser, GerP 1078918 (1960) & CA **55** 17007 (1961) [Desensitized Cyclonite expl: RDX 69, Al (100 μ size) 29, lacquer (Ethyl cellulose 7.7, dibutylphthalate 3.3, centralite 1.5 & mixed aromatic hydrocarbons 87.7%) 16 grams] 32) W. Kegler, Explosivst **8**, 1-4 (1960) & CA **54**, 8085 (1960) (Production of Composition B casts having a high degree of chem & phys homogeneity: use of high concns of RDX having the fines present, addn of 0.5% NC, 0.1 - 0.2% o- & p-nitrotoluenes, by melting & casting in vacuo, and by controlled cooling) 33) Dynamit-Nobel AG, GerP 1093717 (1960) & CA **55**, 21592 (1961) (RDX rocket propInts mech stable: made by incorporating 26-50% of NGu in the form of fine crysts, the moist mixt of which is extruded thru a screw press, during which H_2O is removed) 34) T.G. Blake, USP 2982640 (1961) & CA **55**, 17007 (1961) [Deflagrating expl for 20 & 40mm Projectiles: NH_4ClO_4 36, TNT 4, Al 54 & RDX (coated with paraffin wax 0.2%) 5.8%] 35) J.W. Dawson & F.H. Westheimer, USP 2982641 (1961) (Improved aluminized expl: RDX 42, TNT 40 & Al 18% with 0.5% anhyd $CaCl_2$ added) 36) W.A. Gey, USP 2982639 (1961) & CA **55**, 17008 (1961) (Non-

sticky HE with reduced sensitivity: RDX + 10% of an ether of linear methylene nitramine) 37)H. W. Koch & H. Freiwald, Explosivst **9**, No 10, 221-33 (1961) & CA **57**, 16950 (1962) (Rheological & other props of suspensions of RDX in molten TNT) 38) J. N. Ayres & C.W. Randall, "RDX/Calcium Stearate Binary System Explosive Sensitivity Calibration", NOLTR **63-91** (May 1963) 39) J. Wilby, "The Thermal Decomposition of RDX/TNT Mixtures", Part 2, ARDE Rept (X) **7/63** (June 1963) 40) E. Viner, "Evaluation of Cast Loaded and Press Loaded 86/14 RDX/Wax Filler in Projectile, HEP-T, 105 mm, M393E1 (U)", Chamberlain Corp Contract DA-11-022-AMC-2140 (Z) (Nov 1963) (S) (Not used as a source of info) 41) W.R. Tomlinson, Jr & O.E. Sheffield, "Properties of Explosives of Military Interest", USP AMCP **706-177** (1963) [A reprint of ORDP 20-177 based on PATR **1740** (1958)] 42) J. Wilby, "The Thermal Stability of Some Explosive Compositions Containing RDX", ARDE Memo **16-64** (April 1964) (C) (Not used as a source of infor) 43)Addnl Ref: A.B. Bofors Nobelkrut, "Analytical Methods for Powders and Explosives," Bofors, Sweden (1960), pp 201 & 203

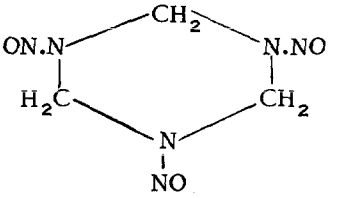
Cyclotrimethylenetrimitramine (Cyclonite) Homolog (1,3,6 - Trinitro - 1,3,6 - Triazacycloheptane),



mw 236.15, N 35.59%; two polymorphs are obtd: (α) heavy prismatic crysts and (β) short & acicular crysts, mp 165-1.65.5° (with decompn); very sol in acet, methyl ethyl ketone, nitromethane & dioxane; insol in alc, benz, acetic acid & chlf; was prepd by nitrolysis of methylene-*bis*- 3,6-dinitro - 1,3,6 - triazacycloheptane, which was synthesized from N, N' - dimethylol - 1, 2 - dinitraminoethane, formaldehyde & ammonia. This Cyclonite homolog is much less stable than Cyclonite in that it is 7% destroyed in 2 hrs by boiling water and completely decompd in 10 mins by 5% aq NaOH

Refs: 1) Beil - not found 2) G.S. Myers & G. F. Wright, CanJRes **27B**, 489-98 (1949) & CA **43**, 9073 (1949)

Cyclotrimethylenetrinitrosamine; Hexahydro- 1,3,5 - trinitroso-s-triazine; 1,3,5-Trinitroso-1,3,5-triazocyclohexane and R-Salt (called 1,3,5-Trinitroso-trimethylentriamin & code name R-Salz in Ger; and Cyclo-triméthylène-trinitrosamine in Fr),



; mw 174.13, N48.27%; yel ndls or prisms (from alc), mp 105-07; Q_f^v -914 cal/g, Q_c^v 876 cal/g & Q_e^v 876 cal/g (Refs 1-4); readily sol in acet & boiling alc; sl sol in eth, chlf & benz; insol in petr eth

R-Salt was discovered in 1888 simultaneously by P. Griess & G. Harrow and by F. Mayer when Na nitrite reacted with hexamethylenetetramine in acid soln. This compd was studied later by P. Duden & M. Scharff (1895), M. Delépine (1896) and others (Refs 1,2,3 & 4). Because R-Salt could be made at first only in low yield, many years elapsed before it was considered as a practical expl. Studies by Prof Römer (Ref 5) in Germany created new interest in this expl, especially as to its behavior in mixts with other sub stances

Preparation: All investigators, prior to WWII prepd R-Salt by essentially the same method (Ref 1). It was Bachmann & Deno (Ref 6) who discovered that the chief factor that determines the nature of the product is the pH of the soln. At pH1, the trinitroso is formed exclusively; at pH 2, a mixt of Trinitroso & dinitroso derivs is produced; and in the range pH 3-6, the dinitroso compd is formed exclusively. Thus, some of the early apparently conflicting results as to conditions & nature of the products were made clear. Bachmann & Deno (Ref 6) added simultaneously a soln of $NaNO_2$ in w & 6N HCl, at a rate reqd to maintain a pH of 1, to a soln of hexamethylenetetramine in ice & water. The mixt was held at 0° for 30 mins. The yeild of practically pure product was 50% (mole for mole basis)

Aubertein (Ref 7) also studied the factors affecting the prepn of R-Salt and discussed the mechanism of the reaction. The apparatus used and experiments made on pilot-plant batch & continuous methods of production to obtain the desired

cryst sizes were reported by Ficheroulle & Kovache (Ref 8)

Explosive and Other Properties of R-Salt. Tada (Refs 9 & 16) studied the effect of neutral salts and of solv effects on the acid decompn of R-Salt. Šimeček (Ref 12) reported the treating of R-Salt with AN & H_2SO_4 to obtn *N, N-Dinitro- N''-nitroso-cyclotrimethylenetriamine* which on further treating with the nitrating mixt yielded 98% of RDX. The decompn of R-Salt by concd H_2SO_4 was also reported by Šimeček (Ref 13). The thermal decompn of R-Salt is the subject of a report by Fowler & Tobin (Ref 10) who also detd its IR spectrum

Médard & Dutour (Ref 11) prepd R-Salt and detd its expl props and some other props in detail. They found R-Salt to be brisant & powerful, but easily decompd in the presence of acid

Abel's test at 110° - no color to iodine-starch paper in 15 mins

Coefficient de self-excitation (CSE) (See Vol 1, p XIV, under Gap Test) - 3 explns out of 3 trials at 10cm distance betw cartridges and 3 failures in 3 trials at 12cm distance for chge at d 0.85/cc vs mean distances of 21cm for RDX at d 1.20g/cc, 7 cm for TNT at d 1.00 g/cc & 13.5 cm for PA at d 1.00 g/cc

Coefficient d'utilisation pratique (CUP) (See Vol 1, pp IX - X) - specific work by Pb block test is 126% vs 100 for PA & 135 for RDX

Combustion test (See Vol 1, p X). The powdered sample, placed in a 20mm hemicylindrical trough, burns easily & regularly with a flame height of 10 cm and a rate of advance of 10cm/min; a 1 kg conical pile burned in 4½ min with a yel-orn flame 1m high leaving a large vol, small mass residue

Compatibility with metals - Tests of R-Salt with Cu, Fe & Al powders (in mixts 2 parts R-Salt to 1 p metal) showed little reaction when heated in sealed tubes for 30 mins at 115°

Compatibility with other expls - A mixt of 2 parts Picric Acid & 1 part R-Salt gave violent decompn after 2 hrs at 60° with a large quantity of nitrous gases leaving a dk-brn, tarry residue; at 100° the decompn occurred in 10-15 min. A similar mixt of NG & R-Salt gave no apparent decompn after 5 days at 90°. It was believed that acidity produced the decompn in PA/R-Salt mixts

Density of packing - 0.84 g/cc

Density under pressure - 1.10 g/cc at 170kg/sq cm, 1.23 at 340, 1.37 at 680, 1.44 at 1020, 1.53 at 1700, 1.57 at 2380 and 1.59 g/cc at 3000 kg/sq cm pressure

Detonation rate (tests in 30 mm diam cartridges) - 5180 m/sec at d 0.85, 5320 at 0.90, 5760 at 1.00, 6250 at 1.10, 6000 at 1.20, 6970 at 1.30, 7330 at 1.40, 7500 at 1.48, 7600 at 1.50, 7720 at 1.52, and 7800 m/sec at d 1.57 g/cc. For cast R-Salt 7000-7300 at d 1.42 g/cc

Fusion Tests (of mixts with TNT) - 74° at 10% of R-Salt, 68° at 20, 62° at 30, 55° at 40, 61° at 50 69° at 60 and 77° at 77% R-Salt, with a eutectic of 55° at 42%. The solidified eutectic had a deton rate of 7000m/sec

Impact sensitivity (10kg wt) - 50 explns out of 100 tests at 1.25 m with Bourges capsule contg 25-30 mg sample; with 18mg of sample, the same wt and height of fall gave 32 explns out of 100 trials (see also Vol 1, p XVII)

Sensitivity to initiation (30 mm diam cartridges)- grams of MF to cause complete deton at density (d), 0. 30g at d 0.85, 0.40 at 1.20, 0.50 at 1.40 and 2.5g at d 1.57 g/cc. In these same cartridges, cast R-Salt at d 1.42 reqd a Briska detonator for complete detonation (See also Vol 1, p XXIII)

Sensitivity to initiation (by heat) - when 0.02g sample in a 10mm test tube was immersed 10 min in a molten metal bath and heated at 5°/min, melting began at 105°, decompn began at 150°, nitrous gases evolved at 160°, and the sample was entirely decompd at 170°. When the tube was plunged into a bath at 300°, decompn was immediate, at 250° decompn was in 40 sec, at 200° in 2 min, at 150° decompn proceeded very slowly, at 110° some bubbles appeared but no increase in decompn occurred in 20 mins (See also Vol 1, pp XXII - XXIII)

Specific work - See Coefficient d'utilisation pratique

Stability in storage - loosely packed in covered wooden boxes for 6 yrs at ambient temp and protected from the sun, R-Salt showed no change in color but its mp decreased from 104.5 to 104° and its CUP value from 125.5 to 123.5

Some addnl props of R-Salt detd at PicArsn are given in Ref 15

Uses. R-Salt was proposed in Germany as an ingredient of expl mixts. Römer (Ref 5) and Fedoroff (Ref 14) list the R-Salt expls in Table, p C 632

Refs: 1) Beil **26**, 6 & (3) 2) R. McGill, OSRD Rept **830** (1942) 3) F.R. Benson, "Study Explosive Characteristics of Aliphatic Nitramines of of Low Carbon Content", PATR **1174** (1942) 4) Blatt, OSRD **2014** (1944) 4) W.E. Bachmann, "Studies on RDX (B) and Related Compounds", OSRD **5186** (1945) 5) D.G. Römer, "Report on

Explosives", BIOSGP 2-HEC 5742 (1946) (PB Rept #85160) 6) W.E. Bachmann & N.C. Deno, "The Nitrosation of Hexamethylenetetramine and Related Compounds", JACS **73**, 2777-79 (1951) 7) P. Aubertein, MP **33**, 227-31 (1951) 8) H. Ficheroulle & A. Kovache, MP **33**, 241-53 (1951) 9) H. Tada, JChemSocJapan, Pure Chem Sect **77**, 434-41 (1954) & CA **51**, 438 (1957) 10) J.P. Fowler & M.C. Tobin, JPhChem **58**, 382-83 (1954) 11) L. Médard & M. Dutour, MP **37**, 19-24 (1955) & CA **50**, 12480-81 (1956) 12) J. Šimeček, ChemListy (Czech) **51**, 1323-26 (1957) & CA **51**, 17942 (1957) 13) J. Šimeček, ChemListy (Czech) **51**, 2367-68 (1957) & CA **52** 6367 (1958) 14) B.T. Fedoroff et al, "Dictionary of Explosives, Ammunition and Weapons", German Section, PATR **2510** (1958), p Ger 170 15) O.E. Sheffield, "Properties of Explosives of Military Interest", Section 2, ORDP **20-178**, 2-6 (1958) (Conf, not used as a source of info) [A reprint of PATR 1740, Rev 1 (1958), pp 354ff] 16) H. Tada, JACS **82**, 263-66 (1960) & CA **54** 10477 (1960)

R-Salt Explosives

Ingredients and Some Properties	Composition (%) of Mixtures:							
	1	2	3	4	5	6	7	8
R - Salt	96.5	46.5	36.5	46.5	36.5	96.5	36.0	40.0
Phenathrene	2.5	2.5	2.5	2.5	2.5	-	-	-
Diphenylamine	1.0	1.0	1.0	1.0	1.0	1.0	1.0	-
RDX (Hexogen)	-	50.0	50.0	-	40.0	-	50.0	40.0 (H_5)
Aluminum powder	-	-	10.0	-	20.0	-	-	20.0
K nitrate	-	-	-	50.0	-	-	-	-
Dimethylethylenedinitramine	-	-	-	-	-	2.5	12.0	-
Unaccounted	-	-	-	-	-	-	1.0	-
Casting Temp, °C	92	92	95	94	95	92	84	-
Density (cast)	1.55	1.65	1.64	1.77	1.74	1.55	-	-
Vel of Deton, m/sec	7600	-	-	6100	7750	7850		-
Pb Plate Test. The mixture is more effective than	TNT	Cyclo-tol	40/60 Amatol	40/60 Amatol	40/60 Amatol	-	-	-
Fragment Density Test (TNT - 40 m)	44 m	47 m	47 m	47 m	-	-	-	-
Stability at 100°	Satisfactory stability for all explosives							
Exudation at 70°	No exudation for any of the explosives							

Notes:

a) H_5 is Hexogen (RDX) phlegmatized with 5% Montan wax

b) Mixture (8) was claimed to be very powerful

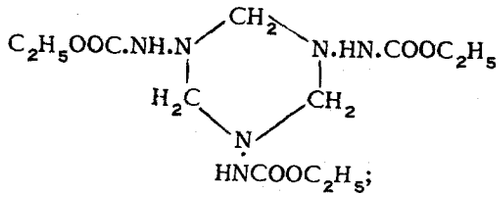
c) R - Salt forms with 28% dimethylethylenedinitramine (DMEDNA) a eutectic mixture, freezing point (fr p) 74°. Fr p of R - Salt with 10% DEMEDNA is 89° and with 5% DMEDNA 93°. Fr p of R-Salt alone 104-106° and of DMEDNA 137°

Cyclotrimethylenetrinitrosamine (R-Salt), Analytical Procedures. No information is available as to the detection, identification or analysis of R-Salt either alone or in the presence of other expls. However, it is believed that R-Salt can be detected qualitatively by the tests for N-nitrosamines given by Feigl (Ref 1) and quantitatively by the chromous chloride method given by Jamison (Ref 2) and Gutmacher et al (Ref 3)

See also Analytical Procedures under RDX in this Volume

Refs: 1) F. Feigl, "Spot Tests in Organic Analysis", Elsevier, NY (1960), pp 167-69 & 178-79 2) C.C. Jamison, Jr , "Determination of Nitrogenous Compounds of Ordnance Interest by Chromous Chloride Reduction", PATM **ACS-3-60** (1960) 3) R.G. Gutmacher et al, "Some Recently Developed Chemical and Physical Analytical Methods", Univ of Calif Radiation Lab Rept UCRL **6639** (1961)

Cyclotrimethylenetriurethane (CTMTU),



mw 348.36, N24.13%; crysts (from alc) mp 100-02°; was prepd by addg 36% formalin & concd H_2SO_4 to a flask contg water, and while the temp was kept below 25° urethane was added. Pptn of a solid which is methylenediurethane occurred and on standing changed to an oil which is impure CTMTU. At this point, the reaction mixt was refluxed for 45 mins and then allowed to cool. It was neutralized with $NaHCO_3$ and the oil extracted with ether. The extract when dried failed to crystallize when cooled & allowed to stand. It was then refluxed with a mixt of acetic acid & acetic anhydride for 12 hrs. The anhydride & acid were distilled and the oily residue poured into water. A semi-solid formed which was recrystd from alc. Yield 28%. Its expl props were not detd

All attempts to nitrate CTMTU (Cyclotrimethylenetriurethane) and thus convert it to RDX failed

Refs: 1) Beil - not found 2) A.T. Bloomquist, OSRD **4134** (1944), 135-36

Cyclotron is an apparatus for imparting high speeds to atomic particles by magnetically accelerating them in spiral paths. It is used especially for bombarding atoms to produce transmutation and artificial radioactivity (Ref 7)

In the cydotron, the particles are forced to move in a spiral of slowly increasing radius and are given a single "kick" by an electric field during each revolution. In this way, particles can gain energies up to hundreds of Mev even though the accelerating voltage is only a few kilovolts. The particles are held in a spiral by a steady magnetic field

In contrast to electrostatic accelerators, the particles in a cyclotron can be accelerated over long distances & extensive time intervals. The problem of focusing, however, is critical. The loss of phase synchronization is also a serious problem in cyclotrons employing fixed frequency accelerating voltages. This problem is solved in a modification of the cyclotron known as the *FM Cyclotron or Synchrocyclotron*, in which the frequency of the accelerating voltage is changed (modulated) as acceleration proceeds so that particles can stay in phase synchronization as they increase their velocity and move out in radius. There are two new aspects to FM cyclotrons. In one of these "the phase oscillation" type-particles vary in phase about some average value, and in the other "the short-duty cycle" type - particles can be accelerated only a short fraction of the time because of the manner in which the frequency changes

Cyclotrons consume large amts of power both for the magnet and for the accelerating voltage. The largest cyclotrons require about 500 kilowatts of power for the accelerating voltage Essentially all of this power is ultimately dissipated in heat, and the actual beam of particles may possess a power of only 10 watts (Ref 9)

The first cyclotron was constructed by E.O. Lawrence & M.S. Livingston in the early 1930's. It had a magnet of only 2½-inch pole diam and accelerated protons to 80,000 ev. This was followed at the Univ of Calif by the 9, 11-, 27½-, 37-, and 60-inch cyclotrons, and finally the 184-inch machine (Ref 8)

Some 50 cyclotrons are now in use in research labs throughout the world. One-half of these are in the USA. Cyclotrons are used to accelerate protons & heavier particles. With higher energy than is available from electrostatic generators, it is possible

to explore almost all aspects of nuclear structure, and with the highest energies one can generate beams of π-mesons. In addn, cyclotrons are used to produce certain radio isotopes that can not be formed in nuclear reactors (Ref 9)

See Refs below and also **Betatron** and **Bevatron**, Vol 2, pp B108-L & B109-R, respectively
Refs: 1) W.B. Mann, "The Cyclotron", Chemical Publishing Co, NY (1940) 2) H.S. Taylor, E.O. Lawrence & I. Langmuir, "Molecular Films, The Cyclotron and the New Biology", Rutgers Univ Press, New Brunswick, NJ (1942), New edit (1946) 3) M.E. Nahmias, "Le Cyclotron", Editions de la Revue d'Optique Theorique et Instrumentale, Paris (1945), 254pp 4) H.O. Tayler, E.O.Lawrence & I. Langmuir, "Couches Moléculaires, Cyclotron et Nouvelle Biologie", A. Michei, Paris (1948), 198 pp 5) M.B. Mann, "The Cyclotron", Wiley & Sons, NY (1950), 103pp 6) N.D. Fedorov, "Tsiklotron-Tsiklicheskii Rezonansnyi Uskoritel' Ionov" (Cyclotron-Cyclic Resonance Ion Accelerator), AtIzdat, Moskva (1960) 88pp 7) OrdTechTerm (1962), 89 8) EncyclBritannica **1** (1963), 66-70 (under Accelerators, Particle) 9) Collier's Encycl **18** (1965), 465-68 (under Particle Accelerator)

CYH. An ABL modified double-base propInt for rockets. Its compn & props are given in Conf "Propellant Manual" SPIA/M2 (1962), Unit No 596

CYH (77). A Hercules Powd Co modified double base propInt for rockets. Its compn & props are given in Conf "Propellant Manual" SPIA/M2(1962) Unit No 638

CYI (75). An ABL modified double-base propInt for rockets. Its compn & props are given in Conf "Propellant Manual", SPIA/M2 (1962), Unit No 637

Cylinder Barrel. A type of shotgun barrel with cylidrical bore, that is without *Choke* (qv)
Ref: OrdTechTerm (1962), 89

Cylinder, Recoil. A cylinder with component parts designed to cushion the backward motion of a cannon by springs, or by the slow passage of air or fluid thru holes in the piston when the gun is firing. Also part of the recoil mechanism
Ref: OrdTechTerm (1962), 90

Cylindrical Explosions. For the past several years many theoretical & exptl investigations have been made to gain a more detailed understanding of blast waves (See Vol 2, p B180 BLAST EFFECTS IN AIR, EARTH AND WATER). The behavior of the spherical blast wave is now well understood. Collins (Ref) has reported work which demonstrates that finite source cylindrical explns can be produced by bursting pressurized glass cylinders. The use of several types of cylindrical glass diaphragms has been studied with a view to producing reasonably strong, symmetrical shock wave patterns. The cylindrical diaphragms (2" diam x 4" long & 2mm thick walls) were initially pressurized with air or a combustible gas mixt (hydrogen-oxygen-helium). The cylindrical blast was produced by breaking the diaphragms with a mechanical breaker, or igniting the mixt with an exploding wire and letting the cylinder break due to overpressure

The properties of the cylidrical expln flow were recorded photographically. Features such as the main shock wave, contact front, and imploding second shock wave previously predicated for the spherical blast were shown experimentally to be present in the cylindrical expln. A representative sketch of the features found in a pressurized (chged with air to a pressure of 115 psi) cylindrical expln is shown in the Fig A. The open-end cylidrical glass diaphram also proved satisfactory in generating stronger cylindrical blasts (See Fig B). The wave system produced using a combustible gas mixt shows the addn of a deflagration front to the basic cylindrical wave system (See Fig C)

The multi-spark schlieren photographs obtd by Collins (Ref) have proved most valuable for their qualitative insight into the general behavior of the cylindrical blast wave. The results of this investigation show that cylindrical explns can be satisfactorily generated and adapted to laboratory observation & study
Ref: R. Collins, "Some Methods of Generating Cylindrical Explosions", Univ of Toronto, Inst of Aero-physics (UTIA) Tech Note **43** (1960)

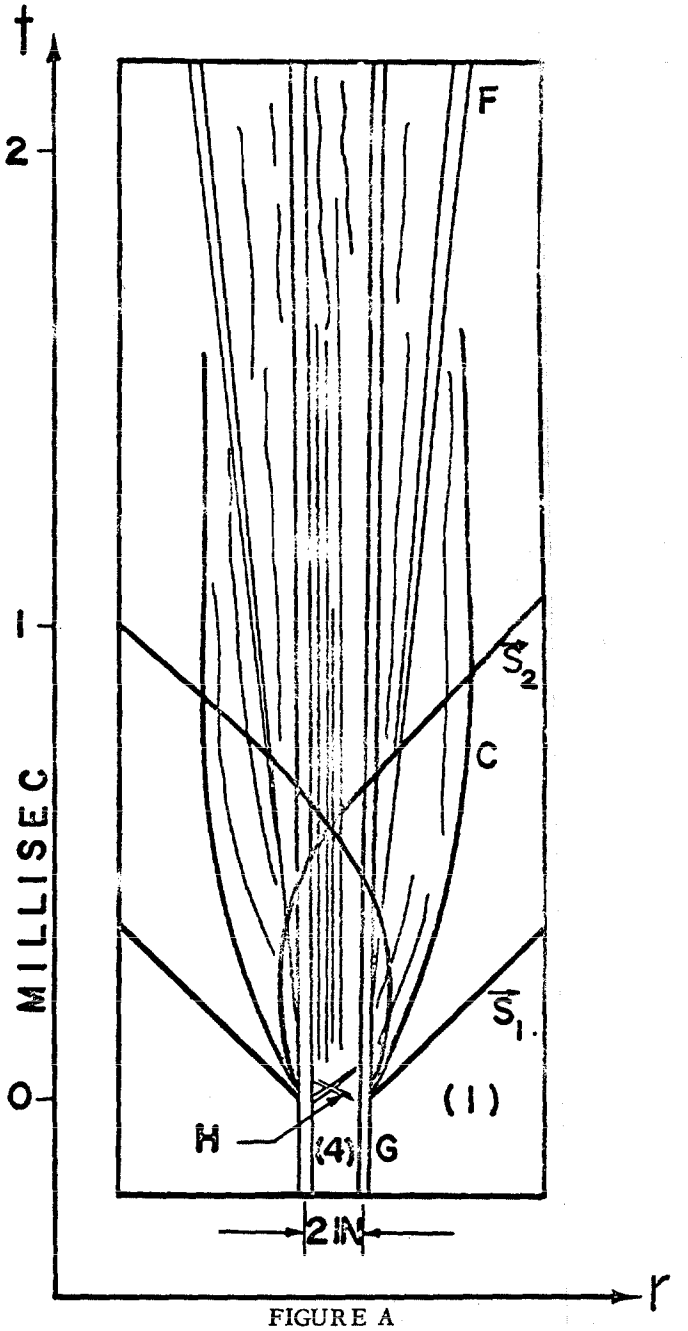


FIGURE A

BLAST FROM A 2" DIAM PRESSURIZED SEALED GLASS CYLINDER

G = Sealed end glass cylinder 2½" long
F = Glass fragments
S_1 = Main shock wave
S_2 = Second shock wave
H = Head of rarefaction wave
C = Contact surface air/air, initial inner pressure 115 psi, temp 297°K
(1) = atm external to cylinder
(4) = interior region of cylinder

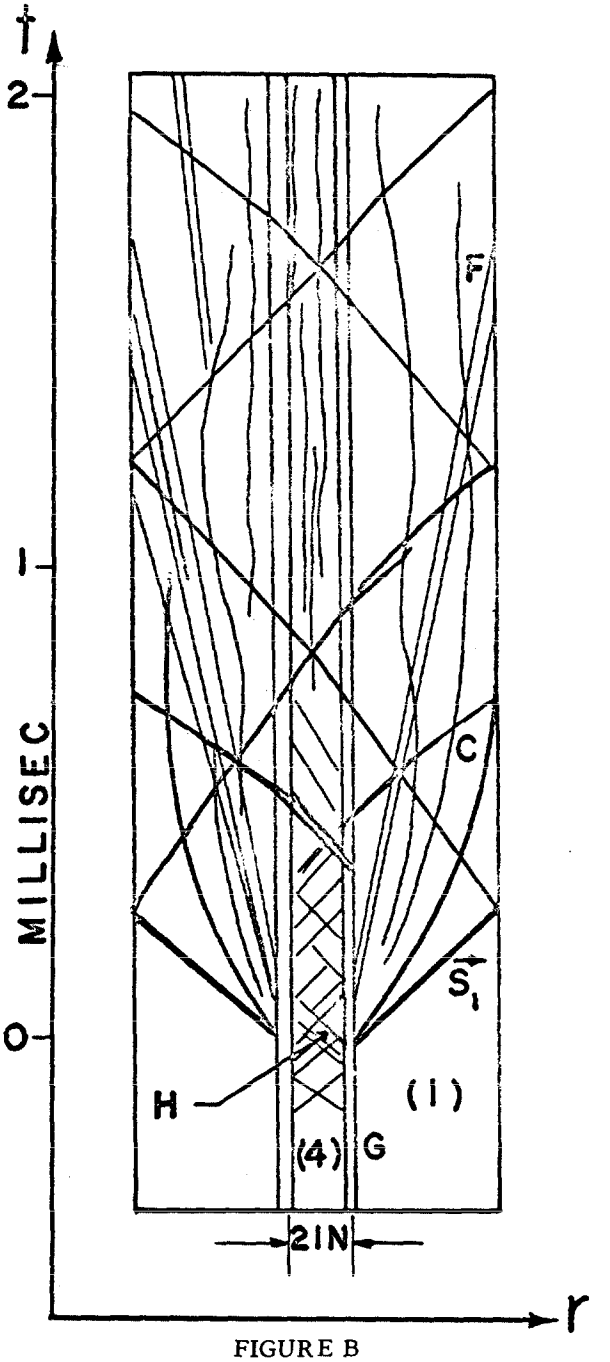


FIGURE B

CYLINDRICAL EXPLOSION FROM A PRESSURIZED OPEN-END GLASS CYLINDER

G = Open-end glass cylinder 2" diam × 2½" long
F = Glass fragments
S_1 = Main shock wave
H = Head of rarefaction wave
C = Contact surface air/air, initial inner pressure 350 psi, temp 300°K
(1) = atm external to cylinder
(4) = interior region of cylinder

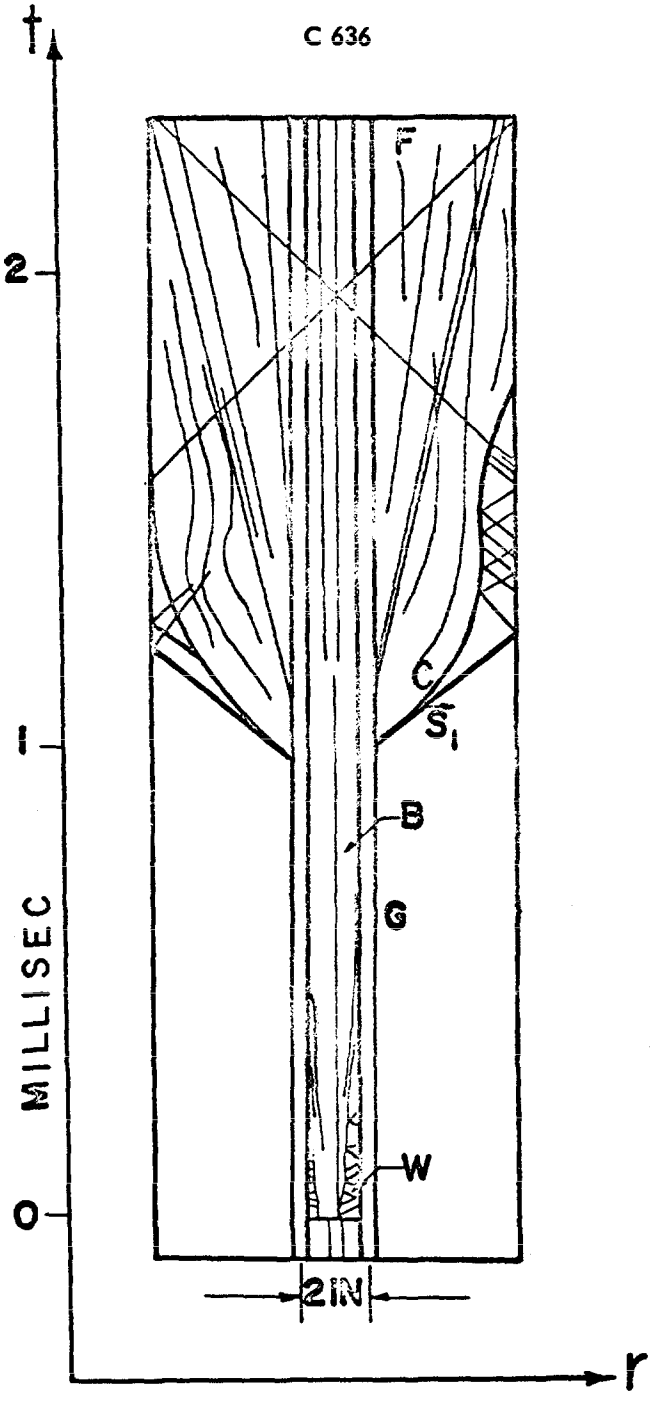


FIGURE C
COMBUSTION DRIVEN CYLINDRICAL EXPLOSION
FROM A GLASS CYLINDER

G = Open-end glass cylinder 2" diam × 2½" long
F = Glass fragments
E = Exploding wire initiator
B = Burning flame of gas mixt
S_1 = Main shock wave
C = Contact surface air/combustible gas (0.2 H_2 + 0.1 O_2 + 0.7 He) initial inner pressure 88 psi, temp 299°K

CYMENES

Cymene, Isopropyl-toluene or 1-Methyl-isopropyl-benzene (o-Cymene) and iso-Cymene (m-Cymene) (called o-Cymol, m-Cymol, p-Cymol, Methyl-isopropyl-benzol or Isopropyl-toluol in Ger), $CH_3C_6H_4CH(CH_3)_2$; mw 134.21, H 10.51%. Three isomers are known and described in the literature

o-Cymeme or 1-Methyl-2-isopropylbenzene, col liq having an aromatic odor, fr p -71.6, bp 178.6, d 0.8766, n_D 1.5006 at 22°; sol in alc, eth & chlf; insol in w; can be nitrated to form expl compds; other props & methods of prepn are given in Beil (Ref 1)

m-Cymene or 1-Methyl-3-isopropylbenzene, col liq having an aromatic odor, fr p -63.8°, bp 175.5° d 0.8610 at 25°, n_D 1.4925 at 25°; sol in alc eth & chlf; insol in w; can be nitrated to form expl compds; other props & methods of prepn are given in Beil (Ref 2)

p-Cymene or 1-Methyl-4-isopropylbenzene, col liq having an aromatic odor, fr p -67.7 to -68.9°, bp 177.3°, d 0.8573 at 20°, n_D 1.4907 at 20°; sol in alc, eth & chlf; insol in w; obtd commercially as a by-product in the sulfite digestion of spruce or other resinous wood pulp used in paper manuf; other methods of prepn & props are given in Beil (Ref 3). This is the only cymene produced commercially. It is used in org synthesis and in synthetic rubber manuf. It can be nitrated to form expl compds

Refs: 1) Beil **5,** 419, (204), [322] & {946} 2) Beil **5,** 419, (204) [322] & {948} 3) Beil **5,** 420-23, (204-05), [322-25] & {948-55}

Nitrocymenes

The following nitrocymens are described in the literature. Of these, only the nitro derivs of p-cymene may be considered of interest at present because p-cymene is the only compd available commercially

Mononitrocymenes, $CH_3C_6H_3(NO_2)CH.(CH_3)_2$; mw 179.21, N 7.82%. *4-Nitro-o-cymene,* liq, bp 144-45°, d 1.078 at 20°, n_D 1.543 at 20° (Ref 1); *6-Nitro-m-cymene,* liq, bp 255-65° (dec); was prepd by nitrating m-cymene with cold fuming HNO_3 (Ref 2); and *2-Nitro-p-cymene,* oil having a pleasant smell, bp 134° at 15mm pressure, d 1.074 at 20°, n_D 1.5301 at 20°, Qvapzn 1410 kcal/mol; may be prepd by nitration of p-cymene with mixed HNO_3/H_2SO_4 acid or by other methods (Ref 3); and *3-Nitro-p-cymene,* col ndls (from dil alc), mp 50.5°, bp 133.5° at 20mm pressure, Qvapzn 1290 kcal/mol; was obtd in addn to other products by nitrating p-cymol, or by diazo-tizing either 3-nitro-2-amino-1-methyl 4-isopropylbenzene or the 5-nitro deriv in concd H_2SO_4 or in glac AcOH & concd H_2SO_4 and boiling the diazonium salt soln with alc (Ref 4)

Tisell & Villaume (Ref 5) patented in 1918 a process for manufg expls by mixing nitrocymenes with O-carriers, to which may be added combustible substances & silicons for raising the temp of expln, but not addg NC. One such mixt contd nitrocymene 15-20, NH_4ClO_4 or NH_4NO_3 70-80, ferrosilicon 5-10 & woodmeal 1-5%

Refs: 1) Beil **5,** {948} 2) Beil **5** 420 3) Beil **5,** 424, (206) [326] & {960-61} 4) Beil **5,** {961} 5) C.L. Tisell & E.J. Villaume, BritP 126196 (1918) & CA **13,** 2280 (1919) 6) Jordan (1954), Chapt 7, p 186 (gives vapor pressure values for p-cymene, 2-Nitro-p-cymene and 3-Nitro-p-cymene)

Dinitrocymenes, $CH_3C_6H_2(NO_2)_2CH(CH_3)_2$; mw 224.21, N 12.50%. *2, 3-Dinitro-p-cymene, mp* 90-90.5° (Ref 1); *2,5-Dinitro-p-cymene,* prisms (from dil alc), mp 77-78°; readily sol in alc, eth & benz; was prepd by heating p-dinitrosocymene with a large excess of HNO_3 (d 1.35) (Ref 2); and *2,6-Dinitro-p-cymene,* crysts (from MeOH), mp 51-54° bp 161-63° at 5-6 mm; sol in alc & eth; can be prepd by nitration of p-cymene or of 2-nitro-p-cymene with HNO_3 (d 1.52) (Ref 3). This latter compd cannot be detonated by Tetryl booster. It was proposed as a plasticizer for NC

Refs: 1) Beil **5** (962) 2) Beil **5** 425 & {962} 3) Beil **5,** 425 (206), [326] & {962}

Trinitrocymenes, $CH_3C_6H(NO_2)_3CH(CH_3)_2$; mw 269.21, N 15.61%. Only two isomers are known: *Trinitro-m-cymene,* yel plts (from petr eth), mp 72-73°; v sol in alc or eth; was prepd by nitrating m-cymene with mixed HNO_3 + H_2SO_4 (Ref 1); and *2, 3, 6 - Trinitro-p-cymene,* ndls (from alc), mp 124-25°; was prepd by heating 2,3 - dinitro-p-cymene or 2,5 - dinitro-p-cymene with fuming HNO_3 & fuming H_2SO_4 at 60-70° (Ref 2)

The trinitrocymenes are expl compds, although such props were not detd & reported

Refs: 1) Beil **5** 420 2) Beil **5** [327] & {964}

Cystamine or Cystogen. Trade name for hexamethylenetetramine (qv) used in medicine as an antiseptic for the urinary tract, and in the manuf of plastics and as an accelerator for the vulcanization of rubber. Hexamethylenetetramine can also be nitrated to form the expl called RDX or Cyclonite (See this Volume)
Ref: Davis (1943), p 397

CZ. Abbr for Combat Zone. A region in a theater of operations where fighting takes place, or where space is designated for the operations of friendly combat forces, extending from the front line to a line or boundary designated by the theater commander. Also a combat area
Ref: OrdTechTerm (1962), pp 77 & 90

Czech Arms and Weapons Plants. Chinn (Ref 3) describes the following automatic weapons of Czech production:
a) *ZB* Machine Guns produced by the Czeskoslovenska Zbrojovka Akciova Spolecnost v Brne (of Brünn). These included: ZB Model 1925, cal 7.92 mm; ZB Model 1926, cal 7.92 mm; and ZB Model 50, cal 7.92 mm
b) *ZB* Machine Gun Model 50-1932, cal 7.92 mm, the only weapon designed by ZB based on the short recoil principle
c) *Bren* Machine Gun, Mk 1, cal 303 was developed in 1935; Bren Machine Gun, cal 7.92 mm was adopted in 1937 by the British and manufd in Canada for the Chinese
d) *ZB* Machine Gun, 7.92 mm was developed in 1936 for the Japanese Army and known there as Model '96. It was manufd by the Jap in one of captured Chinese arsenals. This gun appeared in 1937 as Model '97 for use in tanks, and also as Model '99 with a lighter construction for infantry & paratrooper use. Both models were chambered for the 7.7 mm cartridge
e) *ZB* Machine Gun Model 53-1937, 7.92 mm.
Speechly et al (Ref 2) and Andrews et al (Ref 1) describe the Skoda Werke, Pilsen, Czechoslovakia. This was originally a small arms plant founded in 1859 by the Count of Waldstein, and acquired in 1869 by M. de Skoda. It soon became one of the great armament plants of the world (Ref 5). The Skoda Works plant at Pilsen, in addn to small arms & artillery ammunition, also made parts for howitzers, tanks, motor cars, trailers, bicycles and machine tools. The plant located at Konigratz made tank hulls

Some parts of Czech ammo have been studied at Picatinny Arsenal and the expl components detd (Refs 4 & 6). No info is available to us on Czech arms, ammo & expls used during & since WWII

Refs: 1) J.S.V. Andrews et al, "Armament Design and Development at the Skoda Works, Pilsen, Czechoslovakia", PB Rept **4350** (1945), 90pp 2) G.K. Speechly et al, "Skoda Works, Pilsen, Czechoslovakia", CIOS Rept File **XXXI-70,** Items 2, 18-19 & 21 (1946) (PB Rept **16696**) 3) G.M. Chinn, "The Machine Gun", Bureau of Ordnance, US Navy, Washington, DC, Vol 1 (1951), pp 429-40 & 664 4) A.B. Schilling, "Examination of Unfired Grenade, Hand Offensive (CZECH) One Piece Type, MOD T-34 FMAM-2334", PATR **1949** (1954) (Conf, not used as a source of info) 5) W.H.B. Smith, "Small Arms of the World, Military Publishing Co, Harrisburg, Pa (1955), 104 6) V.T. Riedinger, PAMR **156** (1958) (Conf) and J.J. Campisi, Picatinny Arsenal GLR **58-H1-316** (1958) (Conf) (Not used as sources of info)

END OF LETTER "C"

D

Alphabet letter D begins in Vol 3, p D1

D. Abbr for Detonator

D. Abbr for Di- as in DNT (Dinitrotoluene)

D. Abbr used in Amer jato unit nomeclature which designates a cast double-base proplnt
Ref: OrdTechTerm (1962), 91

D. A letter added to the designation of Fr proplnts to signify that diphenylamine (DPhA) is used as a stabilizer. For example, **BD** is *Poudre B* (See Vol 2, p B1) and when stabilized with DPhA, **BFD$_2$** is Poudre B, for rifle, contg 2% DPhA

D-1 or PNL. A mixt of paraffin wax 84, NC (12%N, ½ sec viscosity) 14 and lecithin 2% developed during WWII in England for the purpose of desensitizing Torpex. The compn known as *Torpex D-1* was Torpex-2 (RDX 42, TNT 40 & Al 18%), contg 5% of D-1
Ref: Anon, "Summary Technical Report of Division 8, NDRC", Washington, DC, Vol 1 (1946), 32

D-2. A modification of D-1 which contained a higher melting paraffin wax. It was used as a desensitizer in expls such as *HBX-1* RDX 39.6, TNT 37.8, Al 17.1, D-2 5.0 & $CaCl_2$ 0.5%; *TNT/D-2-95/5* & *Tritonal/D-2-95/5*
Ref: 1)Anon, "Summary Technical Report of Division 8, NDRC" Washington, DC, Vol 1 (1946), 153 2)Anon, "Military Explosives", Washington, DC, (1955), 153 3)Encycl, Vol **3**, p C488-R (Composition D-2)

DA. Amer abbr for Department of the Army, Chemical Agent *Diphenylachloroarsine* (See Vol 2, p C167-L) & Detroit Arsenal, US Army, Warren, Mich
Ref: OrdTechTerm (1962), 91

DAF Amer abbr for Department of the Air Force
Ref: OrdTechTerm (1962), 91

Dagger. A hand weapon with a short blade, the diminutive of a sword. Because of its convenient size a dagger can be drawn quickly, worn inconspicously and used in a confined space. Its general utility as both a weapon & tool has made the dagger popular with both soldiers and civilians of all ages and cultures. Among the strictly military daggers would be the musketeers dagger which developed into the Bayonet (See Vol 2, p B26-L), the artillerist's stilleto with his scales etched on the blade, and down thru the trench knives of WWII & 2288
Ref: Encycl Britannica **6** (1963), 993-94

Daggs. A short pistol equipped with a wheelock, introduced during the time of Gustavus Adolphus of Sweden (early 17th century)
Ref: J.R.Newman, "The Tools of War", Doubleday Doran & Co, NY (1943), 37-8

Dahmen, Johann, von. An Austrian expls specialist who patented in 1888 one of the low-freezing Dynamites. It was based on NG contg 3-10% of an aromatic nitro compd, such as NBz. He also patented in 1893 *Dahmenit A* (also known as *Victoria Powder*) and *Britaninite* (See Vol 1, p B300-R). In 1895 were patented many AN expls, such as: AN 92.0, phenanthrene 5.5 & $K_2Cr_2O_7$ 2.5% and in 1897: AN 30, NG 30, sawdust 35 & $K_2Cr_2O_7$ 5%. In any of these expls the bichromate could be replaced by Mn dioxide, as for example: AN 90, anthracene 7 & MnO_2 3%. In 1898 was patented the use of acids or their salts as ingredients of safety expls. Eg: 1)NG 93 & CH_3COONa 7% 2)AN 85.5, DNBz 11.2, CH_3COOH 1.4 & oleine 1.9% 3)AN 79.0, DNBz 16.5 & CH_3COOH 4.5% and 4)AN 73.00, DNN 21.75 & CH_3COOH 5.25%
Refs: 1)Daniel (1902), 790-93 2)PATR **2510** (1958), p Ger 32-R

Dahmenit A. (Dahmenite A). One of the expls patented in 1893 by vonDahmen: AN 90.8, $K_2Cr_2O_7$ 2.2, naphthalene 6.5 & curcuma 0.5%; its deton vel is 3680 m/sec at d 1.0
Refs: 1)Marshall **2** (1917), 493 2)PATR **2510** (1958), p Ger 33-L

Daisite. A French Thermite mixt composed of Fe_3O_4 58, Al 18 & sulfur 24%, used during WWII. Sulfur served as a binder and as a fuel
Ref: A.E.Gaul & L.Finkelstein, "History of Research and Development of The Chemical Warfare Service in World War II (1 July 1940 - 31 Dec 1945)", Vol **18**, Part **2 Incendiaries**, Chemical Corps, US Army Chemical Center, Md (1952), p 95

Daisy Cutter Bomb. A British version of an American antipersonnel fragmentation bomb, equipped with an impact fuze, known as the

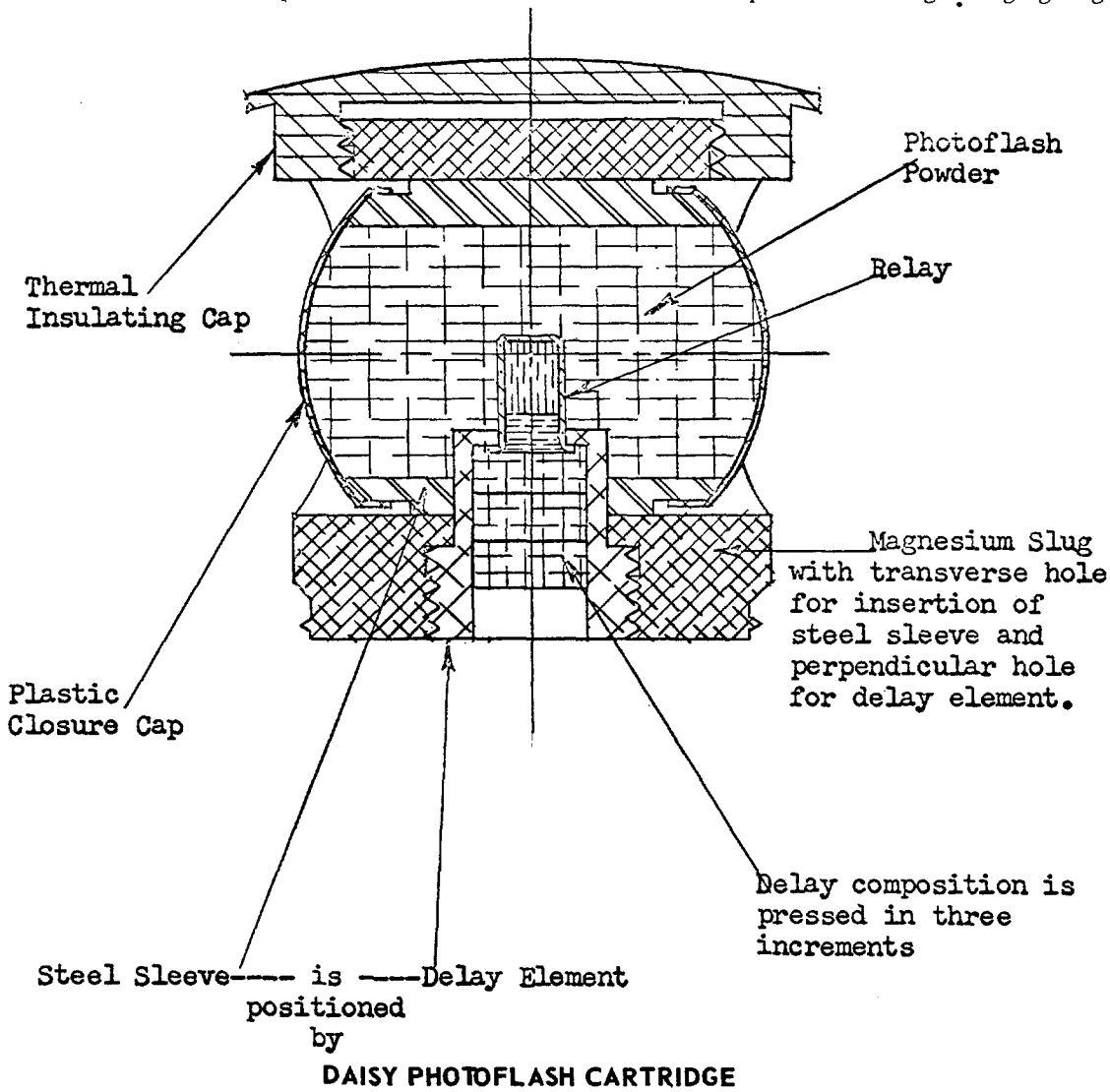
Blue Steel Stand-Off Bomb. It is designed to operate at extremely low altitudes after launching (Refs 1 & 2)

Also a GP bomb, developed during WWII, made more effective by adding a hollow piece of pipe 3 ft long to the nose. US Air Force commandos at Bien Hoa Air Base in Vietnam have been improvising such bombs by removing the nose fuzes from 500-lb bombs. A 3-foot section of pipe, already welded to a shipping plug, is then screwed into the front of each bomb. When a daisy cutter is released from a tactical fighter-bomber, the pipe hits the ground first. Shock triggers a tail fuze, and all or most of the bomb explodes above ground, providing a much greater range of fragmentation delivery (Ref 3)

See also BOMBS in Vol 2, p B225-R

Refs: 1)OrdTechTerm (1962), 91 2)Anon, Ordn **48**, 488 (1964) 3)Anon, Ordn **50**, 101 (1965)

Daisy Photoflash Cartridge (A Space Signal). This is an item, developed at PicArsn, for use with the Atlas missile (cited in Vol 1, p A498-R), which will provide light emission characteristics adequate for tracking but with no missile damage due to fragmentation of the item. An early version of the *Daisy Recoilless Slug* consisted of a solid metal cylinder contg a traverse hole loaded with photo flash compn (60/40 $KClO_4$/atomized Al), sealed by plastic tape and having an intersecting hole for a delay compn. On firing, the cylinder remained intact and there was no recoil but the slug formed damaging fragments. More promising results with respect to reducing damaging frag-



DAISY PHOTOFLASH CARTRIDGE

ments occurred with a polystyrene case

A usuable end item design used a steel sleeve held in the traverse hole of a Mg slug by the delay element (See Fig). The steel sleeve had a high impact polystyrene end cap. The cartridge contd 7.8g of photoflash compn (as above), a 0.6" height of 93/7 Ba chromate/boron delay compn pressed in 3 increments, a relay, and a propulsion system (1.4g of 85/10/15 $BaCrO_4$/boron/Mg,plus 1.4 grains of Type A3 BkPdr

Results of tests at low temp for a simulated altitude of 80,000 ft showed: a)no damaging fragments b)no slug recoil and c)light output to a peak of 25 million candles and an integral light (to 1/10 of peak value) of 40000 candleseconds for a duration of 5 milliseconds

Further development of the Daisy Cartridge is planned to meet the requirement of an ejection distance of 50±5 ft and to increase the integral light to 50000 candleseconds

Ref: J.Hershkowitz, "Development of the Daisy Photoflash cartridge for Tracking the Atlas Missile," PA FRL Pyrotechnics Lab Progress Rept **2** (1959) (Ord Proj TS5-5200 and DOA Proj 50401-031)

Damage Effects of Organic High Explosives. Dr Price of the US Naval Ordnance Lab, Md has studied & reviewed this subject in detail (Ref). The damage ability of an expl has been considered frequently in terms of its *Brisance or Shattering Effect* (See Vol 1, pp VIII - XI and Vol 2, pp B265-B300) and its *Power* (See Vol 1, p XX) or work capacity. These are not always well-defined or well-understood terms, but their qualitative meaning can be described. Target damage must result from the force exerted upon the target, the time profile of that force, and the strength of the target itself. Qualitatively, brisance corresponds to the portion of the pressure -time curve above the yield point of the target, while power reflects the entire pressure-time curve. In her review, Dr Price has replaced the less exact terms of power & brisance by the well -defined parameters of detonation pressure and detonation energy

It has been shown from field-scale damage tests as a measure of expl performance that fragment velocity, shaped-charge penetration, and *Blast Effects in Air, Earth and Water* (See Vol 2, pp B180-L to B184-R) are related to the expl props of detonation energy & pressure

The present work has shown further that, for org expls near their voidless density, the close-in effects (such as shaped-charges & fragmentation) vary directly with detonation pressure, whereas the more distant effects (such as air blast & underwater performance) depend primarily on total detonation energy. The data also show that the third detonation parameter, deton vel, is of little use in correlating the effects by itself although it is, of course, essential in defining the relationship betw the other two deton parameters. These relationships depend upon the rapidity of the chemical reaction of org expls and cannot be expected to hold for more slowly acting expls, for example a mixt contg an inorganic oxidizer (such as AN) and a fuel

Ref: D.Price, "Dependence of Damage Effects upon Detonation Parameters of Organic High Explosives", ChemRevs **59**, 801-25 (1959)

Damage Potential of Air and Ground Blast Waves. As the result of hundreds of accidental explns, a great deal is known regarding the major damage & minor damage potential of air blast waves from unconfined expls (See *Blast Effects in Air, Earth and Water,* Vol 2, pp B180-L to B184-R). Extensive compilations & analyses of these data have led to the establishment of " The American Table of Distances for Storage of Explosives" as Revised and Approved by the Institute of Makers of Explosives [Table given in Ref below, pp 354-56; in Sax (1957), pp 154-55; and in many other sources] and of the US Ordnance Quantity-Distance Tables and Classifications for the storage and handling of explosives [See AMC Safety Manual (1964), Sections 17, 18 & 19]

According to Cook (Ref), the influence of meterological conditions on air blast effects is so great as regards peak pressure or wave intensity that it is most difficult to evaluate influences of chge mass & distance. On the other hand, it is nearly always possible to predict air blast intensity when the temp-altitude & wind -vel-altitude data are available, and vice versa. Also the influence of relatively small dirt cover on air blast loudness & peak pressure is very pronounced

Ground disturbances created at long range by explns are complex and vary markedly even with constant chges, cover & distance. Three types of ground-wave disturbances have been observed: 1)direct or surface ground waves (called S-waves)

2)indirect or subsurface waves (called SS-waves) 3)air-blast induced ground disturbances (called induced ground waves)
The S-wave & SS-waves are, in general, small in magnitude beyond the distances where they might produce damage. They are usually fairly reproducible when measured at the same location, except under conditions of reinforcement by the induced ground wave. When SS-waves occur, they may appear either ahead or behind the S-wave, depending on the depth of and the vel in the strata in which they are returned to the surface. Generally S-waves & SS-waves are much less intense at long range than the induced ground wave produced by unconfined shots or shots under small dirt cover

Cook (Ref) reported that only the S-wave and the induced ground wave were apparent in seismic disturbances from surface explns at Tooele, Utah and for unconfined or dirt-covered shots the induced ground wave had an index 5-30 times greater than the S-wave. Synchronous ground-wave induction apparently requires simply matching of the frequency of the air blast with the natural frequency of the ground in the region of the blast. It is possible therefore to obtn this synchronous ground-wave induction in demolition work by adjusting chge size, dirt cover & distance. Conversely, one should expect to eliminate it by careful selection of chge size, dirt cover & meteorological conditions so that it does not occur

Obviously no induced wave can be more intense than its source. Air blast waves, therefore, are always more likely to cause damage than ground disturbances. Even air blast or induced ground waves beyond the range of damage serve merely as sources of annoyances rather than sources of actual damage
Ref: Cook (1958), pp 353-75

Damming of Explosives. The effect of damming by means of a 5mm thick mantle of NaCl, on the detonation of expls (consisting of NG, NGc, AN, TNT, NaCl & woodmeal) was investigated by Deffet (Ref). As a result of jacketting with pressed NaCl, the deton rate of the various expls increased by 170 to 980 m/sec in comparison to undammed chges, all 26mm in diam (paper jacketed). Further experiments with tubes made of polyethylene 5mm thick and 20 & 26mm diam showed that the length of the reaction zone was shortened by an increase in chge diam, thus increasing its deton rate. At the same diam of 26mm, the length of the reaction zone increased with increasing deton rates for all chges (undammed, polyethylene tube, NaCl dammed). With cases below the thickness of the reaction zone (3 to 4mm), their d & inertia have a decisive effect, while with thick cases (4mm & above) compressibility becomes important. These studies of the effects of chge diam, case thickness and others on the reaction zone & deton rates should lead to an increase in safety of expls
Ref: L.Deffet, Explosivst **10**, 7-11 (1957) & CA **55**, 991 (1961)

Damper. A hydraulic or pneumatic device, incorporated or attached to a *Cartridge Actuated Device* (See Vol 2, p C70-R), in order to limit (dampen) the velocity of the stroking member
Refs: 1)Anon, "Propellant Actuated Devices", ORDP **20-270** (1961), 146 2)OrdTechTerm (1962), 91

DANC Solution. This is a 6.25% soln of RH-195 (1,3-dichloro-5,5-dimethylhydantoin) in acetylene tetrachloride (tetrachloroethane, $Cl_2CH.CH.Cl_2$). Because of the corrosive nature of the DANC soln, its components are packed separately in a dual -compartmented container and are combined just before use. The solv is the most toxic chlorinated org compd known, being 10 times as toxic as CCl_4, and less than 20 drops ingested can cause death

DANC solution is a satisfactory decontaminant, in small-scale operations, for mustard gas (See *HD* in Vol 2, p C168-L under Chemical Agents or Chemical Warfare Agents)
Ref: Anon, "Military Chemistry and Chemical Agents", TM 3-215 and AFM 355-7, US Depts of the Army and the Air Force (1963), 86-87

Danger of Explosion. In the course of self-condensation of esters by alkali ethoxides, if experiments are not conducted according to instructions there is danger of an expln. For example, air should never be allowed to enter the reaction flask contg the finely powdered metal as an expln may occur when ale is added. The procedure of Brändström (Ref) is a simplified method for the prepn of β-keto esters, the reaction is more easily controlled and gives the same yield as the older method
Ref: A.Brändström, ActaChemScand **4**, 1608-10

(1951) (in Engl) & CA **45**, 7960-61 (1951)

Danger of Handling Explosives. Any compd or mixt which may be potentially expl should be regarded with suspicion and handled with more than usual care. Such a compd or mixt is one whose heat of formation ($\triangle Q_f = -\triangle H_f$) is smaller by more than 100-200 cal/g than the sum of the heats of formation of its products. When any doubt exists about an expl system, its potential energy can often be calcd

Expls are chem compds or mixts of compds in the form of solids, liquids or gases which are heat or shock-sensitive to varying degrees, and from such influences can undergo very rapid self-propagating decompn with the liberation of heat and the development of a sudden pressure effect. The hazards involved in working with a potentially expl system are directly proportional to the rate with which energy is released and to the amt of gas evolved. Thus, since rates are more difficult to predict than potential energy content, caution is always advised. The vol of gas, of course, can be readily calcd if this energy is known. Despite the ability to calculate a "hazard index", it is most helpful to have structural & other indications to suggest props & possible hazards. Certain groups (such as $-ONO_2$, $-NHNO_2$, $=N.NO_2$, $-N_3$, NO_2, -NO, -N=N-, -O-O-, -CN, =NOH, -O-, etc) when present in a molecule either confer or enhance expl props and may increase sensitivity

Thus, when a new expl compd is first prepd in the lab it is essential for max safety that a series of preliminary tests be conducted to determine its order of sensitivity (See *Physical Tests for Determining Esplosives and Other Properties*, Vol 1, pp VII-XXVI). These tests are designed to determine whether the compd can be handled at all, and if so, with what care it must be treated. It is important that much less than 1g (ca 0.2g) of solvent-wet or dissolved new expl be prepd, because for some expls even 1g is more than sufficient to kill an unprotected person. For this reason, the tests referred to above should be conducted in the order:

a)match flame sensitivity
b)impact sensitivity
c)friction sensitivity (qualitative, a few milligrams rubbed in a mortar)
d)stability (expln temp, heat & vacuum tests)
e)performance tests

Since expls vary widely with respect to their sensitivities & stabilities, this information must be considered in their study, use, handling, storage & transportation

Because a wide variety of expl mixts or muliti-component HE mixts can be prepd, the possibility of reactions between the ingredients must always be considered. Operations requiring the grinding and or blending of expl mixts must be conducted with extreme care and then only after some indication of the sensitivity of the material is known. Grinding & blending must always be done behind a *barricade* (qv)

Some expls belong to a group which may be less hazardous to handle than others. These include *igniter powders, some pyrotechnic compns, proplnts,* and *high energy electric initiators*

Primary expls (initiating) are used to start the train of reactions which results in the deton of secondary HE's or of the functioning of ammo. These expls are the most sensitive of all chemical components in military ammo, hence, the most hazardous. Primary expls can be ignited by an elec heater or spark, heat from mechanical friction, a free flame, or by an RF (radio frequency) signal. The ideal primary expl is a single compd, since this affords greatest control of its characteristics. Unfortunately, such a compd is not always available so that mixts must usually be used

Because primary expls are so sensitive (very small amts release appreciable quantities of energy and significant volumes of gas in incredible short times), not more than a few milligrams of a new compd should be synthesized. Before the various components are mixed, one should have a thorough knowledge of the sensitivity, stability, and reactivity of each, not only individually, but also in contact with the others

Finally, one should always follow general safety rules for the handling of all expls as well as special safety rules for the prepn & handling of experimental new HE's & HE compns

Many refs are available on this subject. The following is a selected list of a few which may be consulted for further details

See also Dangerous (Hazardous) Chemicals and other Materials

Refs: 1)Marshal **2** (1917), 422-39 2)Davis (1943), 1-27 3)Anon, "Military Explosives", TM **9-1910**/TO **11A-1-34**, US Depts of the Army

and Air Force, Washington, DC (1955), 5-15 4)Sax (1957), 152-64 5)Cook (1958), 1-21 6) P.Rochlin, "Safety Handbook", Expls Lab FRL, PicnArsn, Dover NJ (1959) 7)Anon, "AMC Safety Manual", AMC **385-224**, Headquarters, US Army Materiel Command, Washington, DC (1964) 8)Urbański **2** (1965), 122 (Safety rules in NG manuf) & 406 (Safety rules in a NC plant)

Dangerous (Hazardous) Chemicals and Other Materials. Hazardous conditions in lab & plant practices dealing with chemicals are often created as a result of new or unfamiliar operations. Many compds are always dangerous, but many others, although relatively inert by themselves, react with varying degrees of violence when combined with other chemicals. Many common lab chemicals are poisonous and all should be handled with care. Toxic substances can enter the body in several ways including the lungs, digestive tract & skin. Entry via the skin is especially dangerous as undamaged skin is permeable, particularly by fat-sol substances

Following is a list of compds, mixts, & reactions which are or maybe hazardous. Where available refs to original literature or to CA should be consulted for further details. This list cover the period 1937-57 for the most part. No attempt has been made at completeness nor were sources other than open literature searched. Only those compds, mixts or reactions believed to be of general interest to workers in expls and related items are included. A thorough search of the literature should always be made before preparing or working with any chemical or special mixt which may be hazardous. This list is based principally on the report of Rochlin and of memoranda of W.R.Tomlinson, Jr (now deceased) who was formerly Chief, Explosives Research Section, PicnArsn (See Ref 117)

Acetic acid (glacial) will ignite at elevated temps (flash point 40°C). It will also irritate the skin & mucuous membranes of the mouth & throat. See also Acetone and Derivatives, Vol 1, pp A25 -L to A27-R

Acetic anhydride reacts violently with some hydroxyl compds. It is known to explode in contact with ethylene glycol & $HClO_4$ (Ref 55). See also Acetic Anhydride and Derivatives, Vol 1, pp A29-R to A31-L

Acetone decomposes violently when brought in contact with H_2SO_4/HNO_3 mixtures such as are used in nitration reactions. This is especially true when the reaction is partially confined by dropping the acetone into a narrow-mouthed bottle containing the mixed acids. Thus, acetone should not be used to degrease vessels used as mixed acid reactors (Ref 116). See also Acetone and Derivatives, Vol 1, pp A33-R to A45-L

Acetone-water solutions are flammable over a very wide range. A 5% solution will flash at 60°C if an ignition source (a match!) is present. The higher the percent of acetone present, the lower the flash point, until pure acetone is reached with its flash point of -17.8°C

Acetylene, under certain conditions, forms highly explosive compounds with copper, silver, and mercury. Copper acetylides are easily detonated by heat or shock. Silver acetylide (formed in the determination of acetylene by absorption in $AgNO_3$ solution) is explosive when dry. See also Acetylene and Derivatives, Vol 1, pp A58 -L to A68-R

Acetyl nitrate is a powerful nitrating agent which can explode spontaneously (Ref 37). See also Vol 1, p A84-R

Acetyl peroxide (solid) explodes on shock. It should be prepared and used without interruption and should be handled by remote control (Refs 54 & 71). The commercially available 30% solution in dimethyl phthalate will not give crystalline acetyl peroxide under ordinary circumstances. The solution has a flash point of 45°C. Heating it above 50°C causes orderly, but fairly rapid decomposition (Ref 62)

Alkali metals. The principal hazards involved are: 1)explosions or fire resulting from contact with water, chlorinated hydrocarbons, or other reactive agents; 2)fires resulting from exposure to air; 3)personnel injury from direct contact with hot alkali metals; and 4)caustic soda burns from the residue of a Na-H_2O reaction. Storage areas must be dry. Protective clothing must be worn (Ref 105)

Alkali metals (Na or K) and *metallic hydrides*, on contact with water, produce hydrogen and sufficient heat to ignite the gas with explosive rapidity. Waste scraps of sodium should be added in small proportions to a high-boiling alcohol, such as propanol or butanol, and safely disposed of after all action ceases. Nonaqueous fire extinguishers such as dry soda ash are the only kind that should be used on an alkali metal fire. See also Alkali and Alkaline Earth Metals, Vol 1, p A125-L

Aluminum Powder with ammonium persulfate and water may cause an explosion (Ref 110, p 30). Mixtures with CH_3Cl, CCl_4, or CCl_4-$CHCl_3$ may also explode. With CH_3Cl, aluminum powder may form spontaneously combustible $Al(CH_3)_3$ (Ref 38). Finely divided *aluminum* is explosive (following Zn, Mg, and Ti in the scale of explosive dusts). Al & Mg react violently with chlorinated hydrocarbons, with Na_2O_2 and $AlCl_3$, and with others (Refs 90 & 91). See also Vol 1, pp A142-L to A144-R

Aluminum sheet is not, or is only slightly, attacked by CCl_4. Certain mixtures of a nonexplosive liquid and a nonexplosive solid are sensitive to detonation. In general, an explosion is to be anticipated when a strongly exothermic reaction may occur between intimate mixtures, especially if one of the components is a liquid or gas (Ref 67a)

Aluminum chloride, $AlCl_3$(I), added to *nitrobenzene* (II) containing 5% *phenol* (III) rose in temperature and exploded violently. Heating nitrobenzene with either I or III causes no explosion, but at 120°C the three together react violently and form a tar. This shows the danger of working with I, II, and an oxidizable organic material (Ref 89). See also Vol 3, p C236-L

Aluminum chloride-nitromethane catalyst is believed to have exploded at a temperature below 10°C (Ref 52)

Aminoguanidine nitrate in water solution exploded violently while being evaporated to dryness in vacuo on a steam bath. The compound had previously been prepared without explosion. The possibility of explosion is not mentioned in Organic Syntheses, although guanidine *nitrate* and *nitroguanidine* are classed as explosives. In working with guanidine derivatives, nitrates should not be used (Ref 107). See also Aminoguanidine and Derivatives, Vol 1, pp A210-L to A214-L

Ammonia and *silver ions* may produce an unstable product which detonates violently, eg, when the solution is stirred (Ref 110, p 29). See also Ammonia, Vol 1, pp A296-L to A306-L

Ammonium azide, solid. Decomposition, explosion, and combustion data (Ref 99). See also Vol 1, p A521-L

Ammonium nitrate decomposes exothermally above 160°C producing gaseous decomposition products. With large quantities or in a confined space (such as a closed container) this reaction develops into a violent explosion. Mixtures of AN and oxidizable substances such as metal powders and organic materials are liable to a spontaneous exothermic reaction and can explode (Refs 48, 67b & 84). See also Vol 1, pp A311-L to A340-R

Ammonium periodate, NH_4IO_4 can explode (Ref 73)

Aniline is a blood poison and may enter the body through the skin, the lungs, and the digestive tract. It has no appreciable local irritating action but illness can result from inhalation of vapors or from absorption through the skin. Thus, aniline should be used only in the hood so as to keep vapors out of the air. Many derivatives of aniline (such as methyl violet, nitraniline, dimethylaniline, diaminobenzene) have similar poisonous properties. (See also Vol 1, pp A406-L to A419-R

Barium azide, BaN_6, is explosive despite its small heat of decomposition (13.7 kcal). It is slightly sensitive to shock, but extremely sensitive to friction. It does not have the burning properties of a primer, but large quantities of it burn explosively. It is very slightly toxic, but it must be kept from contact with strong acids which liberate HN_3 (Refs 25, 26 & 77b). See also Vol 1, pp A523-L to A524-R

Benzene is both flammable and highly toxic, and it should not be used if another solvent can be substituted. Its vapor is considerabley heavier than air so that it can travel along floors and benches. Benzene and its homologues should never be poured down the sink. See also Benzene and Derivatives, Vol 2, pp B41-R to B52-R

Benzoyl peroxide can detonate. Dry Bz_2O_2 and its solution in chloroform explode rapidly above 95°C (Ref 58). The compound should be purified by precipitating with methanol from a chloroform solution (Ref 46) rather than by the method given in Org Syntheses Coll Vol 1 (1941), p 432, which is dangerous

Bismuth and $HClO_4$. See under Perchloric Acid and in Vol 2, p B161

Boron and *Boron Hydrides.* See Vol 2, pp B251-L to B257-L

Brominating agents (solid) are expl. Any reagent forming free Br radicals should first be tested on a small scale (Ref 80). See also Vol 2, p B305-L

Bromine (liquid) causes immediate burns on the skin and mucous membrane. Bromine should be handled only in the hood. Goggles and rubber gloves should be worn. See also Vol 1, p B305-L

Bromine trifluoride, BrF_3, and some metallic halides (NH_4F, NH_4Cl, KBr, KI) react very

violently (Ref 57). See also Bromine Fluorides, Vol 2, pp B305-R to B306-L

Carbon disulfide is poisonous, endothermic, and can be detonated by shock. It has a very low flash point and a high vapor pressure at room temperature. Mixed with air or oxygen it forms a violently explosive mixture which can be fired by flame or shock or by catalytic agents such as iron rust (Ref 110, pp 20 & 78). See also Carbon Disulfide and Derivatives, Vol 2, pp C60-R to C61-R

Carbon monoxide is highly toxic and is specially treacherous because it is both odorless and tasteless. It is present in smoke, in the gases produced by a burner which has flashed back, in a luminous flame, or a flame in contact with a cold surface. Most commonly it is found in the exhaust of an internal combustion engine

Carbon monoxide is also produced in the smoke of cigars, cigarettes, and pipes, the quantity depending on the velocity with which the tabacco is consumed. Rapid, nervous smoking, especially when combined with inhalation of the smoke favors intoxication with CO (Ref 110, p 109). See also Vol 2, p C62-L

Chlorates and *Chlorate Compounds* See Vol 2, pp C181-L to C202-L

Chloric Acid. See Vol 2, pp C209-R to C210-R

Chlorine and *hydrogen* or various *hydrocarbons* can ignite spontaneously (Ref 17). Chlorine and *gaseous formaldehyde* can react explosively under certain conditions at temps as low as 100° (Ref 3). See also Chlorine and Compounds, Vol 3, pp C242-L to C244-R

Chlorine dioxide in liq or gaseous state can explode (Refs 77 & 78). Chlorine dioxide and *sulfur* in contact with each other may result in an explosion (Ref 27). See also under Chlorine Oxides, Vol 3, p C243-R

Chromates, Dichromates, Trichromates & Tetrachromates. See p C274-Lff

Chromic Acid. See p C298-R to C299-L

Chromium and Its Compounds. See pp C300-Lff

Diazonium compounds and *sodium sulfides* react vigorously and are known to explode. Similar explosive reactions have been observed with H_2S, also (Refs 40 & 41)

Dimethyl sulfoxide decomposes violently when in contact with a wide range of acyl halides and related compounds, such as cyanuric, acetyl, benzoyl, benzenesulfonyl, thionyl, and phosphoryl chlorides and PCl_3. DMS should be used with caution as a solvent in exploratory reactions (Ref 109)

2,4-Dinitrobenzenesulfenyl chloride is an explosion hazard both in its preparation and its use. Because of its explosive nature, temperatures above 90°-100°C should be avoided in its preparation. Care should be exercised in its storage. The reagent may be destroyed by addition to several volumes of 20% NaOH and washing away with water (Refs 47 & 67). See also under Benzenesulfenic Acid and Derivatives, Vol 2, p B61-L

2,4-Dinitrochlorobenzene. An explosion occurred while the compound was being distilled at 1-mm pressure (Ref 75). See also under Chlorobenzene and Derivatives, Vol 3, p C249-R

Ether. Diethyl ether is very flammable at room temperature. It has a high vapor pressure, low flash point, and low ignition temperature. Mixtures of ether vapor with air or oxygen are explosive within a wide range. Never use a flame in the presence of ether. When stored in contact with air, ether slowly absorbs oxygen, forming peroxides which are very explosive. Sunlight hastens the reaction so that ethers should be placed in containers which exclude light. Peroxides are also formed in ethers other than diethyl ether and in substances such as dioxane and unsaturated hydrocarbons (Ref 110, p 25ff)

Because of these peroxides, distilling of dry ether always involves the risk of dangerous explosions (Refs 7, 14, 20, 32, 33 & 45). Even when present in only small quantities, Et_2O_2 may pass over with the ether if distillation is conducted at a rapid rate. Water increases the probability of the peroxide remaining in the residue. If sulfur is also present, the residue is violently explosive, even below 100°C (Refs 8 & 9)

Ether-chloroform mixtures (1:1) burn with a smoky flame and evolve $COCl_2$; a 1:2 mixture is ignited with difficulty (Ref 12)

Ethylene ozonide is highly sensitive to shock. Pouring from one vessel into another has led to violent explosion (Ref 87)

Fluorine is extremely reactive and hazardous. At 20°C, mixtures of F and ClO_2 react explosively to form chlorine and oxygen (Ref 34)

Formaldehyde and NO_2 mixtures are not explosive below about 160°C. At about 180°C, the reaction becomes explosive (Ref 63)

Hydrazine vapor (pure) when sparked at 100° explodes, and a yellow flame accompanies the

decompn. The products are ammonia, hydrogen and nitrogen (Ref 21)

Hydrazine perchlorate begins to decompose at 145 °C and is complete by 230°C if heating is careful. Otherwise, a strong deflagration occurs. Shock or friction causes violent detonation (Ref 61). Its *Ni salt,* $Ni(N_2H_4)_2(ClO_4)_2$, is an extremely dangerous substance (Ref 79).

Hydrogen chloride gas is a primary irritant with a destruction action on mucous membranes

Hydrogen cyanide and other *cyanides* and *cyanogens* are well-known highly poisonous compounds. Poisoning proceeds very rapidly causing a reflex cessation of the heart action so that first aid generally comes too late. See also Cyanides and Cyanocompounds in Vol 3

Hydrogen fluoride is a poison with a strongly irritant action on the skin and the mucous membranes. The concentrated acid produces open wounds which heal slowly. *Soluble fluorides* are toxic. Particular care should be takes that no HF gets under the nails as this causes painful and prolonged inflammation

Hydrogen peroxide detonates readily in the pure state, but will not detonate in aqueous solutions containing less than 94% H_2O_2, although aqueous solutions will decompose explosively under catalytic influence (Ref 63a). H_2O_2 causes burns which should be treated with hot water or with a solution of sodium thiosulfate. In concentrations above 50%, H_2O_2 can be a fire hazard in the presence of easily oxidizable material. Mixed with organic material (eg, glycerine) it is unusually explosive, especially when contaminated

Hydrogen sulfide is very poisonous. Although low concentrations have a very disagreeable odor, after a relatively short exposure, or in higher concentrations, the sense of smell rapidly becomes temporarily atrophied

Lithium aluminum hydride is not safe for drying methyl ethers (Ref 86). *Tetrahydrofuran* can cause fire when used as a solvent for $LiAlH_4$ (Ref 93). Extreme caution should be used in reducing any fluorinated compound with $LiAlH_4$. Violent reactions have been observed with two different fluoro compounds (Refs 76 & 95). Excess *trimethylamine,* NMe_3, reacts with an ethereal solution of $LiAlH_4$ in vacuo at -50°C to form a white addition compound which is slightly soluble in ether, insoluble in benzene, and spontaneously flammable in air (Ref 98a)

Magnesium perchlorate may be used as a drying agent and as a quantitative absorbent of vapors of certain polar compounds. Caution must be taken to exclude flammable materials, mineral acids, or substances liable to hydrolyze them. Serious explosions have resulted from using magnesium perchlorate for drying an organic liquid which had been in contact with sulfuric acid and had been inadequately washed. The compound used in drying unsaturated hydrocarbons exploded on heating to 220°C (Refs 18, 31 & 96)

Manganese heptoxide, Mn_2O_7, which explodes at 70°C, can form in $KMnO_4/H_2SO_4$ mixtures. A few drops of water may generate sufficient heat to initiate the explosion. Contact with organic matter such as stopcock grease in a separatory funnel has had the same effect (Ref 51)

Mercuric nitrate and organic compounds reacted vigorously and exothermally at room temperature without exploding during attempts to remove sulfides (Ref 53)

Mercury and *ammonia* gas are known to explode (Ref 50a)

Methyl azide. A serious explosion occurred in condensation with dimethyl malonate in the presence of sodium methylate (Ref 69)

Methyl bromide at certain concentrations can inhibit explosions of mixtures of methyl chloride and air (Ref 13)

Methylene chloride cannot be dried over sodium owing to formation of explosive compounds (Ref 33)

Methyl nitrate is the most explosive of the nitrate esters (Ref 100)

Nitric acid (Concd) etches the skin causing a yellow stain which remains for a considerable time. The vapor is a serious respiratory poison. Use of a face shield or goggles, protective clothing, and adequate ventilation is imperative

Nitric acid is also a powerful oxidant, causing fire and explosion hazards when mixed with combustible materials (Refs 59 & 60). Mixtures of the concd acid with an organic substance such as acetone, acetic anhydride, alcohol, or aniline, may detonate. Traces of an organic substance with HNO_3 and mercury may form a highly explosive fulminate

Nitrobenzene and HNO_3 form dangerous sensitive mixtures (Sprengel Explosives). *Nitrobenzene* and *liq* N_2O_2 form a powerful and sensitive high explosive. See also under Benzene and

Derivatives, Vol 2, pp B45-R to B46-L
m-Nitrobenzoic acid. Its prepn from benzene, fuming HNO_3 & H_2SO_4 and heating of the mixture above 100°C leads to an explosion (Ref 34a). See also under Benzoic Acid and Derivatives, Vol 2, p B71-R
o-Nitrobenzoylchloride and *o-Nitrophenacetyl chloride* may explode if heated above 100°C (Refs 43 & 44). See also Benzoylchloride and Derivatives, Vol 2, p B89-R to B90-L
Nitro compounds are very toxic. They may enter the body via the lungs, thru the skin, or with food. Recovery from poisoning with nitro compounds is slow. Protection against such poisons is based on cleanliness, ventilation, protective clothing, goggles and respirators. These statements apply equally to *nitrobenzenes, nitromethanes,* and *nitrochlorobenzene*
Nitroform, $CH(NO_2)_3$, and *2,2,2-trinitroethanol* must each be distilled carefully as they may explode. Temperature must be carefully controlled in all operations (Ref 68)
Nitrogen chloride, NCl_3, when frozen in liquid air or thawed in vacuo explodes spontaneously due to spontaneous decomposition of NCl_3 vapors. Admixtures of air hinder this decomposition (Ref 19)
Nitrogen oxide fumes are most hazardous as they can develop unexpectedly (eg, when a bottle of HNO_3 breaks, or when HNO_3 comes into contact with organic substances such as sawdust). Any atmosphere in which nitrogen oxides can be smelled must be considered dangerous. Reactions in which nitrogen oxides may be produced must be performed in a hood. Nitrogen tetroxide, N_2O_4, reacts whth aliphatic ethers very vigourously, and sometimes with explosive violence (Ref 80a)
Nitromethane is only moderately sensitive to explosion by shork or by heat and pressure, and it need not be considered dangerous if used with precautions taken with flammable liquids (comparable to kerosine). However, it can explode at elevated temperature and pressure. Flash point 35-37°C (Refs 16, 55a & 77a). Sodium & potassium salts of nitromethane are easily detonated and are powerful explosives (Ref 1)
Nitroparaffins are sensitive to *alkali,* and many sensitive combinations ranging from addition complexes, through metal salts, to the very dangerous fulminates, can be formed depending on the base involved. All of these compounds are usually more sensitive than the original nitroparaffin

Nitroparaffins such as *nitromethane, nitroethane, nitropropane,* are mild oxidizers and should not be heated with easily oxidized hydrocarbons. With water, they form nitronic acids as tautomeric forms, which form explosive salts with inorganic bases (Ref 83)
Nitrophenol on filter paper deposited in a waste basket can give rise to an unexpected explosion
Nitrosyl chloride, NOCl, the reddish-brown gas dissolved in aqua regia is a very reactive reagent. It reacts with most of the elements and with many of the compounds, and is very corrosive in the presence of water (Ref 115)
Nitrosyl perchlorate, $NOClO_4$, is generally considered stable at room temperature and,by itself, should be completely safe. Decomposition begins just below 100 °C; above 100°C (115°-120°C) low order explosions occur (Ref 103)
p-Nitrotoluene - H_2SO_4 mixtures explode when heated to 160°C
Perchlorates in mixtures with reducing materials are more stable than the corresponding *chlorates* and are not as subject to dangers from the presence of free acids. Nevertheless, it should not be assumed that all perchlorate mixtures are safe. A perchlorate mixture can be initiated by friction, impact, or spark at energies which cannot be considered safe; some of these mixtures are extremely dangerous (Ref 42)
Perchloric acid, $HClO_4$, is an extremely powerful and dangerous oxidizing agent which may react explosively with reducing agents and organic compounds. It should be used with extreme caution and in the smallest quantities possible; personnel should wear protective clothing, gloves, and goggles or a face shield, and should always work behind a barricade

Perchloric acid should always be used in a hood made from noncombustible material, preferably a hood reserved exclusively for this purpose. Before using the acid, the exhaust fan should be run for a few minutes to remove all loose particles of dust, etc from within the hood. Never use $HClO_4$ on or near wooden benches or tables. Always use tongs to manipulate a crucible or a dish containing the acid

Solutions of perchloric acid should be heated with an electric hot plate or a heating mantle, never on an oil bath. Organic matter

in samples should first be oxidized with concd HNO_3 before adding the $HClO_4$ (CA **32**, 882, 6463). Never heat $NClO_4$ with sulfuric acid or any other high boiling acid as dehydration will produce the explosive anhydrous $HClO_4$

Combustible materials (filter paper, rags, etc) which have been soaked with aqueous $HClO_4$ and dried should be treated with extreme care as they might be ignited by impact, friction, or heat; the condensed vapor will form explosive mixtures with ammonia or with HNO_3

Never use rubber or cork with apparatus when working with $HClO_4$. Use an all-glass apparatus, and do not use stopcock grease. Thoroughly clean all apparatus which has been in contact with $HClO_4$

The 60-70% commercially available aqueous solution is stable and can be stored safely. However, avoid storage of more than a 1-lb bottle in the laboratory. At concentrations above 85% the acid is liable to spontaneously explode

Anhydrous perchloric acid is stable only at liquid air temperatures; eventually, it decomposes violently & spontaneously near 20°C (Ref 49); in contact with oxidizable material it explodes at once

Discolored (or contaminated) acid should be poured carefully into cold water and flushed to discard

Perchloric acid reacts with *bismuth* to form a spontaneously explosive compound (Ref 4)

Perchloric acid esters are also violently explosive. An *alcoholic solution* of $HClO_4$ should never be heated unless considerable water is present, as it may explode (Refs 5 & 6)

Perchloryl fluoride, $FClO_3$, is a powerful oxidizing agent and should be handled accordingly. It is stable by itself, but a mixture with methanol ignited and exploded when sodium methylate was added (Ref 114)

Performic acid, HCOOOH, detonated at -10° in a glass distilling app. All precautions had been taken to insure cleanliness but the compd is particularly sensitive to shock & dust particles (Ref 82)

Permonosulfuric acid solutions, H_2SO_5, *in organic media* (some lower alcohols and acetonitrile). Explosive reactions are possible with sec and tert alcohols and generally in cases of too high concentrations of the peracid (Ref 11)

Peroxides are sometimes highly explosive. They tend to form from compounds having an ether group (butyl ether, dioxane, glycol-ethers). The hazard involved in distilling the compounds boiling above 100°C is believed to be comparatively small because the high temperature decomposes the peroxide as fast as it is former. However, under vacuum distillation, the temperature may be low enough to make the distillation dangerous. In general, any ether-like compound that gives a strong brown color with aqueous 10% KI solution should not be distilled without special treatment and precautions (Refs 87 & 92, pp 121-22)

Phenols are highly toxic. They are strongly corrosive to the skin and the mucuous membranes. If the skin or the clothes are contaminated with phenols, the soiled clothes should be removed at once, and the skin flushed with warm water and washed with dilute alcohol. When working with phenols, goggles should always be worn.

Cresols are similarly poisonous, but are less toxic than phenol. *Napthols* are much less irritant and toxic

Phenyldiazosulfide is a red solid which explodes when air dried. The *p-nitrophenyl derivative* is only slightly less sensitive (Ref 72)

Phosgene, $COCl_2$, is a lung irritant which may be inhaled for some time without producing perceptible reactions so that poisoning is difficult to diagnose in the early stage

Phosphorus trichloride. An explosion occurred in the preparation of acetyl chloride from acetic acid and PCl_3, the AcCl being distilled with a free flame. PH_3, is believed to have been the cause: PCl_3 reacts with water to give phosphine, which ignites spontaneously (Ref 15)

Picrates. Melting points over 210° should not be taken of picrates in the usual liquid bath apparatus. Use a melting point bar such as the Dennis bar or a Fisher-Johns apparatus (Ref 88)

Potassium dichromate, $K_2Cr_2O_7$, mixtures with NH_4NO_3 or organic nitro compounds are not explosive nor flammable. Mixtures of *potassium permanganat,* $KMnO_4$, with these substances burn easily when subjected to flame, heat, concd H_2SO_4, or shock but not enough gases are liberated to cause an explosion (Ref 10)

Pyridinium perchlorate formed during purification of pyridine with $HClO_4$ is known to explode violently (Ref 66)

Silver nitrate crystals obtained from HNO_3 solution in reclaiming AgCl residues were rinsed

with ethanol to remove adhering dilute HNO_3 and other impurities. About ¼-lb of alcohol-moist crystals exploded violently when touched with a porcelain spatula. The explosion was caused by *silver fulminate*, AgONC, produced by reaction of the $AgNO_3$ with HNO_3 and ethanol (Ref 28). Violent explns of *ammoniacal silver solns* have been reported (Ref 85).

Silver nitrosoguanidine is an explosive compound (Ref 70)

Silver perchlorate, $AgClO_4$, crystallized from benzene was reported to have exploded with extraordinary violence (Ref 24). This compound has also exploded while a filter cake of it was being pulverized in a mortar (Ref 108)

Sodium covered with sodium methylate or butylate, formed in the presence of toluene exploded when thrown into water. Clean sodium in water merely sizzled (Ref 64)

Sodium chlorate is a very powerful oxidizing agent, and its expl props (Ref 81) have caused some spectacular fires. It has an extremely high rate of burning, and the ease with which ignition can occur makes it mandatory that any clothing or other object splashed with sodium chlorate solutions be thoroughly washed with water. Drying of the material with plenty of ventilation will ensure that all of the chemical has been removed (Refs 81 & 116). See also Vol 2, pp C197-R to C200-R

Sulfur and *iron* can ignite spontaneously (Ref 17)

Concentrated sulfuric acid and *oleum* cause burns on the skin and have a destructive action on all organic matter. Concd H_2SO_4 sometimes reacts violently with many organic substances, nitrates, sulfides, chlorates, perchlorates, halogenides, carbides, and many metals. Fe sulfate & H_2SO_4 have detonated (Ref 74)

Concd H_2SO_4 should never be used in a desiccator for drying an explosive material unless it is certain that the two will not react if mixed. Accidental spills have been known to cause explosions

tert-Butylperbenzoate, vacuum distilled above 100°C has resulted in an explosion (Ref 87)

Tertiary hydroperoxides are usually relatively stable. However, the reactions leading to their formation are treacherous, and all safety precautions must be used to prevent serious explosions (Ref 56)

Tetrahydrofuran forms explosive peroxides comparable to ether. Al least one explosion during distillation of THF has been reported (Refs 65 & 101). See also *Lithium Aluminum hydride* in this section

Tetranitromethane forms with hydrocarbons (such as benzene and toluene) one of the most brisant, destructive mixtures known. TNM is very poisonous, affecting the membranes of the eyes and nose. It is always formed as a by-product in nitration of aromatic hydrocarbons and always accompanies crude TNT (Refs 30 & 35).

Titanium. There is a danger from explosions with titanium in contact with HNO_3, especially the red fuming HNO_3 (Refs 94, 102 & 106)

Toluene is less toxic than *benzene*, but more dangerous than *xylene* with respect to its toxicity

Trichlorethylene, often recommended for use in place of CCl_4, is about half as toxic as is often used instead of it. Some persons even grow adicted to it (Refs 80b & 110, p 84)

Trinitrotoluene. Pb and Fe produce explosive substances with TNT in the presence of HNO_3. These can be ignited by heat, shock, friction, or contact with concentrated nitric and sulfuric acids (Ref 85a)

TNT and *aniline* will explode spontaneously when mixed

Vinylidene chloride reacts rapidly with oxygen from air to form a violently explosive peroxide (Ref 50)

Xylene, although toxic, is less dangerous than *toluene* or *benzene*. However, it is more irratating to the skin than is benzene

Many lists of common, dangerous & hazardous chemicals have been prepd and some books and pamphlets are available for supplementary reading on the subject. Some of these are included in the following refs

See also Danger of Handling Explosives

Refs on Dangerous Chemicals: 1)N.Zelinsky, Ber **27**, 3406-07 (1894) 2)Dr A.Langhans, "Explosionen, die Man nicht erwartet" (Unexpected explosions), SS **25**, Suppl, 65-70 (1930) 3)R. Spence & W.Wild, JChemSoc **1934**, 1588-93 (Thermal reaction between Cl & gaseous formaldehyde) 4)D.G.Nicholson & J.H.Reedy, JACS **57**, 817-18 (1935) (Expl reaction of bismuth with perchloric acid) 5)O.Hackl, ZAnalChem **107**, 385-87 (1936) & CA **31**, 2007 (1937) (Expln danger of perchloric acid) 6)J.Meyer & W.Sporman, ZAnalChem **107**, 387-88 (1936) & CA **31**, 2007 (1937) (Perchloric acid behavior) 7)E.C. Williams, Chem & Ind **1936**, 580-81 & CA **31**, 4123

(1937) (Expln of ether) 8)E.F.Degering, JChem-Educ **13**, 494 (1936) (Expln hazards from isopropyl & other ethers) 9)A.F.MacCulloch, Chem & Ind **1936,** 964 & CA **31**, 8928 (1937) (Explns arising from storage of ethers) 10)I.F.Blinov, ZhKhim-Prom **14**, 1151-53 (1937) & CA **32**, 781 (1938) (Explosiveness of $KMnO_4$ or $K_2Cr_2O_7$ with AN or org nitrocompds) 11)G.Toennies, JACS **59**, 554-55 (1937) (Expln of permonosulfuric acid & ethyldimethylcarbinol, isoamyl alcohol, or methylisobutylcarbinol all while in a freezing mixt) 12)D.B.Dott, PharmJ **140**, 357 (1938) & CA **32**, 3909 (1938) (Flammability of ether-chloroform mixts) 13)C.Randaccio & T.Giavarrini, AnnChim-Appl **28**, 254-58 (1938) & CA **32**, 9503 (1938) (Expln limits for mixts of methyl chloride, methyl bromide & air) 14)J.Tandberg, TekTid, Reprint **1938**, No 24, 4pp & CA **32**, 9503 (1938) (Expln of ether due to presence of peroxide) 15)R.D.Coghill, JACS **60**, 488 (1938) 16)D.S. McKittrick et al, IEC, AnalEd **10**, 630-31 (1938) (Hazards in the use of Nitromethane) 17)J.E.E. Blow, Fire **31**, 80-82 (1938) & CA **33**, 1940 (1939) (Spontaneous ignition of finely divided metals & hydrocarbons) 18)M.J.Stross & G.B. Zimmerman, IEC, NewsEd **17**, 70 (1939) & CA **33**, 1940 (1939) (Hazards in the use of magnesium perchlorate) 19)A.Ya.Apin, ByullVsesoyuz-KhimObshch im D.I.Mendeleeva **1939**, No 3-4, 40-41; Khim Referat Zhur **1939**, No 8, 17 & CA **34**, 5009 (1940) (Expl decompn of nitrogen chloride) 20)F.Eckhardt, WochschrBrau **56**, 338-39 (1939) & CA **34**, 7608 (1940) (Expln danger of ether contg peroxides) 21)C.H.Bamford, TrFaradSoc **35**, 1239-46 (1939) (New observations on Hydrazine) 22)H.F.Coward & G.W.Jones, "Limits of Inflammability of Gases and Vapors", BurMinesBull **279**, 146pp, 3rd ed (1939) 23)Associated Factory Mutual Fire Insurance Companies, "Properties of Flammable Liquids, Gases and Solids", IEC **32**, 880-84 (1940) (A table of pertinent data as to fire hazard of nearly 400 industrial liquids, gases and solids) 24)S.R. Brinkly, Jr, JACS **62**, 3524 (1940) (Instability of Silver Perchlorate and deton of its addn comp with ethyl alcohol) 25)P.L.Gyunter et al, SbornikStateiTeoriiVV (Moskva) **1940**, 66-79; KhimReferatZhur **1940**, No 10-11, 120 & CA **37**, 1270-71 (1943) (Thermal decompn of Barium Azide) 26)P.L.Gyunter et al, Ibid, 120-21; KhimReferatZhur **1940)** No 10-11, 120-21 & CA **37**, 1271 (1943) (Decompn of BaN_6 by x-rays) 27)F.Taradoire, BullFr **8**, 860-64 (1941); ChemZtr **1942II**, 1325 & CA **37**, 5669 (1943) (Action of ClO_2 on sulfur) 28)J.P.Tully, C & EN **19**, 250 (1941) & CA **35**, 3092 (1941) (Expln of Ag nitrate crysts washed with alc) 29)F.W.Brown, "Theoretical Calculations for Explosives. I. Explosion Temperatures and Gaseous Products and the Effects of Changes in Carbonaceous Material", BurMinesTechPaper **632**, 21pp (1941) 30)A. Stettbacher, Tech-IndSchweizChem-Ztg **24**, 265-71 (1941) & CA **36**, 4339 (1942) (Tetranitromethane-hydrocarbon mixts, the most brisant expl mixt known) 31)P.M.Heertjes & J.P.W Houtman, ChemWbl **38**, 85 (1941) & CA **36**, 5349 (1942) (Expln of magnesium perchlorate) 32) (?).Nestle, Farbe u Lack **1941**, 102; ChemZtr **1941II**, 1422 & CA **38**, 1880 (1944) (Danger of peroxide contg ether) 33)K.Würth, Farbe u Lack **1941**, 58; Farben-Ztg **46**, 547 (1941); ChemZtr **1941II**, 2354 & CA **38**, 1880 (1944) (Danger in use of ether contg peroxide and proposal for subsn by CH_2Cl_2) 34)H.Schmitz & H.J.Schumacher, ZAnorgChem **249**, 238-44 (1942) & CA **37**, 3679 (1943) (Reaction of fluorine & chlorine dioxide) 34a)T.Nishi & H. Toki, JSocChemInd (Japan), Suppl binding, 37-38 (1942) & CA **44**, 11099 (1950) (Prepn of m-Nitrobenzoic Acid and its expln when reaction mixt was heated above 100°) 35)H. Muraour, Chim & Ind (Paris) **47**, 477 (1942); ChemZentr **1942II**, 1426 & CA **37**, 5591 (1943) (Tetranitromethane & toluene mixts) 36)F.W. Brown "Theoretical Calculations for Explosives II. Explosion Pressures ", BurMines TechPaper **643**, 26pp (1942) 37)J.P.Wibaut, ChemWbl **39**, 534 (1942); ChemZtr **1943I**, 384 & CA **38**, 3129-30 (1944) (Spontanesou expln of Acetyl Nitrate) 38) C.G.Clogston, Underwriters' Labs BullRes **34**, 5-15 (1945) & CA **40**, 209-10 (1946) (Reactions of Al & Mg with chlorinated hydrocarbons) 39)NFPA Comm on Hazardous Chemicals & Explosives and ACS, "Table of Common Hazardous Chemicals", C & EN **23**, 1248-56 (1945) 40)H.H.Hodgson, Chem & Ind **1945**, 362-63 & CA **40**, 5251 (1946) (Expl reaction of diazonium compounds with Na sulfides) 41)P.Nawiasky et al, C & EN **23**, 1247 (1945) & CA **39**, 4228 (1945) (Expl reaction of diazonium compds with sulfides of Na) 42)I.Kabik, US BurMinesInfoCircular **7340**, 6pp (1945) & CA **41**,

4311 (1947) (Hazards from chlorates & perchlorates in admixtures) 43)May & Baker Ltd, Chem & Ind **1946**, 89 & CA **40**, 2985 (1946) (Hazards in handling o-Nitro aromatic compds) 44)W.A.Bonner & C.D.Hurd, JACS **68**, 344-45 (1946) (Hazard involved in distillation of o-Nitrobenzoyl chloride) 45)W.G.Bailey & A.Roy, JSCI **65**, 421-22 (1946) & CA **41**, 2899 (1947) (Danger of expln in handling ether) 46)K.Nozaki & P.D.Bartlett, JACS **68**, 1686-92 (1946) (Kinetics of decomp benzoyl peroxide in solvs) 47)N.Kharasch et al, JACS **69**, 1612-15 (1947) & CA **41**, 6217-18 (1947) (Reactions of derivs of sulfenic acid with olefins) 48)A.H.Nuckolls, Underwriters' Labs-BullRes **39**, 27pp (1947) & CA **41**, 7754-55 (1947) (Expln hazard of AN contg org matter) 49)J.H. Kuney, C & EN **25**, 1658-59 (1947) & CA **41**, 5308 -09 (1947) (Expln of perchloric acid & oxidizable materials) 50)R.C.Reinhardt, C & EN **25**, 2136 (1947) & CA **41**, 6075 (1947) (Hazards in the handling of vinylidene chloride) 50a)J.J.Sampey, C & EN **25**, 2138 (1947) & CA **41**, 5723 (1947) (Mercury & ammonia, an expln hazard) 51)J.R.Archer C & EN **26**, 205 (1948) & CA **42**, 2773 (1948) (Lab expln of Mn_2O_7) 52)F. M. Cowan & O.Rörsö, C & EN **26**, 2257 (1948) & CA **42**, 7045 (1948) (Caution in the use of $AlCl_3$- Nitromethane catalyst) 53)R.Y.Mixer, C & EN **26**, 2434 (1948) & CA **42**, 8447 (1948) (Violent reaction of $Hg(NO_3)_2$ & aromatic sulfides and thiophenes) 54)L.P.Kuhn, C & EN **26**, 3197 (1948) & CA **43**, 405 (1949) (Warning to users of acetyl peroxide) 55)E.Kahne, CR **227**, 841-43 (1948) & CA **43**, 1982 (1949) (Expln hazards of mixts of perchloric acid & acetic anhydride 55a)R.M.Corelli, Ann Chim Appl **38**, 120-22 (1948) & CA **45**, 8967 (1951) (Flammability of Nitromethane) 56)R.Criegee & H.Dietrich, Ann **560**, 135-41 (1948) & CA **43**, 6189 (1949) (Prepn & props of tertiary hydroperoxides) 57)A.G.Sharpe & H.J.Emeléus, JCS **1948**, 2135-38 (Reaction of bromine trifluoride with metallic halides) 58)J.Prat, MCSE **34**, 385-87 (1948) & CA **44**, 5593 (1950) (Expln of dry benzoyl peroxide & its soln in $CHCl_3$) 59)H.H.Fawcett, C & EN **27**, 1396 (1949) & CA **43**, 5593 (1949) (Hazards of HNO_3 & alc) 60)F.Fromm et al, C & EN **27**, 1958 (1949) & CA **43**, 7229 (1949) (Expln of Bi-HNO_3-alc soln) 61)J.Barlott & A.Marsaule, CR **228**, 1497-98 (1949) & CA **43**, 8934 (1949) (Prepn & props of Hyrazine Perchlorate) 62)E. S.Shanley, C & EN **27**, 175 (1949) & CA **43**, 3199 (1949) (Further warning as to expln hazard of dry acetyl peroxide with recommended precautions) 63)F.H.Pollard & P.Woodward, Tr-FaradSoc **45**, 767-70 (1949) & CA **44**, 25 (1950) (Expl reaction of formaldehyde & nitrogen dioxide) 63a)L.Médard, MP **31**, 273-85 (1949) & CA **46**, 11687 (1952) (Expl props of hydrogen peroxide & expl decompn of aq solns) 64)T.E. Gilbert, C & EN **28**, 1550 (1950) & CA **44**, 5594 (1950) (Expln of sodium) 65)H.Rein, AngChem **62**, 120 (1950); R.Criegee, Ibid **62**, 120 (1950) & CA **44**, 6129 (1950) (Expln of Tetrahydrofuran Peroxide) 66)R.Kuhn & W.Otting, ChemZtg **74**, 139-40 (1950) & CA **44**, 6129 (1950) (Accidental expln while using Pyridinium Perchlorate) 67)N. Kharasch, JACS **72**, 3322-23 (1950) & CA **44**, 10673 (1950) (Expln hazard in the prepn & use of 2,4-Dinitrobenzenesulfenyl Chloride) 67a)E. W.Lindeijer, ChemWbl **46**, 571 (1950) & CA **46**, 7769 (1952) (Expln of CCl_4 & Al powder and the effect of CCl_4 on sheet Al) 67b)E.Banik, Brandschulz **4**, 100, 127 (1950) & CA **47**, 8371 (1953) (AN explns) 68)N.S.Marans & R.P. Zelinski, JACS **72**, 5329-30 (1950) & CA **45**, 4642 (1951) (Expln of both nitroform & Trinitroethanol during distillation) 69)C.Grundmann & H.Haldenwanger, AngChem **62A**, 410 (1950) & CA **45**, 355 (1951) (Danger in handling Methyl Azide) 70)A.Okáč & J.Gruber, ChemListy **45**, 49-51 (1951) & CA **45**, 6541 (1951) (Nitrosoguanidine and its Ni, Cu & Ag salts) 71)M.S.Kharasch et al, JOC **16**, 533-42 (1951) & CA **46**, 1485 (1952) (Prepn of Acetyl Peroxide and its expln by shock) 72)W.R.Tomlinson Jr, C & EN **29**, 5473 (1951) & CA **46**, 3279 (1952) (Phenyldiazosulfide & its p-Nitrophenyl deriv are dangerous expls) 73)G.F.Smith, C & EN **29**, 1770 (1951) & CA **46**, 3279 (1952) (Ammonium Periodate is an expln hazard) 74)H.Cohen, C & EN **29**, 1770 1951) & CA **46**, 3279 (1952) (Expln of an abandoned H_2SO_4 feed line) 75)B.D.Halpern, C & EN **29**, 2666 (1951) & CA **46**, 3279 (1952) (Expln of 2,4-Dinitrochlorobenzene) 76)T.S.Reid & G.H. Smith, C & EN **29**, 3042 (1951) & CA **46**, 3279 (1952) (Expln of perfluorosuccinamide & $LiAlH_4$) 77)A.E.Cameron & A.V.Faloon, C & EN **29**, 3196 (1951) & CA **46**, 3279-80 (1952) (Expln of liq Chlorine dioxide) 77a)L.Médard, MP **33**, 125 -35 (1951) & CA **47**, 5683 (1953) (Expl props of

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Dangerous Materials, Shipping and Storage of. Disastrous effects of neglecting of physical & chem props of stored materials are: fires, explosions, emission of toxic gases, vapors, dusts & radiation, and various combinations of these effects. Because a wide variety of materials must be stored in laboratories, factories & warehouses, good storage practices are not only important but also may be safer & more economical. In order to group the many thousands of individual items in such a way that the best use is made of available space, the following group categories are given by Sax (Ref 3)

a) *Acceptable Explosives.* See Vol 1, p A11-L
b) *Flammable Materials* (solids, liquids & gases)
c) *Oxidizing Agents* (oxides, peroxides, chlorates, perchlorates)
d) *Water-Sensitive Materials* (lithium, sodium, hydrides, nitrides, silicides, acids, & alkalies)
e) *Acid- and Acid Fume-Sensitive Materials* (Concd alkalies, metals & cyanides)
f) *Compressed Gases* (oxygen, hydrogen & acetylene)
g) *Toxic Materials* (carbon tetrachloride & radioactive substances)
h) *Corrosive Materials* (acids, anhydrides & alkalies)

A thorough knowledge of the physical & chemical props of each material, its degree of flammability, disaster conditions & fire control must be known, as well as compatibility of materials within a group for the safe location & construction of storage areas. Existing laws & established regulations regarding the storage of dangerous materials are helpful. Also many manufacturers issue pamphlets with respect to the safe storage, transportation & use of their products

The packing & shipping regulations of dangerous materials, including expls, are prescribed in the US by the Interstate Commerce Commission (ICC), Bureau of Explosives Tariff No 10 and are mandatory for their safe transportation within the jurisdiction of the US(Ref 3, Sect 11). German regulations for the handling & shipping of dangerous materials are found in Ref 2

See also *Danger of Handling Explosives* and *Dangerous (Hazardous) Chemicals and Other Materials* in this Volume

Refs for Shipping and Storing Dangerous Materials: 1)M.M.Kostevich, "War Ammunition Breaking - Down and Explosives and Gas Recovery Factory Rules for Employees", Talleres Gráficos-Kondratowicz y Lasecki, Buenos Aires (1945) (Contains general rules & regulations, special rules & precautions, and factory fire precautions & rules) 2)Anon, Explosivst **1956**, pp 38-40, 60-61, 162-65, 188-90 & 215-16 (Contains German regulations) 3)Sax (1957), pp 147-70 and Sect 11 ICC Shipping Regulations) 4)US Army Regulations **AR 55-55** (1964) (Transportation of radioactive & fissionable materials other than weapons) 5)US Army Materiel Command Regulations **AMCR 385-101** (1964) (Safe shipping criteria for Etiologic Agents & Biological materials) 6)US Army Material Command Regulations **AMCR 385-224** (1964) (Safety Manual) 7)US Dept of the Army Technical Manual **TM 9-1300-206** (1964) (Care, handling, preservation, and destruction of ammunition)

Danyaku. Japanese for ammunition

Dapremont's Powder. Patented in 1877, it consisted of $KClO_3$ 48.5, sugar 41.5 & sawdust 10%

Ref: Giua, Trattato **6** (1) (1959), 393

Darapsky's Incendiary Composition. Intended for loading incendiary projs, it consisted of 3 parts yel phosphorus dissolved in 1.3 part of carbon bisulfide, placed in one compartment and in another compt was placed petroleum. On bursting the proj, CS_2 evaporated allowing the P to catch fire which, in turn, ignited the petroleum

Ref: Daniel (1902), 153

Darcet Alloy (Alliage Darcet in Fr). Used in France as a heating medium (instead of Wood's metal as in the US) to determine expln or ignition temp of expls

Ref: A.LeRoux, MP **32**, 124 (1950)

Dark and Dim Igniters. The usual pyrotechnic igniter compns, used in tracers, burn rapidly with a bright flash. In order not to blind a gunner nor to disclose the position of the gun to the enemy, it is desirable that the igniter compound not produce visible light at the muzzle and that the tracing action start only at some specified distance from the gun. Ig-

niters which produce no visible light, even at night, are called *Dark Igniters*. A dark-burning igniter, patented by Heiskell (Ref 1), consists of Bi_2O_3 45-85, Mn 15-55, stearic acid binder up to 10 & graphite lubricant up to 10%

While a dark igniter usually works satisfactorily at night, it does not in daylight because then it is more difficult for a gunner to pick up the path of his tracer. This difficulty can also happen at night, expecially in a maze of cross fire. In order to avoid this difficulty, the so-called *Dim Igniter* is used. This exhibits a faint streak from the gun muzzle to the point where the tracer compn begins, without producing any flash or area illumination (Ref 2)
Refs: 1)H.E.Hirschland & S.Ricklin, Ordn **32**, No 164, p 91 (1947) 2)R.H.Heiskell, USP 2716599 (1955) & CA **50**, 8208 (1956)

Darrieus Effect (l'Effet Darrieus in Fr). The variation of air resistance with temp and its application to ballistics, named in honor of Captain Darrieus of France, is referred to in the Ref
Ref: M.M.Garnier, MAF **25**, 823-24 (1951)

Dart. A US Army surface-to-surface missile developed by Redstone Arsenal & the Aerophysics Development Corp. Its length is 5 ft, diam 8 inches, wt 300 lbs, range 5 miles. It uses a composite solid proplnt and a shaped -charge warhead

The Dart is designed to attack tanks. It is capable of pinpoint accuracy due to its command-guided system which is accomplished by means of trailing wires which unwind from the missile. The Dart missile can be fired from a field launcher, an armored transport jeep, or a helicopter
Refs: 1)F.I.Ordway III & R.C.Wakeford, "International Missile and Spacecraft Guide", McGraw-Hill, NY (1960), p 3 (Under USA) 2)Ord Tech Term (1962), 91-R

Dart Composite Propellant, CBS 128K. Its ballistic evaluation is given by I.Spiess, PicArsn FREL, Propulsion and Propellants Lab Rept **3** (July 1958) (Conf)

Darts. An Amer term for small incendiary bombs dropped in clusters during WWII from airplanes. See also under Incendiary Warfare

Dash. Abbr for Drone Antisubmarine Helicopter
Ref: P.Eleson, Ordn **47**, 611 (1963)

Dashpot. A type of buffer used in some recoil systems to cushion the movement of the recoiling parts as they near the end of recoil and again during return to battery
Ref: Glossary of Ord (1959), 88-R

Dashpot, Anchor, Underwater Mine. A device consisting of a cylinder and hydrostatically operated piston, with a means of securing at both ends. Its purpose is to delay release of the anchor plummet by restricted passage of fluid thru an orifice or around the piston. The release mounted on the anchor serves to separate the anchor from the mine case at a predetermined depth
Ref: Glossary of Ord (1959), 88-R & 241-L

DATB. An abbr for Diaminotrinitrobenzene, described under Diaminobenzene

DATB-HMX Explosive Compositions for High Temperature Resistance and High Energy. They are discussed by B.A.Stott & D.A.Sbrocca in conf NAVWEPS Rept **7589** (NOTS TP 2576) (Dec 1960)

Dautriche, H (1872-1914). A Fr scientist who contributed largely to the science of expls. He invented a simple method for detn of *Detonation Velocity* (See item **H** under CHRONOGRAPHS, p C311-R). Dautriche died from the expln of a kneader filled with a perchlorate compn
Refs: 1)Stettbacher (1933), 57 2)Peppin Lehalleur (1935), 445

Davey's Powder 1. Patented in England in 1852, the powder consisted of a mixt of $KClO_3$, $K_2Cr_2O_7$, KNO_3, K ferrocyanide & Sb trisulfide (Ref 1). Giua (Ref 2) gives the following compns (in parts): 1)$KClO_3$ 6, $K_2Cr_2O_7$ 2, KNO_3 5, K ferrocyanide 2 & Sb_2S_3 5 and 2)$KClO_3$ 6, KNO_3 3, K ferrocyanide 4 & Sb_2S_3 3
Refs: 1)Daniel (1902), 179 2)Giua, Trattato **6**(1) (1959), 391

Davey's Powder 2. Patented in England in 1858, the powder was a modified BkPdr in which the

charcoal was replaced by starch, flour or bran and KNO_3 was replaced by $NaNO_3$. Davey also proposed (in 1862) to incorporate in BkPdr 4 to 6% of the product obtd on boiling 1:3 nitric acid water with starch, dextrin, gum flour or sugar and to replace part of the sulfur with a hydrocarbon
Ref: Daniel (1902), 179-80

Davey's Powder 3. Patented in England in 1877, the powder was prepared by treating NC with solns of chromates or dichromates with or w/o K nitrate, glue or hydrocarbons and then drying. It was used for the prepn of safety fuses
Ref: Daniel (1902), 180

Davey & Watson's Powder. Patented in England in 1874, the powder consisted of BkPdr impregnated with either liq or solid hydrocarbons
Ref: Daniel (1902), 180

Davies & Curtis' Safety Explosive. Patented in England in 1899, the expl consisted of a mixt of KNO_3 85, lignite 14 & sulfur 1%
Ref: Daniel (1902), 180

Davies' Powder. Patented in England in 1860 with the intention to replace BkPdr, the powder consisted of a mixt of $KClO_3$ 47.0, K ferro(or ferri)cyanide 23.5, sugar 23.5 & sulfur 6.0%
Refs: 1)Daniel (1902), 180 2)Giua, Trattato **6** (1) (1959), 391

Davies' Safety Explosive. Patented in England in 1899, it consisted of a mixt of AN 80-90 & lignite 20-10% to which could be added 10% paraffin
Ref: Daniel (1902), 181

Davis' Ammonia Process. It is reported to be capable of producing soft & pliable AN-Fuel mixts sensitive enough for reliable propagation in regular dynamite cartridges, even of small diameter. These mixts, however, have no intrinsic water or moisture resistance and are not easily waterproofed without excessive loss of sensitiveness. They would, therefore, require the use of waterproof containers
Ref: Cook (1958), 13

Davis, Tenney L.(1890-1949). An American scientist, formerly professor of org chem at MIT and Director of Research & Development at National Fireworks Inc, was an authority on org chem, fireworks & expls. Davis was the author of numerous publications, including the book "The Chemistry of Powder and Explosives", Wiley & Sons, NY (1943), 490pp
Refs: 1)H.M.Leicester & H.S.Klickstein, J ChemEduc **27**, 222-24 (1950) & CA **44**, 4731 (1950) (T.L.Davis and his work in the history of chem) 2)Ibid, Chymia **3**, 1-16 (1950) & CA **45**, 7 (1951) (Portrait & bibliography of historical papers of TLD)

Davy Crocket (Automatic Rifle). See under Atomic Weapons and Ammunition, Vol 1, p A504-R and also Anonymous, Ordn **45**, 110 (1960)

Davy, Sir Humphry (1778-1829). An Engl chemist who isolated Na, K, Ba, Ca & Sr metals by electrolysis and devised the *miner's safety lamp*, briefly described in Vol 3, p C362-R, under Coal Mine Explosions and Fires
Ref: Hackh's (1946), 253-54

Dawson & Karstairs' Powders. Patented in England in 1899, they were prepd by mixing urine (previously concd by evapn of about 0.1 its vol) with nitric acid & alcohol in one of the following proportions:

Composition, %	No 1 Powerful Explosive	No 2 Weak Explosive
Urine (concd)	37.5	60.0
Nitric Acid	37.5	33.3
Alcohol	25.0	6.7

Each mixt was evapd in vacuo at low temp to the point of formation of crysts. After decanting the mother liquor, the crysts were pulverized & coated with a soln of crude rubber (or other gummy subst) in petroleum naphtha and then either pressed or grained. Mixture No 1 was suitable for miliatry purposes and was recommended for loading torpedoes
Ref: Daniel (1902), 181-82

DB. A cannon proplnt developed by Hercules Powder Co. Its compn exclusive of TV (total volatiles) was: NC(13.25%) 76.45±2, NG 19.5 ±1, Ba nitrate 1.40±0.2, K nitrate 0.75±0.25, DNT 1.00±0.25, DPhA 0.6±0.15 & graphite

0.30±0.10. TV 0.5% max for zero web; ash 0.4% max and stability 40mins at 120°
Ref: G.D.Clift, formerly of Hercules Powder Co; private communication (1950)

DBP or DBPh. Abbrs for Dibutylphthalate

DBX (Depth Bomb Explosive). An Amer aluminized expl developed during WWII to replace the Brit,more sensitive Torpex. It consists of TNT 40, RDX 21, AN 21 & Al 18%. Its props are as follows: gray solid which can be cast at temps 90-95°; *Density* (for cast material) 1.61 to 1.76g/cc; *Hygroscopicity* sl hygroscopic; soluble in acet (except Al component); *Linear Coeff of Thermal Expansion, %* (betw 73° & +75°) 45×10^{-6} per °C; *Vacuum Stability* at 100° 0.6cc of gas evolved in 40hrs; *Brisance* 48g sand crushed vs 43g for TNT; *Detonation Velocity* 6630m/sec at d 1.65 & 6800 at d 1.76g/cc; *Energy of Air Blast* 138% of TNT; *Energy of Water Shock* 143% of TNT; *Explosion Temperature* 200° in 5secs; *Impact Sensitivity* (PicArsn App, 2kg wt) 10 inches, vs 14 for TNT; *Power by* Ballistic Mortar 146% of TNT; and *Rifle Bullet Test* - ca 49% of detons from impact of cal .30 bullet at distance of 90 ft
Refs: 1)All&EnExpls (1946), 130 2)PATR **1740** (Rev 1) (1958), 97-100; AMCP **706-177** (1963), 83-86

DC. Chemical Warfare Service (CWS) symbol for Diphenylcyanoarsine. See Vol 2, p C167-R

DD Device. See Vol 1, p A392-R under Amphibious Devices for Tanks

DD Explosifs. Fr expls developed before and during WWI. They consisted of Picric Acid & Dinitrophenol in various propns

PA	60	63	71	75
DNPh	40	37	29	25

Of these, the 1st compn, known as *DD 60/40, Mélange DN 60/40 or Explosif MDn,* mp 85°, was used to a great extent during WWI for cast loading various projectiles. This mixt corresponds to Brit *Shellite*

All DD expls could be prepd either by fusing DNPh with PA or by direct nitration of phenol, dissolved in CCl_4, until the mixt of desired propns of DNPh & PA is obtd

Refs: 1)L.Desvergnes, Chim & Ind (Paris) **26**, 515 & 1271 (1931) and **27**, 280 (1932) 2)Vennin, Burlot & Lécorché (1935), 457 3)Pepin Lehalleur (1 35), 441 4)Davis (1943), 166-67 5)All & En Expls (1946), 100 6)Anon, "Italian & French Explosive Ordnance", Depts of the Army & Air Force TM9-**1985-6** & TO 39B-1A-8 (1946), 177 & 180-82

DDP. Mixt of DINA, DBuPH & EtCentr: 88.5/10/1.5 was used in a WWII proplnt called *Albanite* (Vol 1, p A119-R)
Ref: Anon, Summary Technical Report of Div 8 NDRC, Vol 1 (1946), 15 & 153

DDP (Propellant). ABL modified double-base proplnt described as Unit No 597 in
Ref: Propellant Manual SPIA/M2 (1962) (Conf)

DDP 70 (Propellant). See DGV

DDP 77 (Propellant). Hercules Powder Co modified double-base proplnt for rocket motors, described as Unit No 639 in
Ref: Propellant Manual SPIA/M2 (1962) (Conf)

DDT. Abbr for Deflagration-to-Detonation Transition
Ref; M.A.Cook et al, JApplPhys **30**, 1579-84 (1959) & CA **54**, 1851 (1960)

Deactivated Apparatus. It has been observed that during vapor-phase nitration of hydrocarbons, the yield & conversion drop off after continued operation of the equipment. When this occurs, the reaction vessel is described as "deactivated". This phenomenon is similar in nature to the poisoning of a catalyst, such as of Pt, used in catalytic reactions (Ref 3). Martin (Ref 2) patented a process for inhibiting deactivation by means of a molten salt bath. The preferable salts are those of alkali & alkaline earth metal compds that are stable in the presence of HNO_3. Landon (Ref 1) overcame deactivation by constructing the reaction vessel from materials (such as glass, fused silica, gold or platinum) having substantially no negative catalytic effect. A diagram of equipment suitable for conducting vapor-phase nitrations, together with a description

of the process, is included in the patent
Refs: 1)G.K.Landon, USP 2161475 (1939) & CA **33**, 7319 (1939) 2)J.Martin, USP 2260258 (1941) & CA **36**, 779 (1942) 3)W.deC. Crater, IEC **40**, 1631-32 (1948)

Dead-Cooling (Tot-kühlung in Ger). When a MF cap was cooled in liq air or liq oxygen, the expl lost its prop to initiate, just as if it were dead-pressed (qv)
Ref: Stettbacher (1948), 72

Dead Cotton. This name is given to unripe fibers of cotton having very thin walls. These fibers have died before reaching maturity as a consequence of attacks by parasites, genetic factors or competition for nutriment. Such fibers are less easily nitrated or acetated than ripe cotton and constitute a large proportion of the "fly" which passes into cotton waste

See also Cotton, Vol 3, pC545-R
Ref: Marshall **1** (1917), 167; **2** (1917) 696; and **3** (1932), 33

Deadeners. Same as Deterrents (qv)

Dead Fibers. Same as Dead Cotton

Dead Oil. The name given to a mixt consisting chiefly of coal-tar hydrocarbons. It was used in Amer blasting expl *Rackarock* as a partial replacement of NBz
Ref: Thorpe **4** (1940), 545

Dead-Pressed Explosives. Some expls, such as MF, LSt, TATNB, Tetracene or N_4S_4, lose their sensitivity to detonation (become insensitive) to flame, hot wire, spark or fuse if they are previously subjected to very high pressures, such as 25000-30000psi (Muraour, Ref 3 gives for MF 700kg/cm ^{2}). These expls are then said to be *dead-pressed* (surcomprimée, in Fr). Accdg to Muraour et al (Refs 3 & 5), MF or LSt will, under these conditions ignite on the surface. If, however, a small amt of other initiating expl, such as pressed LA, will be brought in contact with dead-pressed MF or LSt and LA detonated, then MF or LSt will detonate also. Muraour has shown that contrary to prevalent opinion the brisance of dead-pressed MF or LSt increases with the degree of compression. The phenomenon of dead-pressing should not be confused with *hypercompression,* as takes place with AN or chlorate expls. When these expls are very highly compressed they cannot be detonated even under the action of the strongest initiating expls, such as LA (Ref 5)

Muraour et al (Ref 3) think that the reason for MF not to detonate when ignited is that it burns on the surface only and the hot gases cannot penetrate inside the mass of expl, while in the case of not highly compressed material, they easily penetrate inside the mass, thus causing the deton. If this theory is accepted, then it would be understandable why LA cannot be dead-pressed. This is because when it is touched with a hot wire, it detonates without previous combustion. Diazodinitrophenol, Ag azide, Hg Azide, Ag Fulminate also cannot be dead-pressed

Muraour & Wohlgemuth (Ref 2) have shown that if dead-pressed MF is preheated to 120-130° and then touched with a hot wire, it will detonate without previous combustion. This means that MF behaves in this case like LA. Direct deton of dead-pressed MF takes place either at atm pressure or in vacuo
Refs; 1)Anon, Army Ordn **3**, 58 (1922) & CA **16**, 3395 (1922) (Dead-pressed expls) 2)H. Muraour & J.Wohlgemuth, Chim & Ind (Paris) **36**, 472-73 (1936) 3)H.Muraour, MAF **18**, 895 -97 (1939) & CA **34**, 4905 (1940) (Sur la surcompression des explosifs d'amorçage) 4)K.K. Andreev, DoklAkadN (ComptRendAcadSci) (Russia) **31**, 456 (1941) 5)H.Muraour & A.Demay Chim & Ind (Paris) **56**, 463-67 (1946) & CA **41**, 2899 (1947) 6)A.R.Ubbelohde & P.Woodward, TrRoySoc **241A**, 237 (1948) 7)W. Schneider, Explosivst **1953**, 1-10; Engl trnsln by W.W.Lee,Jr, Atlas Powder Co Rept **RXL-67-75** (July 1954)

Dead Shot Powder. A smokeless proplnt intended for small arms ammo. It can be prepd as follows:

A slightly moistened pulped NC is agitated in tumbling drums until the mass forms itself into compact separate rounded grains which are sufficiently coherent to maintain their form in the handling and screening steps which follow. The screened grains are dried, moistened with a small amt of a solvent (such as ether-alc or acet) and tumbled in closed drums, which are slightly heated. As result of this the solvent disseminates thru the mass of grains as far as it can be by permeation of the solvent vapor

and by contact of the grains which have absorbed less of the solvent with those which have received more of it

EC and Schultze powders were made in a similar manner

Ref: C.R.Borland, USP 1752881 (1930) & CA **24**, 2606 (1930)

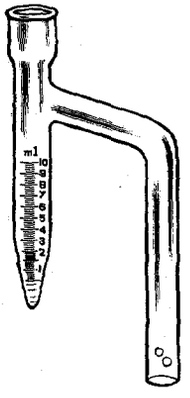
Dead-Stop End Point Method of Titration was described by C.W.Foulk & A.Bowden, JACS **48**, 2045 (1926). It is also described by F.P. Treadwell & W.T.Hall, "Analytical Chemistry" Wiley & Sons, NY (1942), 48; and by Vogel, Inorg Analysis (1961), 945, 956 & 1051

Dean Explosive. Patented in England in 1881, it was prepd by blending 10 parts of pulped Collodion Cotton (or powdered dextrin), 100 pts NG & 2-3pts of water, followed by graining and drying

Ref: Daniel (1902), 182

Dean-Stark Tube. A double-branch tube used for detn of moisture in substances by solvent distillation. If a sample contains sufficient moisture this method is convenient

Procedure: A sample (such as of an expl), accurately weighed, is introduced into a 500 ml short-necked round-bottom flask. To this is added 100-150ml of toluene or xylene (previously saturated with water by shaking with w and alowing to stand for separation) and the neck of the flask is connected thru a cork stopper to the right hand branch of a previously thoroughly cleaned Dean-Stark tube or its equivalent (such as described in Vol 1, pp A370-A371). The left hand branch of the tube is connected by means of a cork stopper to a vertical reflux condenser and distillation can



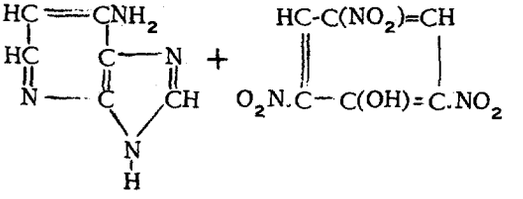
begin. The flask is usually heated in an oil bath at a temp such that the solvent distills at the rate of ca 100 drops per min. The moisture evaporates and is carried over by the solvent vapor. Both are condensed, but the w, being heavier and practically insol in the solvent, gradually settles at the bottom of a calibrated tube. If any w adheres to the walls of condenser, it can be washed down by pouring ca 5ml of solvent thru the top of condenser. The droplets of w in condenser or the tube may also be moved down by means of a long spiral wire. When the greater part of w has distilled over, the distn rate is increased to 200 drops per min until the vol of w fails to increase, but the distn should not be less than 2 hrs

Then the tube is cooled to 20° and the vol of w in the tube read

An all glass app is preferred to that using cork stoppers

Refs: 1)F.D.Snell & F.M.Biffen, "Commercial Methods of Analysis", McGraw-Hill, NY (1944), 41-2 2)StdMethodsChemAnalysis **2A** (1963), 529

1-Deazadenine Picrate or 7-Aminoimidazo [b] pyridine picrate,



$C_{12}H_9N_7O_7$; mw 363.25, N 27.00%; yel crysts (from w), mp 310° (block); was prepd by treating 2,3,4-triaminopyridine dichloride in cold water with 40% formalin, then with $Cu(OAc)_2$, boiling 4 hrs, cooling & filtering, washing with cold water, decompg with H_2S, and then adding Picric Acid. This compd was prepd also by condensation of the dichloride with Na dithioformiate (HCS_2Na). Its expl props were not detd

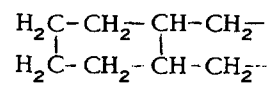
Ref: F.Kögl et al, Rec **67**, 40 (1948) (in German) & CA **42**, 5015 (1948)

Decarboran or Decaboron Tetradecahydride. See Vol 2, pp B254-L & B255-L

Decalin, Dekalin, Decahydronaphthalene, or Bicyclo (4.4.0) decane and Derivatives

Decalin (called Bicyclo-[0.4.4]-decan;

Dekahydronaphthalin and Dekalin in Ger),



$C_{10}H_{18}$; mw 138.24; H 13.12; col liq which exists in two isomers: cis, fr p -43.3, bp 194.5 & d 0.8967; and trans, fr p -30.7, bp 185.8 & d 0.8700. The coml product is a mixt of both isomers. It is used as a solv for many org compds, as a cleaning fluid and as a substitute for terpentine & lubricating oil

Refs: 1)Beil **5**, 92, (46), [56] & {242} 2)Hackh's (1944), 257 3)Kirk & Othmer **1** (1947), 354; **7** (1951), 610; **9** (1952), 230; **12** (1954), 668, 677, 682, 729; and **13** (1954), 869 4)Sax (1956), 534

Azidodecalin, $C_{10}H_{17}N_3$, not found in Beil or in CA thru 1961

Nitrated Derivatives of Decalin

4a-Nitroso-trans-decalin, $C_{10}H_{17}NO$, mw 167.24, N8.38%; blue crysts, fr p -16, bp 87° at 18mm, d 1.004; other props & methods of prepn are given in Beil

Ref: Beil **5**, {251}

Mononitrodecalin, $C_{10}H_{17}NO_2$; mw 183.24, N7.64%. The 1-Nitro deriv, bp 108-09° & d 1.0825 at 20°; and 9-Nitro deriv in cis & trans forms are found in Beil. The mononitro deriv was prepd & examined in the US during WWII and found to be an expl, but not very powerful nor brisant

Ref: Beil **5**, [60] & {251}

9,10-Dinitro-trans-decalin, $C_{10}H_{16}N_2O_4$; mw 228.24, N 12.27%; crysts (from alc), mp 167° (dec); obtd with other products by nitration of decalin (Refs 1 & 2). Its expl props were not detd

Refs: 1)Beil **5**, (252) 2)Pl.A.Plattner & J. Hulstkamp, Helf **27**, 220, 228 (1944) & CA **38**, 4570 (1944)

Trinitrodecalin, $C_{10}H_{15}N_3O_6$, was not found in Beil or in CA thru 1961

Decalin Hydroperoxide, $C_{10}H_{18}O_2$; mw 170.24, O 18.80%; ndls (from petr eth), mp 96.5°, bp 103° at 1.2mm, d 1.15 at 4°; was obtd by bubbling O thru coml decalin at the rate of 5cc per min under UV radiation for 200 hrs at 70°. The hydroperoxide liberates iodine from HI solns, colors titanic acid solns, and after treatment with CH_2O decolorizes an alk soln of methylene blue (Refs 2 & 3)

Its *p-Nitrobenzoate Ester,* $C_{17}H_{21}NO_5$, lt-yel ndls, mp 94° (dec) & exploding on a spatula placed in a flame, was obtd by interaction of the hydroperoxide with p-nitrobenzoyl chloride at 0° for 5 hrs (Ref 4)

Refs: 1)Beil - not found 2)R.Criegee, Ber **77B**, 22-24 & 722-26 (1944); CA **39**, 1862 (1945) & **40**, 6433 (1946) 3)K.I.Ivanov & V.K.Savinova CR AcadSci (Russia) **48**, 31-34 (1945); Dokl-AkadNauk **48**, 32-35 (1945) & CA **40**, 4706 (1946) 4)R.Criegee & H.Dietrich, Ann **560**, 133-34 (1948) & CA **43**, 6189 (1949)

Decanitrocellulose or Pyrocellulose, $C_{24}H_{30}O_{10}(ONO_2)_{10}$, mw 1098.56, N 12.57%. One of the varieties of NC's used for making smokeless proplnts. See under Cellulose Nitrates in Vol 2, pC100-R

Decantation. A method of separating a sediment (solid or liquid) by pouring off the superimposed liquid layers. In *continuous countercurrent decantation,* the finely divided solids (such as ground ore, chem ppts, residues form leaching opns, etc) are separated from liquids contg dissolved substances by washing with w or other liq. For this the suspension of solids to be septd from liquids passes thru sedimentation tanks, called *thickners,* placed in series. In each thickner part of the solid settles and a weaker liq overflows from subsequent thickner of the series. The direction of this overflowing is opposite to that of feed

For more detailed description, see the Refs

Refs: 1)Perry (1950), 950-55 2)Kirk & Othmer, Vol **12** (1954) (Under Sedimentation), 126-45

DECHEMA. See Vol 1, p Abbr 14-L

Deck Loading. See under Density of Blasting Explosives; Density in Borehole, etc

Decolorization. This is the process of making dark substances lighter in color and opaque substances more nearly white or transparent by either chemical destruction, or removing of coloring impurities by adsorption. Decolorization must be performed without damaging effects upon treated material. In the chem decolorization method, known as bleaching, the material is treated with an oxidizing or

reducing agent. In the adsorption method, the material can be either suspended in a nonsolvent together with an adsorbent or filtered thru a layer of it. The most common adsorbents are: activated charcoals, adsorbent clays, alumina, silica gel, ion exchange resins & kieselgur

Decolorization may also be conducted by extraction

Refs: 1)Thorp **2** (1938), 315 2)W.R.Aenelt, "Entfärbungs- und Klärmittel", Steinkopf, Dresden (1943) (Bd 48 of Technische Fortschriftsberichte) 3)Kirk & Othmer **4** (1949) 874

Decomposition, Destruction and Disposal of Ammunition, Explosives, Propellants and Pyrotechnic Compositions.

Decomposition of Explosives. This may be achieved by the following methods: a)Chemical (as briefly described in Vol 2, p C173-L) b)Thermal (as briefly described under Burning Ground in Vol 2, p B357-R) c)Electronic and d)Photochemical

Noyes & Vaughan (Ref 5) investigated thermal, electronic and photochemical decompns of solid bodies, among them some expls. They found that in many cases where the rate of thermal decompn can be detd without ambiguity, the variation with temp is satisfactorily given by the Arrhenius equation: $K = Ae^{-E/RT}$, where K is the specific reaction rate, A is a constant, E "the heat of activation", (the critical increment per gram-molecule), R the gas constant per g mol per °C and T the absolute temp

The reactions usually obey the equation for a unimolecular reaction: $-dM/dt = KM$, where M is the mass of solid in grams and t is the time in secs. In some cases, however, the rate is proportional to the surface of the solid instead of its mass, and, in addition, the equation sometimes needs modifications because of such phenomena as autocatalysis

Appropriate equations for photochemical and electronic decompns were also derived and are given in Ref 5

Marshall (Ref 6) stated that the work carried in England at Woolwich Arsenal after WWI showed that all expls decompose to some extent in storage, evolving gas in accordance with the equation: $V = k^t + C$, where V is volume of gas evolved, t is the temp and C is a constant. For every 5°C increase in temp, the rate of decompn is approx doubled but may increase as much as 50 times if the expl is melted. The rates of decompn depend largely on the conditions, especially on the presence or absence of impurities (even traces), which may act as catalysts. In nearly all cases, decompns are partly catalytic and partly noncatalytic and if catalytic influence is eliminated the decompn usually slows down. Sometimes, as for example, in the cases of NG and NC, the decompn proceeds at an accelerated pace due to autocatalysis. In the case of aromatic nitrocompds, such as TNT, PA, Tetryl, the decompn can be slowed down by eliminating impurities which are usually less stable than the expl itself. With many expls the presence of moisture increases the rate of decompn and might cause erratic results in stability tests

The loss of wt on decompn of expls, proplnts and pyrotechnic compns can be detd by means of "thermo-balances", such as of Guichard (described in detail inRef 7a), of Ishikawa (Addnl Ref E), of Gill (Addnl Ref F) and of Hikita (Addnl Ref R)

The thermo-balance of Glasner & Simchen (Addnl Ref S), which is of larger capacity (3kg) than previously known, is constructed as follows: The balance is set up on an insulated table, installed sufficiently high above an electric muffle furnace to avoid any undue heating, The furnace is well insulated on the outside. A sample (usually 200g) is placed in a large quartz crucible fitted with a Ni or stainless steel cover and suspended in a cradle of Ni-Cr wire, from which a single strand extends thru the surface cover. A Pt-Rh thermocouple is attached to the supporting leads with its hot junction as near as possible to the center of the chge. The cold end of thermocouple is connected with a potentiometer by means of two fine Cu wires. Then the sample is heated at the desired temp and the loss of wt recorded as a function of time until complete decompn takes place. With this app it is also possible to record loss of wt at constantly increasing temps as in the balance of Guichard (Ref 7a). The advantage of the G & S app over that of Gill (Addnl Ref F) and others is that it measures the temp of the sample itself and not only that of the furnace. Accuracy of weighing is claimed to be 0.1g

Robertson (Refs 14, 15, 16 & 17) developed an app for heating samples to a given temp within a small fraction of a second and the resulting liberation of gaseous products was followed by automatic recording. He studied by this method the decompn of many expls, solid and liquid

Hinschelwood & Bowen (Addnl Ref C) tested substances evolving gases on decompn, by heating a 0.5g sample placed in a small bulb (ca 10ml capacity), provided with a stopcock and connected by a capillary tube to a gas-measuring buret. Each 30mins the stopcock was opened and the gases transferred to a graduated buret. After measuring and recording the volume, the gases were analyzed

Lambrey (Ref 6a) recommended the following method for detn of decompn of N contg expls at storage temps & at reduced pressures: A 7.2g sample of dry substance is placed in a tube provided with two quartz windows to permit the study of the absorption spectra. After evacuating the tube to a pressure of less than 2 microns Hg, the tube is sealed and placed in an oven maintained at 30° or higher temp during several weeks or months. During this treatment, the substance decomposes partly or completely, thus releasing some NO gas. Then the tube is cooled, hydrogen gas is introduced (to bring the pressure to atmospheric level) and the intensity of the NO absorption bands is measured. If the relation betw the intensity of a band and the concn of NO is known, the amt of NO released on heating can easily be calcd. For example, ordinary Fr Guncotton (13.1% N) liberates at 30°, in 100 days, an amt of nitrous gases corresponding to 6.25×10^{-7} mg N, while PETN does not start to release any gas until the temp reaches ca 43°. For a detailed description of the apparatus, see Ref 6a

In the method of Haid & Schmidt (Addnl Ref H), the gases formed on decompn of expls were obtained only when the gases were cooled rapidly by expanding their energy under conditions of performing work, because it was claimed that no reliable info as to the decompn products of an expl can be obtd if these conditions are not fulfilled. In the method of H & S, the gases were cooled by the work done on expanding a cavity drilled in a Trauzl lead block. The mouth of the cavity was tightly sealed by a clamping device and provision was made for drawing off the gases so that their volume could be measured and compn detd by analysis

Riabinin (Addnl Refs K & M) investigated in Russia the influence of high pressures on the rate of decompn of expls. For this a small sample of a solid expl was compressed at ca 25000kg/cm^2 in the form of a thin tablet and this was placed in a small cylinder provided with a plunger. The air in this container was maintained at atmospheric pressure. A second tablet of the same expl, compressed in the same manner as above, was placed inside a small hydraulic press where the air was maintained at ca 2500kg/cm^2. Each container was heated in an electric oven until a slight decompn took place in one of the tablets. This temp was noted and freeh tablets of the same material were heated for 3 mins, as above, but at temp just below the decompn of the first tablet. After noting the temp, the heating was contd until decompn of the 2nd tablet. As it is very difficult to observe the decompn of tablets inside the containers, Riabinin removed the tablets periodically from the containers and heated them in the open at temps slightly higher than those observed before their removal. The absence of a flash on heating in the open indicated that the tablet was already decompd and the heating in a closed container was repeated with a fresh tablet but at a lower temp than the one observed before removal from the container. If the tablet removed from the container flashed on heating it in the open at a temp slightly above that observed on heating in the container, it would mean that the tablet was not decompd before its removal from the container. Then the heating was repeated many times with fresh tablets in closed containers but at higher temps than above, until the temps as close as possible to decompn points were obtd. This work seems to be very tedious

The temp at which the sample just began to decomp was named the "temperature of initial (or induction) period". For a sample heated in container at atm pressure it was designated as T_i°, and for a sample heated in hydraulic press at 2599kg/cm^2, as T_p°

The initial period was followed for many expls by a "period of acceleration", which was autocatalytic in character. This resulted in complete decompn of the expl. The temp of this final stage of decompn was designated as T_1^+ for decompn at atm pressure and for that at pressure of 25000kg/cm^2 as T_p^+

Results of tests are given in table

Explosive	T_1^o	T_1^+	T_p^o	T_p^+
LST	270	276	243	254
MF	147	155	128	161
LA	314	319	314	331
PETN	186	190	220	227
BaA	168	168	233	233

LSt = Lead Styphnate
MF = Mercuric Fulminate
LA = Lead Azide
PETN = Pentaerythritol Tetranitrate
BaA = Barium Azide

This table shows that T_1^o values for LSt & MF are higher than T_p^o's, while for PETN & BaA they are lower and for LA the values are identical T_1^+ values for LSt are higher than T_p^+'s, while for MF, LA, PETN & BaA they are lower

From the values of this table and from some calcns, Riabinin came to the conclusion that high pressures increase the rate of decompn of LSt, (both initial & final stages), and increase also the initial stage of decompn of MF, while decreasing its final stage. The initial stage of decompn of LA is unaffected by high pressures, while the final stage is diminished. In cases of PETN & BaA, high pressures diminish the rate of decompn in both initial and final stages

If the decompn of all expls obeyed the law of LeChatelier, the high pressure would diminish the rate of decompn and not increase it as is in the case of LSt

Hikita (Addnl Ref R) studied the thermal decompn of nitric esters such as NG and came to the conclusion that it is almost monomolecular in nature, but in the presence of even a small quantity of moisture, ionic catalysis becomes of increasing importance. Cu has a positive catalytic action for decompg NG at high temps, while gum has a markedly negative action at lower temps. The order of catalytic effect of various metal surfaces was observed to be as follows: Cu > CuO > Brass > Fe > Mo ~ Zn ~ Pb > Al. This shows that Al has the weakest catalytic action for decompg NG. When the surface of Cu was covered by a thin film of glycerin, the catalytic effect of Cu completely disappeared. It was also found that NG stored in a gum bottle or tube increased its yellow color, and at the same time its stability remarkably improved. When this yel NG was discolored by activated charcoal, its stability went back to the original value. The cause of the improved stability was attributed to the dissolution in NG of a resinous matter present in a gum bottle or tube

Livingston & Tomlinson (Ref 18) conducted research at PicArsn on the decompn of some US military expls at elevated temps

Destruction and Disposal of Explosives.

If a substance to be destroyed is unknown, Parpaillon (Ref 22) suggests to subject it to the following preliminary treatments:

a) Destruction of organic constituents by treating the substance with fuming nitric acid in order to be able to determine inorganic constituents, such as salts of Ca, K, Na, Pb, etc or simply ash content

b) Destruction of org constituents by burning the substance in the presence of a liquid, moderating the rate of burning and preventing the combustion to be transformed into deflagration or even detonation. For example, in detn of Na in TNT, the organic matter is destroyed by burning TNT previously moistened with a combustible liquid such as isopropanol. In cases of hard colloided materials, such as smokeless proplnts, a liquid, combustible gelatinizer for NC (such as castor oil in acetone) is recommended as a moderant in burning

Note: Utmost care is required in conducting the destruction of org matter by the above methods, in order to avoid losses of materials by splattering. The combustion methods cannot be used if it is desired to det the volatile inorganic salts, such as chlorides, chlorates, perchlorates, nitrates, etc

c) Treatment with concd caustic soln as, for example, in analysis of Centralite by distillation

[See Vol 2, p C132-L, item b) Distillation]

d) Treatment with dilute caustic solns in the presence of hydrogen peroxide, as for example, in detn of N in proplnts by the method of Devarda. Treatment of a colloided proplnt with dil caustic is rather time consuming because it requires that the sample be first dissolved in acetone and only then treated with caustic

e) Treatment of aromatic di- and/or trinitrocompds by the method of E.Berl of CIT, Pittsburgh, Pa and W.G.Berl. They dissolved the compd (contg between 2.5 & 3.0mg of N) in 5ml of 30% KOH (contg 2ml of 30% H_2O_2). The system was heated to 100° and 1ml of 30% H_2O_2 was added 5 times after an interval of 5mins. The mixt was cooled and 1 drop of 1% Osmium Tetraoxide soln was added to decomp excess H_2O_2. Three hundred mg of Devarda's alloy was added and the flask quickly connected to a steam distilling app. The distillate entered 25ml N/70 HCl. Steam distillation was commenced after the H_2 evolution ceased. After 30mins of lively steam distillation the HCl was back-titrated with N/70 NaOH. Berl & Berl found that the speed of decompn of nitrocompds increased with an increase in the number of nitro groups present. Mononitro derivs showed results which were too low (Ref: Private communication from Dr E.Berl to Dr B.T.Fedoroff about 1943). This method can be used also when it is desired to determine the N content of nitrocompds

f) Treatment of double-base proplnts by Parpaillon's modification of Berl's method: place in a 500ml Pyrex flask 1g of proplnt to test (in the form of scrapings, raspings or saw cuttings), wet it with 2ml of ethanol and add 4ml water & 10ml perhydrol (30% H_2O_2). While swirling the flask under a stream of running water, add pellets of KOH, one by one, allowing each pellet to dissolve before adding another. After adding a total of 10g, heat the flask on a water bath at 75° until practically complete cessation of the evolution of gas. Remove the flask from the bath, add gradually another 10-ml portion of perhydrol and resume heating in the water bath. Continue to heat until complete dissolution of org matter, except graphite & carbon black, if they are present. Filter and analyze the filtrate (usually colorless) for N content and inorganic constituents

Daniel, in his excellent, although rather obsolete dictionary of explosives (Ref 1) gives the following methods of destruction:

A) Substances based on inorganic nitrates and not contg chlorates, perchlorates, NG, NC, aromatic nitrocompds can be destroyed either by drowning them in water or by burning in the air. Examples of such expls are: some *Ammonals*, such as listed in Vol 1, p A289, Table, and *Black Powder* (See Vol 2, p B177-R)

a) Drowning can be conducted by transferring the expls directly from containers into a large volume of water, preferrably moving water, such as a stream. If still w is used (such as in a tank, pond, etc), its volume should be at least 8-10 times larger than the vol of the expl and the dumped material should be stirred to speed up the dissolution of nitrates. It was recommended not to discard these waters, but to use them for fertilization. The undissolved materials such as charcoal, coal, Al, etc accumulated in the tanks were moved to places unsuitable for cultivation, but if streams were used, the undissolved materials were left there expecting that they will be eventually carried away

b) Burning can be conducted by spreading the substance (previously removed from containers) on the ground (protected from wind) (See Burning Ground in Vol 2, p B357-R), in the form of a long narrow strip (train) and then igniting one end of this strip by a safety fuze or other device. If the substance is in the form of compressed cartridges, the envelopes are removed and the cartridges are placed in a long row, touching each other at the ends. The operator should not be standing close to the cartridges because they sometimes have a tendency to move around or jump

B) Destruction of substances contg chlorates or perchlorates but no NG, NC or aromatic nitrocompds can be conducted by the methods described in A, but with some modifications. When expls are drowned in still waters (such as in tanks), the w should not be allowed to evaporate, because dry salts are subject to spontaneous combustion, especially in the presence of org compds. As perchlorates are rather insol in cold w, hot w should be used for dissolving them. If destruction is conducted by burning, extreme caution should be

exercised and only small quantities burned at one time in order to avoid the possibility of deflagration or detonation

C) Substances contg NC and/or NG cannot be destroyed by drowning because NC & NG are insol in w and are dangerous to handle when dry. They can, however, be burned if the operation is conducted as follows:

When an expl is powdery, or can be reduced to such a state, it can be thrown in small portions into an open fire, while operator is behind a barricade. If an expl consists of hard cylindrical charges, remove them from their containers (such as paper cartridges), arrange the chges on the ground (free from pebbles) in a row (train), end to end. There should be no gaps betw the chges and no detonators present. Then a long piece of safety fuse is attached to one end of the train and the operator retires behind a barricade. The last precaution is necessary because sometimes a combustion transforms into a detonation

If cartridges are equipped with detonators, their destruction may be conducted by detonating each cartridge individually in the open, while keeping behind a barricade, or by placing several cartridges into a pit and then detonating them by influence (sympathetic detonation)

D) Extreme caution should be exercised in the destruction of cartridges of frozen NG expls. One should never try to break or pulverize expls in such cartridges or heat them in a fire. It is recommended to destroy them one by one by detonation, using strong initiators

E) Destruction of free NG can be done by mixing it carefully with sand and then igniting the resulting pasty mass in small portions by means of a safety fuse

F) Destruction of wet Guncotton can be done by detonating it with a primer using dry Guncotton as a booster. It can also be destroyed by throwing small portions into a fire. Detonation method is preferred

G) Some aromatic, water-insol, nitrocompds such as DNT, DNB or TNT can be destroyed by throwing small pieces of them, one at a time, into an open fire

H) Substances contg toxic, water-sol aromatic nitrocompds (such as PA), or their salts (such as Amm or K Picrates), but no NG, can be destroyed by drowning in such a great quantity of water that the concn of picrates in resulting solns is below their toxicity limits (See toxicity of picrates in Ref 29, pp 284, 1024 & 1052). PA & Picrates can also be destroyed by burning small quantities at a time, or by detonating a small chge at a time

I) Some safety expls contg AN but not contg NC and/or NG can be destroyed by breaking them into small pieces (after removing from cartridges) and burning small portions at a time. If it is undesirable to remove expls from cartridges, they may be lined up in a row, leaving a small gap betw the ends of each pair of cartridges and then detonating them by influence

J) Unexploded shells or bombs (such as "duds") can be destroyed directly in a field by detonating them by means of a cartridge of Dynamite placed in contact with them (See also Refs 3 & 7)

Daniel (Ref 1, pp 280-82), described also under the title "La Destruction des Engins Criminels" (Destruction of Criminal Devices) the following Belgian method for handling and destroying unexploded criminal bombs (bombes criminelles, in Fr):

When found, the bomb should be handled with great precaution and not be turned upside down, because some bombs, (called "bombes à renversement") explode on such movement. In order to prevent anybody else from such an action, the upper part of the bomb should be marked with chalk or pencil. Then the bomb is moved carefully into an excavation dug in a field, a cartridge of Dynamite or Tonite (provided with a primer and a 100 meter long piece of fuse), is placed over it and the ensemble covered with earth. Detonation of the cartridge explodes the contents of the bomb and shatters it to pieces

It was not recommended to open the unexploded criminal bombs unless they were previously kept for a while in liquids, such as chloroform or kerosene. Water could not be used because some bombs contained metallic K or Na as primers. Contact with w caused these metals to inflame. (A brief description of criminal bombs will be given under Explosives and Weapons Used by Anarchists and Revolutionists)

Instructions and recommendations for destruction in US of *unusable components*, such as ammunition, expls, proplnts & pyrotechnic items are given in Refs 4, 8, 9, 12, 13, 19, 19a,

23, 31 & 32)

The term "unusable component" that has replaced the term "unserviceable component", includes items that are in such a condition as to render them unfit for their intended purpose or for any other purpose. The unusable components may include materials or items of faulty manufacture (those that failed to pass inspection), deteriorated materials (those becoming ineffective or dangerous to handle), damaged items beyond repair (that can take place during manuf, shipping or storage), obsolescent materials (outdated, that have been replaced by newer, more effective items), captured materials and duds, which are items that have been properly initiated and failed to function, but are dangerous because they can explode any time later (Ref 9)

Expl items can be destroyed by one of the following methods:

I) *Drowning.* It is conducted by dumping them into a large and deep body of water, such as sea or lake. Drowning of partly sol expls, such as Black Powder, is described in Vol 2, p B177-R. Some unusable ammo and w-insol expls may be dumped into the sea but at a distance not less than 10 miles from shore. All items, however heavy, must be carefully removed from their wooden, plastic or metal containers before being dumped overboard, because there is danger that packed materials will be washed ashore, even from long distances. The material to be disposed is loaded on barges which are then towed to sea where a deep place or a ledge sloping seaward is located

II) *Burning.* It is described in Vol 2, p B358-R, as "Burning of Ammunition and Explosives for Destruction". A tract of land designated for such purpose is described in Vol 2, p B357-R, as "Burning Ground or Destruction Site". Disposal of bombs by burning is described in Vol 2, p B233-R

III) *Detonation or Explosion.* It can be conducted either by initiating the expls contained in ammo, or by the force of detonation of other expl materials placed in contact with the item. Ammo items which can be destroyed by these methods include: artillery shells, aerial bombs, mortar shells, shrapnel shells, photoflash bombs, antitank mines, rockets, grenades (hand and rifle), loaded metal components (such as fuzes, primers, detonators, boosters & bursters) and pyrotechnic items

For detonating these items, either the "non-electric" or "electric" firing system can be used. The non-electric firing system consists of a HE [such as TNT Demolition Block, Nitrostarch Explosive Block, Dynamite (40% & 50-60%), Composition C-3, etc], a non-electric blasting cap (which is inserted into the expl), and a length of safety fuse (or detonating cord) inserted in the cap. If TNT is used, the amt required is 0.5 lb for 100 lbs of unpacked ammo and 1 lb for 100 lb of ammo packed in wooden or metal containers. The electric firing system requires an electric detonator and a blasting machine or magnet exploder. All personnel conducting the operations should keep behind suitable barricades out of danger and should rigorously observe all US military safety regulations

The following is a list of military items and description of their destruction:

Artillery shells (except shrapnel shells), *aerial bombs* and *mortar shells* can be destroyed by mass detonation in pits, or directly on the ground when covered with earth as described in Vol 2, p B234 under "Bombs, Destruction". If the above items are scattered in a field and there is danger in moving them (such as if they are suspected to be provided with "antihandling fuzes"), each item should be destroyed separately, directly in the field. For this a charge of HE, placed as close as possible to the fuze or booster of the proj or bomb, is initiated by an electric or non-electric detonator. If, however, the fuze is buried in the ground and its excavation would be dangerous, it is recommended that the chge of HE be placed as close as possible to the bursting chge

Shrapnel shells cannot be destroyed by mass detonation in pits or on the ground as can *Bombs* (described under Bombs, Destruction of Vol 2, p B234) because of the danger of so many shrapnel balls flying around. These shells can be destroyed by the following methods: In the first method, the time fuze is removed from the shell and a blasting cap is placed in the flash tube until it touches the bottom, and the nose is closed by packing damp earth around the safety fuse (or electric wires) leading to the cap. Then the shell is placed horizontally inside a deep pit with vertical walls and its cap & expl chge are detonated. This will cause

the case of the shell to recoil with tremendous velocity and the shrapnel balls to be propelled into the opposite side of the pit. With this arrangement the case and the slugs will not leave the pit and endanger the life of personnel around it. In the second method of destruction, a shrapnel shell, equipped with a blasting cap, as above, is buried, base first, into the side of a six foot deep pit for about 3/4 of its length and with the nose pointing slightly downward. When detonated, the base is driven farther into the earth and the slugs propelled into the low part of the pit. By using this method and electric blasting caps, connected in series, several shrapnel shells can be detonated simultaneously in the same pit

Photoflash bombs can be destroyed by detonating them as described for aerial bombs in Vol 2, p B234 under Bombs, Destruction of. The operators should be instructed not to look in the direction of the bombs when they are being detonated as the flash is very blinding

Antitank mines can be destroyed in the manner similar to aerial bombs but fewer items should be destroyed at one time due to the greater amt of expl per unit weight of ammo

Rockets can also be destroyed by detonation but not before their motors are removed. This is in order to prevent rockets from shooting out of the pits

Grenades (hand- and rifle grenades) can be destroyed either by burning (generally HE grenades) or detonation (generally applied to other types). HEAT rifle grenades should be destroyed in a manner similar to that used for shaped-charge ammo. If by burning, grenades are put in the fire one at a time after each has exploded. The burning pit 2 ft square by 3 ft deep should be fitted loosely with a steel plate or heavy board cover. When destroying grenades by detonation, not more than 20 should be placed at one time in a destruction pit 4 ft deep. With the grenades in close contact with each other, three ½-lb TNT blocks taped together are placed on top of the pile. A type II special elec blasting cap or a type I special nonelectric blasting cap fitted with several ft of safety fuse is attached to one of the TNT blocks. Before deton the pile is covered with one ft of lightly tamped earth (Refs 27a & 34a)

Loaded metal components (which include *fuzes, primers, detonators, boosters & bursters)* can be destroyed by:
a) detonation b) burning or c) dumping at sea. When destroying by burning, caution must be exercised in putting the components into the fire, because normal action cannot be expected. The explosion of each component should be heard before putting in another. When destroying by detonation, small numbers, in contact with each other and in contact with one or more demolition blocks, are detonated in a pit or trench previously covered with logs, earth or other suitable cover. Primers and detonators are destroyed only by burning using the same precautions & procedures as for the destruction of small-arms ammo (Refs 27a & 34a)

Chemical Munitions. See Vol 2, p C176-R

Pyrotechnic items can be destroyed either by burning or by detonation (Ref 19a) as follows:
a) Burning. Pile the packed or unpacked pyro items on combustible materials (such as paper, excelsior, wood shavings, straw, sawdust, rags, brush, etc) on dry ground. Pour some oil or kerosene, ignite at one end and take cover. Explosive pyrotechnic items, such as photoflash bombs should not be included in the pile because they might produce a blast, which may scatter other pyrotechnic items and produce fragments flying a distance up to 200 yds. These bombs are usually destroyed as described in Vol 2, p B234
b) Detonation. Remove pyrotechnic items from their packings and pile them in a trench or a depression of the ground. Place TNT demolition charges equally distributed thruout the pile, using ca 0.5lb TNT for each 100 lbs of pyrotechnics. Prime each chge of TNT for simultaneous detonation as described in Ref 19a, take cover and fire. If some items fail to be detonated, they will be destroyed by fire which usually follows the blast

See also Addnl Ref T

Alphabetical List of Destruction and Disposal Procedures for Explosives and Propellants.

Amatol. See Vol 1, p A162-L

Ammonium Picrate. See Explosive D or Dunnite

Azide, Lead (LA). Laboratory procedures for disposal are given in Vol 1: p A574-R (Kankakee Ordnance Works method); p A574-R (W.

H.Rinkenbach's method); p A575-L (British, DuPont and PicArsn methods)

Destruction of LA on a plant scale (Ref 13) can be done by gradually adding water-wet LA to a wooden tank contg 10% aq NaOH soln in quantity corresponding to 50 lbs soln per each 1 lb of LA. Agitate, while adding, either with a current of air or a wooden paddle. Allow the mixt to stand at least 16 hrs (usually overnight), while agitating it gently once in a while. The following reaction takes place:

$Pb(N_3)_2 + 2\,NaOH \longrightarrow Pb(OH)_2 + 2NaN_3$

The resulting mixt consists of insol Pb hydroxide and sol Na azide, both are non expl, and may be dumped into a sump

For removing LA which settled in dust chambers, crevices, etc, soak the powder repeatedly with a 10% aq soln of Amm acetate, followed by washing with water. Collect the washings and add a 10% aq Na dichromate soln until there is no further pptn of yellow Pb chromate

For removing LA from catchtraps, sinks, etc fill the trap two or three times with a 10% aq NaOH soln, allowing the soln to remain for several hours each time. Rinse with water and remove the insol Pb hydroxide with 2 or 3 washes of dil AcOH, allowing the liquid to remain for several hours each time. Finally rinse the catchtrap or sink with w

LA can also be destroyed by adding to it a 10% aq Na nitrite soln and then small amts of 10% AcOH with stirring until all effervescence ceases. If any residue remains after pouring off the supernatant liquid, add another portion of NaOH soln and then AcOH. Precautions should be taken against the nitrogen peroxide fumes evolved. This can be achieved by wearing a gas mask

Destruction of LA when in mixts with MF is described under Fulminate, Mercury, p D31-R

Other expl azides such as those of Ag, Ba, Ca, Cd, Co, Hg, Ni, Sr & Zn can be destroyed by similar methods

Black Powder (BkPdr). See Vol 2, pp 177-R to B178-R

Cellulose Nitrate or Nitrocellulose (NC). Decompn of NC can be achieved by treating it with a caustic soln. The main product of reaction is regenerated cellulose, as for example in the case of Pyrocellulose:

$C_{24}H_{30}O_{10}(ONO_2)_{10} + 10\,NaNO_2 \longrightarrow$

$C_{24}H_{30}O_{30}(OH)_{10} + 10NaNO_3$

There are, however, other products of reaction. The procedure is, accdg to Ref 13, as follows: Prepare in a wooden or iron vessel a concd soln of Na hydroxide [50 lbs NaOH dissolved in 55 gallons (460 lbs) of water for each 100 lbs of NC to be destroyed] and heat the soln to 70° (160°F) by means of a steam jet, which would also serve as an agitator. Add the unusable NC at such a rate that the reaction does not become too vigorous. After the final addn, continue the agitation for 15 mins and allow the residue to settle. Discharge the supernatant liquid to a sump, add to the residue more NaOH soln and treat as above

Accdg to the Eastern Laboratory of the DuPont Co, refluxing of NC with 15% aq Na sulfide soln (1-1.5 parts of Na_2S per 1 part NC) on a steam-bath resulted in decompn of NC to the extent of 98.9%

Composition A. See Vol 3, p C474-L

Composition B. See Vol 3, p C477-R

Composition C. See Vol 3, p C484-L

Composition C-3. See Vol 3, p C484-R

Cyclonite. See Cyclotrimethylenetrinitramine

Cyclotetramethylenetetranitramine or HMX. See Vol 3, p C605-R

Cyclotols. See Vol 3, p C478-L

Cyclotrimethylenetrinitriamine;Cyclonite or RDX. See Vol 3, p C621-L

Cyclotrimethylenetrinitrosamine or R-Salt. No info on its disposal is available

Dinitrobenzene (DNBz). No info at our disposal

Dinitrotoluene (DNT). See Toluene, Dinitro

Dunnite or Ammonium Picrate. See Explosive D

Dynamites. According to Leitch & Moyer of the US Bur Mines (Ref 11), the best method for destruction of dynamites is by burning. Quantities of regular (powdery) dynamites to be burned at one time should not exceed 100 lbs and of permissible gelatines 10 lbs. If the burning ground (See "Burning Ground or Destruction Site" in Vol 2, p B357-R) is small or in a crowded area, smaller quantities should be used. When more than the above maximum quantities should be destroyed, a new burning space should be selected for each lot as it is not safe to place expls on ground still hot from the preceding burning

Procedure: Spread on the ground a layer of dry combustible material (such as excelsior, wood

shavings, straw, sawdust, etc) and place on top of it the paper cartridges in a single layer which should not be thicker than 2 inches. If the cartridges are of larger diameter, as often used in quarry blasting, they should be cut into long and narrow pieces not thicker than 2 in. If the dynamite is wet or rather hard to ignite, sprinkle some kerosene on pile. Before igniting, lay a train of paper (or similar ignitable material), preferrably on the down-wind side of the pile. This train should be long enough to permit the operator to reach a safe place after igniting the paper. Dynamites usually burn quietly, with a bluish flame. No attempt should be made to return to the burning ground as long as any flame or smoke is observed. If solid, unburned pieces remain on the ground (as sometimes happens, especially if dynamite is wet), it is dangerous to poke about debris, or attempt to handle these pieces for reburning until it is certain that they are cold. Burn the dynamite containers separately

Note: If any livestock grazes around the burning ground, it is advisable either to put a wire fence around the ground or to plow the ground as soon as all dynamites have burned and the site is cooled. This is because it is believed that some of the substances left after burning are toxic

Explosive D; Dunnite; Ammonium Picrate or Ammonium Trinitrophenolate. Accdg to Ref 13, it can be destroyed by means of a concd soln of Na sulfide, using for each 100 lbs of Expl D 400 lbs of $Na_2S.9H_2O$ dissolved in 300 gallons (2490 lbs) of water. Prepare this soln in a large wooden or iron vessel and, while agitating it with a current of air or a wooden paddle, add Expl D in small portions. Continue the agitation for at least 1 hr after adding the last portion. After allowing the residue to settle, discharge the supernatant liquid into a sump and send the residue to the burning ground

A gas mask should be worn during these operations and the building should be adequately ventilated

Fulminate, Mercury or Mercuric Fulminate (MF). The decompn of MF by Na thiosulfate (hypo) is based on the following reaction:

$$2H_2O + Hg(CNO)_2 + 2Na_2S_2O_3 = Hg(S_2O_3)_2 + 4NaOH + (CN)_2$$

As the last member at the right is a poisonous gas, cyanogen, it is recommended that the operator wear a gas mask or, at least, keep himself windward to the reaction vessel

Accdg to Davis (Ref 8a, pp 408-09), the reactions proceed as follows:

$$Hg(ONC)_2 + 2Na_2S_2O_3 + H_2O \longrightarrow$$

$$Hg(S_2O_3)_2 + 2NaOH + NaCN + NaNCO \longrightarrow$$

$$HgSO_4 + Na_2SO_4 + 2NaNCS + H_2O$$

which are products resulting from reaction of cyanogen & caustic soda and those from a secondary reaction when the soln is allowed to stand

The procedure, as described in Ref 13, is as follows: Place in an earthenware vessel a quantity of aq Na thiosulfate soln (20% by wt of $Na_2S_2O_3.5H_2O$) equal to at least 10 times the wt of MF to be destroyed. While stirring the soln with a current of compressed air (or a wooden paddle) add the water-wet MF and continue to agitate for about 2 hrs. Discharge the liquid to a sump

MF accumulated in dust chambers, hoods, etc can be removed by soaking it with Na thiosulfate soln and then mopping up the resulting inert soln

If MF is mixed with LA, treat the mixt with 20% Na thiosulfate as described above and allow the sludge to settle overnight. Discharge the supernatant liquor into a sump and treat the residue with 10% NaOH soln as described under Azide, Lead. Allow the mixt to stand overnight, discharge the liquid into a sump and dispose of the sludge

To remove a mixt of MF & LA from catch-traps and sinks, fill the trap with 20% Na thiosulfate soln and allow the soln to remain for several hours. Repeat with a fresh portion of thiosulfate soln and flush with water. This will remove MF. For removing LA fill the trap with 2 or 3 portions of 10% NaOH soln and remove the resulting ppt of Pb hydroxide by dissolving it in AcOH and then flushing the trap and sink with water

Glycerine Nitrate or Nitroglycerin (NG). Accdg to Ref 2, NG may be destroyed by the so-called "sulfur solution", which is prepd by dissolving S in aq Na carbonate soln. Accdg to Mellor (Ref 5a), S dissolves in Na carbonate with formation of polysulfide, thiosulfate and carbon dioxide:

$$8S + 3Na_2CO_3 \longrightarrow 2Na_2S_3 + Na_2S_2O_3 + 3CO_2$$

A similar soln can be prepd by dissolving

S in a hot caustic soln:
$8S + 6KOH \longrightarrow 2K_2S_3 + K_2S_2O_3 + 3H_2O$

Accdg to Ref 13, NG can be destroyed by treating it with aq Na sulfide soln as follows: For each 100 lbs of unwanted NG prepare a soln of 175 lbs hydrated Na sulfide ($Na_2S.9H_2O$) in 100 gallons (830 lbs) of water in an iron or enameled vessel. Agitate the soln by means of compressed air or with wooden paddles and add slowly the NG. Considerable heat is evolved by the decompn which is, accdg to Naoúm (Ref 4, p 122) and Davis (Ref 8a, p 208) saponification with formation of glycerin. There is no danger in this process as long as the agitation is maintained and the temp held below 50°. Continue the agitation until a sample withdrawn from the reaction vessel shows no oily droplets on standing. Allow the sludge to settle and discharge the supernatant liquid into a sump. If any oily liquid remains at the bottom of the vessel, add a little more Na sulfide soln and repeat the operation as above. Remove the sludge to a burning ground and wash the vessel, first with Na sulfide soln, and then with water
Note: Part or all Na sulfide may be substituted by Na or K hydroxide, but the reaction proceeds much more vigorously and more care should be taken to keep the temp below 50°. Accdg to Ref 4, p 122 & Ref 8, p 208, the following main reaction takes place:
$C_3H_5(ONO_2)_3 + 5KOH \longrightarrow KNO_3 + 2KNO_2 + CH_3COOK + HCOOK + 3H_2O$, but there are also side reactions with formation of aldehyde resins, oxalic acid and ammonia

For laboratory destruction of NG, where comparatively small amts are handled, dissolve NG in acetone or denatured alcohol and add the soln very gradually, in small increments, to aq NaOH soln

HMX. See Cyclotetramethylenetetranitramine

Lead Azide. See Azide, Lead

Mercuric Fulminate. See Fulminate, Mercury

Nitrocellulose. See Cellulose Nitrate

Nitroglycerin. See Glycerin Nitrate

Pentaerythritol Tetranitrate (PETN). Decompn of PETN is achieved by treating it with Na sulfide soln. The reaction of saponification proceeds in the manner discussed under Glycerin Nitrate. Accdg to Ref 13, for destruction of each 100 lbs of unwanted PETN, dissolve it in 120 gal (789 lbs) of technical grade acetone using an iron or wooden vessel, provided with a steam jacket. Bring the temp of soln to ca 40° (105°F) and, while agitating it with compressed air or wooden paddles, add gradually the soln of 250 lbs of $Na_2S.9H_2O$ in 60 gallons (500 lbs) of water which has been preheated to 80° (175°F). Addn of Na sulfide soln should be regulated so that the temp of the reaction mixt does not rise above the bp of acetone (56.5°). After the addn of sulfide soln, continue the agitation for 30 mins and allow the mixt to settle. Discharge the supernatant liquid to a sump and transfer the sludge to a burning ground
Note: Tests conducted at the Eastern Laboratory of the DuPont Co, showed that PETN heated with an aq Na sulfide soln (1-1.5 parts Na_2S per 1 part of PETN), under reflux for 3 hrs, using steam coils and agitation, resulted in 98.8% decomp of PETN

PETN. See Pentaerythritol Tetranitrate

Phenol, Trinitro or Picric Acid (PA). Destruction of PA is based on its decompn by an aq soln of a mixt of Na hydroxide and Na sulfide. As some H_2S & NH_3 are evolved during the treatment, the operator should wear a mask and adequate ventilation should be provided. Davis (Ref 8a) does not give the equation of decompn. Accdg to Ref 13, prepare for each 100 lbs of unwanted PA, a soln of 20 lbs NaOH in 240 gallons (2000 lbs) of water and 420 lbs of hydrated Na sulfide ($Na_2S.9H_2O$). Agitate the soln with compressed air or wooden paddles and add the PA is small increments. Continue the agitation for 30 mins after the last addn of PA and allow to settle. Transfer the supernatent liquor to a sump and, if any insol residue remains in the container, treat it with addnl hydroxide-sulfide soln or send it to a burning ground

Picric Acid (PA). See Phenol, Trinitro

Propellant, Smokeless. Use the same procedure as described for decompn of Cellulose Nitrate. Tests conducted at the Eastern Laboratory of the DuPont Co showed that 96.6% of smokeless propInt was decompd after heating for 1 hr on a steam bath with a 15% soln of Na sulfide (1-1.5 parts Na_2S per 1 part of propInt) (Ref 13)

RDX. See Cyclotrimethylenetrinitramine

R-Salt. See Cyclotrimethylenetrinitrosamine

Smokeless Propellant. See Propellant, Smokeless

Tetryl or 2, 5, 6-Trinitrophenylemethylnitramine.

It can be decomposed by heating it with an aq Na sulfide soln. Davis (Ref 8a), does not give the equation for this decompn. Accdg to Ref 13, 100 lbs of Tetryl required for its destruction 225 lbs of hydrated Na sulfide ($Na_2S.9H_2O$), dissolved in 110 gallons (915 lbs) of w. Prepare this soln in a wooden or iron tank and heat it to 80-90° (175-195°F) by means of a steam coil or a steam jet. Add Tetryl in small increments, while agitating the soln by compressed air or wooden paddles. Continue agitation until dissolution seems to be complete and allow to stand. Discharge the supernatant liquor to a sump and send the ppt (if any) to a burning ground

Tests conducted at the Eastern Laboratory of the DuPont Co, showed that Tetryl heated in a 5% Na_2S soln, for 3 hrs with agitation, was not completely decompd, but left a heavy black residue contg some Tetryl

Toluene, Dinitro or Dinitrotoluene (DNT). It can be decompd by treatment with an aqueous alcoholic soln of Na hydroxide. No equation of of decompn is given by Davis (Ref 8a). Accdg to Ref 13, the following procedure can be followed: Dissolve in a wooden or iron vessel 50 lbs of NaOH in 55 gallons (460 lbs) of w and add 225 gallons (ca 1810 lbs) of 95% denatured alcohol. Heat the soln to 45° (112°F) or somewhat higher, and, while agitating it by compressed air or wooden paddles, add 100 lbs of DNT in portions. Continue agitation for 15 mins after the last addn and allow to settle. Discharge the supernatant liquid to a sump and treat the residue (if any) with alc NaOH as above

Note: DNT, unlike TNT, cannot be destroyed by Na sulfite soln (See under Toluene, Trinitro)

Toluene, Trinitro or Trinitrotoluene (TNT). The usual method of its destruction is by treatment with an aq Na sulfide soln. The procedure is, accdg to Ref 13, as follows: Dissolve 425 lbs of $Na_2S.9H_2O$ in 300 gallons (2500 lbs) of water in a wooden or iron vessel and, while agitating with compressed air or wooden paddles, add 100 lbs of TNT in small portions. Continue the agitation for 30 mins after final addn of TNT and allow to settle. Discharge the supernatant liquor to a sump and treat the ppt with addnl sulfide soln as described above. Any remaining sludge can be burned

DNT may also be destroyed by heating it with live steam in a satd soln of $Na_2SO_3.7H_2O$. The reaction proceeds, accdg to Davis (Ref 8a, p 146), as follows:

$$H_3C.C_6H_2(NO_2)_3 + Na_2SO_3 \rightarrow NaNO_3 + H_3C.C_6H_2(NO_2)_2.SO_2ONa$$

The resulting dinitrotoluene sodium sulfonates are not expl and are sol in w

DNT may also be destroyed with aq alkalies. The resulting products are not dangerous as long as they are in soln, but when dried they can explode on heating

Trinitrophenol. See Phenol, Trinitro.

Trinitrophenol, Ammonium Salt. See Explosive D

Trinitrotoluene (TNT). See Toluene, Trinitro

Refs: 1)Daniel (1902), 189-92 & 280-82 (Destruction of expls & ammo) 2)E.M. Weaver "Military Explosives", Wiley, NY (1907), 153 (Destruction of NG) 3)M.M. Kostevich, "High Explosives and Powder Burning Ground", Imp d'Art Voltaire, Paris (1927) 4)US Army Chief of Ordnance, "Safety and Storage Manual for Explosives and Ammunition" OO Form No **5994**, Washington, DC (1928), secn XVII, pp 8-10 4a) Naoum, NG (1928), 122 5)W.A.Noyes Jr & W.E.Vaughan, Chem Revs **7**, 239-58 (1930) (Thermal, electronic & photochemical decompns of solid substances, such as K chlorate, Ag oxide, oxalic acid and some carbonates) 5a)Mellor **10** (1930), 100 & 103 6)Marshall **3** (1932), 208 (Decompn of expls as detd at Woolwich Arsenal) 6a)M.Lambrey, MP **26**, 101-18 (1934-35) (Method for detg decompn of expls in storage) 7)Pepin Lehalleur (1935) 448-64 (Destruction of expls & ammunition) 7a)Clift & Fedoroff **1** (1942), Chap XII, pp 15-19 {Detailed description and drawing of weighing thermo-balance, system Guichard [See Bull Fr **39**, 1113 (1926) & **3**, 115 (1936) and also Annales de Chimie **9**, 324 (1938)] which can be used for detn of loss of wt on heating not only at a desired constant temp (as a function of time), but also as a function of constantly rising temp. This balance permits to det stability as well as decompn points of proplnts, expls & pyrotechnic compns. It has been used successfully in France} 8)Anon, "US Bombs and Fuzes", US Navy Bomb Disposal School, Washington, DC (1943) (Destruction of bombs & fuzes) 8a)Davis (1943), 208 & 408-09 (Decomposition

of NG & MF) 9)Anon, "Ammunition Inspection Guide", War Dept Tech Manual TM **9-1904**, Wash, DC (1944) 748-78 10)M.M.Kostevitch, "Methods for the Safe Burning-out of Lyddite and Other Shells", Talleres Gráficos, Buenos Aires (1944) 11)R.D.Leitch & P.R.Moyer, US Bur Mines Info Circ IC **7335** (1945), 6 pp (Destruction of damaged, deteriorated, or unwanted commercial expls) 12)Anon, "Destruction of Unexploded Ammunition in Theaters of Operation", US War Dept Tech Bull TB ORD **249**, Wash, DC (1945), 386-87 13)A.J.Clear & W.H. Rinkenbach, PATR **1556** (1945) (Procedures for decompg waste HE's) 14)A.J.B.Robertson, TrFaradSoc **44**, 677-82 (1948) & CA **43**, 2775 (1949) (Thermal decompn of Ethylenedinitramine & Tetryl) 15)Ibid, JSCI **67**, 221-24 (1948) & CA **43**, 405 (1949) (Thermal decompn of PETN, NG, Ethylenediamine Dinitrate & AN) 16)Ibid, TrFaradSoc **45**, 85-93 (1949) & CA **43**, 5187 (1949) (Thermal decompn of RDX & HMX) 17)A. J.B.Robertson, "Third Symposium on Combustion and Flame and Explosion Phenomena", Williams & Wilkins, Baltimore, Md (1949), 545 -51 (Thermal initiation of expln in liquid expls) 18)S.Livingston & W.R.Tomlinson Jr, "Fundamental Research on Explosives. Decomposition of Explosives at Elevated Temperatures", PATR **1737** (1949) 19)Anon, "Artillery Ammunition", US Army Tech Manual TM **9-1901** (1950). 386 -87 19a)Anon, "Military Pyrotechnics", TM **9-1981** (1951), 158-59 20)A.D.Yoffe, PrRoySoc **A208**, 188-99 (1951) & CA **46**, 5845 (1952) (Thermal decompn of azides) 21)J.D.Blackwood & F.P.Bowden, PrRoySoc **A213**, 285-306 (1952) & CA **46**, 10623 (1952) (Initiation and thermal decompn of Gunpowder) 22)M.Parpaillon, MP **34**, 419-20 (1952) (Preliminary treatments of expls before destruction) 23)Anon, "Explosive Ordnance Reconnaissance and Disposal", US Army Field Manual FM **9-40** (1953) 24)A. Schmidt & H.Behrens, "CR 27^{e} Congr Intern Chim Industrielle, Brussels **1954**, **3**; Industrie Chimique Belge **20**, Spec No,296-98 (1955) & CA **50**, 17454 (1956) (Decompn of proplnts at low pressures) 25)M.M.Pavlyuchenko, ZhFiz-Khim **29**, 996-1000 (1955) & CA **50**, 16299 (1956) (Kinetics of decompn of expls) 26)P.A. Bel'kevich, ZhFizKhim **30**, 706-07 (1956) & CA **50**, 16299 (1956) (Critical remarks on Pavlyuchenko's paper) 27)M.A.Cook & M.T.Abegg, IEC **48**, 1090-95 (1956) & CA **50**, 11670-71 (1956) (Isothermal decompn of expls) 27a)Anon, "Care, Handling, Preservation, and Destruction of Ammunition", TM 9-1903/TO 11A-1-37 (1956) 28)R.Sandri, CanJChem **34**, 324-30 (1956) & CA **50**, 12482 (1956) (Flame propagation in ozone) 29)Sax (1957), 161-64 (Destruction of expls) 30)F.C.Iklé, "The Social Impact of Bomb Destruction", Univ of Oklahoma Press, Norman, Okla (1958) 31)Anon, "Ordnance Service in the Field", US Army Field Manual FM **9-1** (1959) (Destruction of ammo) 32)Anon, "Ordnance Ammunition Service", FM **9-5** (1959) (Destruction of ammo) 33)A.B.Amster, "Relationship Between Decomposition Kinetics and Sensitivity" (U), Stanford Research Institute, Menlo Park, California,Repts (1962), Contract No **Nonr 3760(00)** (Conf, not used as a source of info) 34)P.W.M.Jacobs & A.R.T.Kureishy, "Kinetics of Thermal and Photochemical Decomposition of Some Alkali Metal Azides", Imperial College, London, England, Final Tech Rept (1964); Contract **DA-91-591-EUC-2059** 34a)Anon, "Care, Handling, Preservation and Destruction of Ammunition", TM **9-1300-206** (1964) 35)Anon, "Investigations of the Mechanisms of Decomposition, Combustion and Detonation of Solids", Aerojet-General Corp, Sacramento, Calif, Final Rept (1965); Contract **AF 49(638)-851**

Additional Refs: A)C.N.Hinschelwood & E.J. Bowen, Phil Mag **40**, 569-78 (1920) (Thermal decompn of crystalline K permanganate, K perchlorate, Amm dichromate & Tetranitroaniline) B)A.Sieverts & H.Theberath, ZPhysChemie **100**, 463-76 (1922) (Thermal decompn of Ag permangante) C)C.N.Hinschelwood & E.J. Bowan, PrRoySoc **99A**, 203-12 (1922) (Detn of the influence of physical conditions on the velocity of decompn of certain cryst compds, such as permanganates, perchlorates and dichromates, using a specially constructed app) D)O.Poppenberg, ZAngChem **36**, 80-85 (1923) (Decompn of some expls) E)I.Ishikawa & T. Hakisawa, TôhokuImpUniv Sci Repts, Series I, **22**, 1205-28 (1933) (Detn of thermal velocity of decompn of K & Na perchlorates by means of a special app called thermo-balance) F)A.F. Gill, CanJRes **10**, 703-12 (1934) (Thermal dehydration and decompn of certain minerals & salts using an app consisting of conbination of

calcining furnace and weighing balance) G)P. Lesauskis MAF **14**, 487-501 (1935) (Translated from Rivista di Artiglieria e Genio, Feb-March, 1931) (The rate of decompn of a smokeless proplnt depends on the nature of proplnt, the temp of heating and the presence of impurities. The presence of moisture, alkalies or acids speeds up the decompn, while substances like DPhA, Centralites or Acardites slows it down) H)A.Haid & A.Schmidt, SS **34**, 38-41 (1939) & CA **33**, 3589 (1939) (Description of a special method for analysis of gaseous products evolved on decompn or expln) I)W.E.Garner, Chim & Ind (Paris) **45**, Suppl to No 3, pp 111-18 (1941) (Thermal decompn of solid expls and resulting explns) J)H.Muraour & J.Basset, Chim & Ind **45**, Suppl to No 3, pp 218-24 (1941) (Influence of high pressure on the progress of decompn of expls, particularly in initiating expls) K)Yu.N.Riabinin, ZhFizKhim **20**, 1363-66 (1946) & CA **41**, 2970 (1947) (Thermal decompn of Ba azide at pressures up to 4500 kg/cm^2) L)A.F.Belyaev & A.E.Belyaeva, Zh FizKhim **20**, 1381-89 (1946) & CA **41**, 2970 (1947) (Combustion of MF) M)Yu.N.Riabinin, DoklAkadN **58**, 245-48 (1947) & CA **44**, 7539 (1950) (Influence of pressure on the velocity of thermal decompn of expls) N)J. Vaughan & L.Phillips, JCS **1947**, 1560-65 & CA **42**, 3571 (1948) (Thermal decompn in a vacuum of certain mono- and dinitrobenzene -diazo-oxides, with a note on the kinetics of thermal breakdown of 2-nitrobenzene-4-diazo-1 -oxide) O)P.C.Condit & R.L.Haynor, IEC **41**, 1700-04 (1949) & CA **43**, 9045 (1949) (Thermal decompn of nitrobenzene and nitroxylene) P)J. Vaughan & L.Phillips, JCS **1949**. 2736-45 & CA **44**, 5090 (1950) (Study of the delayed expln of MF and kinetics of thermal decompn of MF in vacuum) Q)J.F.von Gizycki, ChemZtg **74**, 649 -51 (1950) & CA **45**, 2209 (1951) (Study of slow thermal decompn of NC at 100° by measuring wt loss and changes in viscosity and N content) R)T.Hikita, Japan Sci Rev, Eng Sci **1**, No 2, 23 -24 (1950) & CA **45**, 2669 (1951) (Study of thermal decompn of some expls) (See also in the text) S)A.Glasner & A.E.Simchen, Bull Fr [5], **18**, 233-38 (1951) (Thermal decompn of K perchlorate in vacuum using a special app) (See also in the text) T)Anon, "Military Pyrotechnic Series. Part 2. Safety, Procedures and Glossary", AMC Pamphlet **706-186**, Headquarters, US Army Materiel Command, Washington, DC 20315 (1963), pp 15-16 U)Blasters' Hdb (1966), pp 467, 468, 471; (1958), 148-54; and (1952), 140-44

Decontaminating Agent DS2. This is a clear soln consisting of diethylenetriamine 70, ethyleneglycol monomethylether 28 & sodium hydroxide 2%. DS2 is flammable and contaminating to the skin. Inhalation of its vapors should also be avoided. DS2 can be removed readily from the skin without harmful effect by immediately washing with water. It is noncorrosive to most metals

Uses. DS2 is an effective decontaminant for all toxic chemical agents. It can be used between temps of -25°F (-31.6°C) & 125°F (51.6°C). One application is used on contaminated surfaces followed in 30 mins by flushing with water. As a general-purpose decontaminant, DS2 reacts with **GB** (See Vol 2, p C167-R) and **HD** (Vol 2, p C168-L) agents to reduce their hazards within 5 mins of application

See also DANC solution and Decontaminating Agent STB

Refs: 1)Anon, "Military Chemistry and Chemical Agents", TM3-215 and AFM 355-7, US Depts of the Army and the Air Force (1963), 87 2)G. H.Fielding, US Naval Res Lab Rept **6191** (1964) (Field studies with DS2) 3)US Military Specification MIL-D-50030C (May 1965)

Decontaminating Agent STB (also referred to as Bleach, Supertropical Bleach, Bleaching Powder, Bleaching Material, and Chlorinated Lime). STB is a wh powder contg 30% of available chlorine. It may be mixed with water to form a slurry for manual application (50/50-STB/water) or for use in a power-driven decontaminating app (40/60-STB/water). Antiset added to the slurry holds the ingredients in suspension and prevents settling out of calcium which would clog pipes and strainers

Preparation. Sufficient Ca oxide is mixed with Ca chlorohypochlorite to reduce the moisture content of STB to less than 3%. This results in greatly increased stability. The product will withstand 6 wks storage at 158°F (70°C) with very little loss of available Cl. STB looses about 2% available Cl per year at this temp

Uses. STB is an effective decontaminant for **H** (See Vol 2, p C168-L), **L** (Vol 2, p C168-R) and **ED** (Vol 2, p C167-R) chemical agents. STB contg $Ca(OH)_2$ & hypochlorite is also effective against **G nerve agents** (dimethyl-aminoethoxy-cyanophosphine oxide, methyliso-propoxyfluoro-phosphine oxide & methyl-pinacolyloxyfluoro-phosphine oxide)

See also DANC Solution and Decontaminating Agent DS2

Refs: 1)Anon, "Military Chemistry and Chemical Agents", TM3-215 and AFM 355-7, US Depts of the Army and the Air Force (1963), 84-86 2)US Military Specification, MIL-D-12468A

Decontamination. The act of removing the biological, chemical or radiological contamination from, or neutralizing it on, a person, object or area

Ref: Glossary of Ord (1959), 70-L

Decontamination of Explosive Establishments. This subject is discussed in the following refs

Refs: 1)US Dept of the Army **TM3-220,** "Decontamination" (Nov 1943) 2)US Army Supply Bulletin **SB5-52**, "Decontamination Procedures" (July 1945) 3)US Depts of the Army and the Air Force **TM3-215** & **AFM 355-7**, "Military Chemistry and Chemical Agents" (1963) 4)AMC Safety Manual AMCR 385-224, Sect 3, Headquarters, US Army Materiel Command, Washington, DC (June 1964)

Decontamination of Waters Containing Dissolved Explosive Wastes. Accdg to McDonald (Ref), decontamination may be achieved by means of a quarternary salt of a halide of Group V, nonmetal. The general formula of such salt is $R.A.M.R^1.R^{11}.R^{111}.X$, in which: R is an alkyl, aryl or an alkyl-substituted aryl group; A is an alkylene group or a free valence bond; M is nitrogen or phosphorus; X is halogen; R^1 & R^{11} are alkyl groups or H; and R^{111} is an alkyl or amidoalkyl group, preferrably contg 8-22 carbon atoms. A satisfactory compd is *benzyldimethylhexadecylammonium chloride.* Such salts are satisfactory when the expl waste has slight water soly and a dissocn constant of the order of 5×10^{-15} or greater. They can be added to a tank along with the waste water. As a result of such treatment, the expl waste would settle as an insol addn compd. Increased efficiency can be obtained by use of a settling tank and addn of a settling agent

Ref: L.McDonald, USP 2647084 (1953) & CA **47**, 10853 (1953)

Decoppering Agent. It is a material (such as finely divided tin) included in a propelling charge or a material inserted in the gun chamber with the propelling charge, for the purpose of removing the "coppering" from the surface of the bore. The term "coppering" is known as the metal fouling accumulated in the bore of a weapon due to repeated firings. The metal is deposited from the rotating bands or jackets of the projectiles (Ref 7)

Some info on American decoppering agents, particularly of lead foil laminated to the cartridge bag is given in Refs 2 & 3

The Russians (Refs 4 & 5), used as their "protivo-omednitel'" or "Razmednitel'" a coiled lead wire or strips of flat lead placed betw the proplnt chge and the base of the proj. Tin or alloys of Pb-Sb or Pb-Zn were also used. It was believed that the decoppering agent fused, on passage of the proj thru the bore, with the Cu alloys which became more brittle and easier to remove than straight Cu or guilding metal

The Germans (Refs 1 & 6) used as their "Enkupferungsmittel" in some 37mm HE rounds, an alloy consisting of tin 60, lead 38, bismuth 1.8 & antimony 0.2% and in 40mm HE rounds an alloy contg tin 61 & lead 39%. Many large caliber rounds contained lead wire wrapped around the proplnt bag or placed betw the bag and proj. Upon deflagration of proplnt chge, the Cu of the rotating band formed a brittle alloy and this was deposited on the surface of the bore. When the next chge contg no decoppering agent was fired, the rotating band shattered the brittle alloy, thus clearing the gun barrel

(See also Fouling of Guns and Antifouling Agents)

Refs: 1)W.R.Tomlinson, "Chemical Compositions of Materials used in German Ammunition", PATR **1555** (1945), p 30 2)R.W.Scharf, "Decoppering Agent for 105mm Howitzer Dual Granulation Charge", PATR **1594** (1946) 3)J.S.Musgrave, "Decoppering Agent for 105mm Howitzer Dual Granulation Charge", PATR **1679** (1948)

4)Blinov, Vol **5** (1949), 163 5)B.T.Fedoroff et al, "Dictionary of Russian Ammunition and Weapons", PATR **2145** (1955), p Rus 4 6)B.T.Fedoroff et al, "Dictionary of Explosives, Ammunition and Weapons" (German Section), PATR **2510** (1958), p Ger 33 7)Glossary of Ord (1959), 82 & 90

Découpage des Poudres. French operation of cutting the proplt strings, following the operation known as "étirage ou filage des poudres"

Refs: 1)E.Chevereau, MP **20**, 151-80 (1923) 2)Vennin, Burlot & Lécorché (1932), 592-94

DeCustro Explosive. Accdg to Ref 1, it is a chlorate expl contg Sb trisulfide & bran or a similar material. It was patented in France in 1883. Accdg to Ref 2, its compn was as follows: K chlorate 50.0, Bran 43.7 & Sb trisulfide 6.3%. It was prepd in the form of a paste by mixing the ingredients in the presence of water. Substitution of bran with wood cellulose did not give the desired plasticity

Refs: 1)Daniel (1902), 183 2)Giua, Trattato **6** (I) (1959), 394-L

Deep Bonding Process (Tiefbonder-Verfahren, in Ger) **for AP Projectiles.** This method, developed in Germany during WWII by the late Dr. V. Duffek & collaborators, consists of deep surface treatment of a sintered iron AP projectile with a phosphate. The method is claimed to diminish the wear of gun barrels and to increase the effectiveness of armor penetration by AP projs

Prior to WWII, the Germans in some of their rapid-firing guns, used projs contg either a lead core or a lead head with a sheath made of cast iron plated with "tombac" metal (an alloy of Cu & Zn). Beginning ca Jan 1941, when a shortage of lead developed, the Germans tried to use sintered iron projs. This did not give satisfactory results because the wear of the bore was so great that only 400 rounds could be fired before the gun became unusable. In order to decrease the friction, an attempt was made to zincate the surfaces of sintered iron projs, but this method did not decrease friction sufficiently to effect a noticeable decrease in the wear of the bore

Knowing that the friction of some crystalline inorganic compds decreases when they are subjected to high temps or pressures and, to a certain extent, to high impact stresses, Dr Duffek proposed to cover sintered iron projs with such substances. The surface covering was achieved by a phosphatizing process, similar to that used in industry for reducing corrosion. The process known in industry as "parkerizing" consists essentially of a treatment of an iron object with an acidic phosphate soln which results in depositing a thin layer of cryst iron phosphate on the surface of iron. When applied to sintered iron projs, the "parkerizing" process decreased only slightly the friction of projs in the bore, because the amt of phosphate deposited on the surface was so small that it was completely removed even before the proj left the bore. The use of this method did not improve the penetration of armor by AP projs

The investigation of Dr Duffek & collabs continued and, on the strength of their suggestions, a process was developed at the Metallgesellschaft AG, Frankfurt a/Main, (Refs 1, 2 & 3), which permitted the deposition of thicker surface layers of phosphate crysts due to deeper penetration of the phosphate solns into sintered iron objects. The process (called in Ger "Tiefbonder-Verfahren") may be conducted by one of three methods, but the following procedure was preferred by Dr Duffek:

a) Treat the sintered iron article with vapors of trichloroethylene in order to remove any oil or fat from the pores

b) Transfer the article to a bath contg 8g Na hydroxide & 2g Na nitrite per liter of water maintained at 95°

c) After allowing the article to stay in the bath for exactly 1 min, remove it and, without rinsing, place in another bath which consists of solns of Zn phosphate & Zn nitrate (contg 5g Zn, 7g P_2O_5 & 6.9g NO_3 per liter of w) maintained at 95°

d) After keeping in the bath for 5 mins, remove the article and rinse it thoroughly with cold w

e) Place the article for 1 min in a bath contg 5g of a mixt consisting of 30% Na silicate, 45% Na nitrite & 25% Na hydroxide per 1 liter of w maintained at 95°

f) Remove and place for 40-60 secs in a bath contg a soln of 0.5g Na chromate per liter of w maintained at 95°

g) Remove the article and dry it

It was claimed by Dr Duffek that when sintered iron bullets treated by the above

method were fired from a pistol, there was no noticeable wear of the bore even after 4600 rounds. This was even better than with the pre-WWII bullets provided with lead cores

On the strength of the success of the above process, Dr Duffek was allowed by the German War Ministry to develop a new type of AP projectile. This was prepd as follows: A sintered iron sheath, consisting of fine grains of iron in the inside layers and coarse grains on the outside of sheath, was welded to the surface of an ordinary solid steel proj. The welding was done by the High Frequency Method (Hochfrequenz-Verfahren, in Ger) developed by the Siemens -Halske AG. Then the surface of the sheathed proj was treated by the Deep Bonding Process

Tests by firing 20mm & 37mm AP projs, prepd by the above method, against a 5cm thick chromium -nickel steel armor plate placed at a distance of 200 meters, showed that the av penetration was about 2/3 deeper than with an untreated standard AP proj

Refs: 1)V.Duffek & collaborators & Metallgesellschaft AG, Frankfurt a/M, GerP M153085 VI/48d, Jan 26, 1942 2)V.Duffek, "Report to the High Command of the German Forces" (Document of the Chemisch-Technische Reichsanstalt, Berlin) and private communication 3)B.T.Fedoroff et al, "Dictionary of Explosives, Ammunition and Weapons", (German Section), PATR **2510** (1958), p Ger 198

Deep Hole Drilling with Explosives. It has been described by:

(?). Ostrovskii, Canad Mining Journal **83**, No 7, 51 (1962)

The paper is abstracted in Explosivst **1964**, p 171, under the title "Tiefbohren mit Sprengmitteln"

Defeat of Armor. It is damage to the armor protection of ships or armored vehicles which will result in endangering the lives of personnel and damage to material behind the armor. The types of ammo which serve for defeating armor are: AP (armor-piercing), HEAT (hyper-velocity antitank), HEP (high explosive plastic), etc. Ammunition serving to defeat concrete is known as A/C (anticoncrete)

Refs: 1)Armament Engrg (1954), 199 & 219 2)Glossary of Ord (1959), 21 & 90 3)Ord Tech Term (1962), 23 & 93

Defense Against Chemical Warfare. See the paper of Dr. E. Plötze, Explosivst **1963**, 115-20 & **1964**, 157-63 and references under Chemical Agents or Chemical Warfare Agents, Vol 2, pp C170-C171 and under Chemical, Biological and Radiological Warfare, p C172

Defense, Civil. The subject has been described in the following refs:

1)US Joint Crossroad Committee, "Radiological Defense", US Govt Ptg Off, Washington, DC, Vol 1 (1948) 2)US Civil Defense, "Health Services and Special Weapons Defense", USGPO, Wash, DC (1950) 3)J.R.Beishline, "Military Management for National Defense", Prentice-Hall, NY (1950) 4)US Natl Security Resources Board, "United States Civil Defense", USGPO, Wash, DC (1950) 5)P.Y.Hammond, "Organizing for Defense", Princeton Univ Press, Princeton, NJ (1961) 6)G.G.R.Williams, "Industrial Defense Planning Manual", The Security of Industries Civil Defense Offices, London (1961)

Defense System, Antiaircraft. A grouping of electronic and/or mechanical equipment for detecting the presence of aircraft within an area defended by AA (antiaircraft) guns

Ref: Glossary of Ord (1959), 90

Defensive Grenade. Same as Grenade, Fragmentation

Deflagrating Explosives and Deflagration. Deflagrating expls are substances which on being heated, such as betw temps of 500 to 1700°C (or subjected to flame, sparks, shock, friction, etc), while unconfined and in small quantities, ignite suddenly and burn much quicker than in ordinary *combustions* (See Vol 3, p C429-R). The burning of deflagrating expls usually proceeds rather violently and is accompanied by a flame (or sparks) and a hissing (or crackling) sound, but not with a sharp loud report as in the case of *detonating explosives* (qv)

Accdg to definition given by Rinkenbach in Ref 9a, "*deflagration* is a mode of explosion distinguished from detonation and constituting the very rapid autocombustion of particles of

explosive as a surface phenomenon. In the case of deflagration, the reaction products flow away from the unreacted material, while in detonation these are at extremely high pressure and flow toward the unreacted material" (See also Ref 8a)

On initiation of deflagration, local finite "hot spots" are developed either thru friction between the grains, or by adiabatic compression of gases in interstices betw the grains and in bubbles within liquids. In either case, heterogeneous decompn of material at the grain or bubble surface produces heat and volatile intermediates. These products undergo highly exotermic reactions in the gaseous phase; and the additional heat is more than sufficient to cause decompn and volatilization from the newly exposed surface. This means that deflagration is self-propagating (Ref 9a)

If an explosive is finely divided and is in the form of a large mass, then deflagration can occur nearly simultaneously throughout the mass. In such case, the confinement of the particles within the mas, because of the viscosity of gaseous products, has the effect of increasing pressure, and this, in turn, causes increase in rate of deflagration and of temperature. For example, the rates of deflagration of smokeless proplnts, which are betw 0.0001 & 0.0004 inches/second at atm pressure, might increase to betw 3 & 11 inches/sec when the pressure is increased to 25000 psi. The final result of increase in pressure of unconfined expls might be a violent deflagration, but no detonation, except when very large quantities are present as in powder magazines

If, however, the expl is under confinement, the deflagration might undergo transition into explosion or detonation. Confinement permits rapid increase in pressure with consequent rapid increase in the rate of deflagration. If the rate reaches the value of 1000-1800 m/sec, it is already not deflagration but "low-order" detonation. If the rate increases to 5000 m/sec, the detonation is "high-order"

It appears that transition of deflagration into detonation is almost discontinuous and may be caused by mechanical shock waves formed within the mass of explosive by the hot product gases that stream from the surface undergoing very rapid deflagration (Ref 9a)

Sensitivity to deflagration is usually detd by measuring the time requiring ignition when an expl is exposed to a flame having a known temperature

Rate of deflagration is measured by burning a column of known length, or in a bomb equipped with a pressure gage that registers on a rotating drum or an oscillograph (Ref 9a) (Compare with Burning and Burning Characteristics of Explosives, etc in Vol 2, pp B343 to B362. See also Combustion in Vol 3, pp 429-R)

There is no sharp distinction between deflagrating and detonating expls, but usually the so-called "low explosives" (LE's) (such as BkPdr, NC, Smokeless Propellants, AN expls not contg NG or high nitrogen aromatic nitro-compds) behave as deflagrating expls, while "high explosives" (HE's) (such as DNB, TNB, TNT, PA, PETN, RDX, Tetryl, etc) act as detonating expls. In many cases the same expl may behave as a deflagrating expl, when unconfined, and as a detonating expl when confined and properly initiated. For example when a small quantity of BkPdr or NC is thrown into a red-hot crucible, a very rapid combustion with evolution of flame takes place. This is a deflagration. When the same substances are confined in a bomb, grenade, etc and are initiated with a strong cap, a rapid bursting of the container, accompanied by a very loud report, flame & some smoke and evolution of an enormous quantity of gas, takes place. This is an explosion or detonation. Before the discovery of NC and smokeless proplnts, BkPdr was used as a low expl for propelling projectiles and as a high expl for bursting grenades, bombs and shells. After the discovery of NC, it replaced BkPdr not only in proplnts, but also as a bursting charge, expecially in warheads of marine torpedoes, and in AP shells. The variety of NC used as a bursting chge was of *Guncotton* type. It was press-loaded, while still wet with 18-20% water, into projs. Such projs were used as late as WWI, especially in Russia

Most high expls behave as detonating expls even if they are used in small quantities and unconfined. For example, if NG or TNT is thrown on a hot plate, it detonates with a loud report. There are, however, exceptions, as for example, with RDX which deflagrates when ignited by a red-hot object

Some expls might start to deflagrate and then suddenly the rate of decompn increases

and detonation takes place. The transition of deflagration to detonation has been discussed in Refs 5, 7, 9, 12, 14 & 15

Refs: 1)Pérez Ara (1945), 181-82 2)Kingzett's Chemical Encyclopedia, Van Nostrand, NY (1946), 281 3)Ohart (1946), 17 4)N.Manson et al, CR **230**, 2156-58 (1950) & CA **44**, 11097 (1950) (Calcn of the speed of deflagration in colloidal proplnts) 5)A.R.Ubbelohde, "Transition from Deflagration to Detonation", pp 566 -72 in the "Third Symposium on Combustion, etc", Williams & Wilkins, Baltimore (1952) & CA **46**, 11626e (1952) 6)Blasters' Hdb (1952), 45 7)S.Travers, MAF **27**, 699-856 (1953) & CA **48**, 12409 (1954) (Discussion on detonation, deflagration, etc of condensed expls) 8)R.Sartorius, Intern Conf of Directors of Safety in Mines Research, 8th Congr, Dortmund-Derne, Germany, Paper No 17, 20pp (1954) & CA **49**, 16435 (1955) (An experimental method to study the deflagration of mining expls under conditions comparable to the normal loading of a mine bore hole) 8a)Anon, "Military Explosives", **TM 9-1910** (1955), 37-8 (Deflagration) 9)M.Taillandier & G.Touzeau, "Deflagration and Detonation", Paper No 36, presented at the 9th Intern Conf of Directors of Safety in Mines Research, Bruxelles-Heerlen (1956) (Engl translation OTIA 3058) 9a)G.L.Clark, Ed, "The Encyclopedia of Chemistry", Reinhold, NY (1957), 284-85 (Deflagration) 10)S.Boutry & C.Fossé, CR Congr Intern Chim Ind 31^{e}, Liège (1958) & CA **54**, 23329 (1960) (Deflagration of mining expls) 10a)Blasters' Hdb (1958), 49 11)J.F.Roth, Explosivst **1958**, 48-52 (Deflagration and detonation) 12)M.A.Cook et al, PrRoySoc **246A**, 281-83 (1958) (Deflagration-to-detonation transition in solid and liquid expls) 13)C. M.Mason et al, US Bur Mines Quarterly Rept Jan 1 to March 31, 1959 (Deflagration and detonation of propellants) 13a) Glossary of Ord (1959) 90 14)M.A.Cook et al, JApplPhys **30**, 1579-84 (1959) & CA **54**, 1851 (1960) (Deflagration-to-detonation transition) 15)F.C. Gibson et al, US Bur Mines Summary Rept No **3863**, Jan 1, 1959 to Dec 31, 1961 (Study of deflagration-to-detonation in proplnts & expls) 16)D.Ahrens, Nobelhefte **1963**, 118 (It is abstracted in Explosivst **1964**, 172 (Deflagration of mining expls) 17)Blasters' Hdb (1966), 25

Deflagration Temperature Test. The test, expecially suitable for smokeless proplnts, consists of placing a small shaving of the substance to test into a short piece of brass tubing of small diam, and closing both ends with glass discs. After inserting a thermocouple, the ensemble is placed in the electrically heated stage of a polarized microscope. Then the temp of the stage is raised and the galvanometer reading is taken at which the sample deflagrates. The deflagration temp of a single -base proplnt with NC contg 13.9% N was found to be 154°

This method was also used for detn of temp of deton of HE's and it gave 206° for NG and 213° for PETN

Ref: A.Michel-Lévy & H.Muraour CR **193**, 40-2 (1931) & CA **25**, 5291 (1931)

Deflagration Test (Essai de défragration, in Fr). See under Ignition (or Explosion) Test in Vol 1, pp XVI & XVII

Deflection *(in Gunnery).* Horizontal clockwise angle between the axis of the bore and the line of sighting. It is also the angle of a deflection shot measured betw the line of sight to the target and the line of sight to the aiming point

Ref: Glossary of Ord (1959), 90

Deflectometers vs Strain Gages. A comprehensive study on strain gages versus reflectometers is given by A.K.Behlert, PATR No 8, Aug 1958

Deformation and Fracturing of Thick-Walled Steel Cylinders under Explosive Attack. In order to obtain basic info on the manner and type of fracturing and plastic flow caused by extremely high pressures acting for short duration, a series of tests has been conducted at the US Naval Ordnance Test Station, Inyokern, California. For these tests, annealed, heavy -walled cylinders of low carbon (1020) steel were filled with expls and detonated

Ref: J.Pearson & J.S.Rinehart, JApplPhys **23**, 434 (1952)

Defraiteur's Explosives. Patented in France in 1898 & 1899, these expls were prepd as follows: a) AN 80-89, Nitrodextrin 15-10 & rosin 5-1% and b) AN 80-93 & Nitrodextrin

20-7%. The 2nd variety was recommended as a safety expl

These expls were similar to *Casteau* expl patented in Belgium in 1899 (See Vol 2, p C83-R

Ref: Daniel (1902), 183

Defuze. To remove a fuze from an item of ammo

DEGDN or DEGN. Abbr for Diethyleneglycol Dinitrate described under Diethyleneglycol (DEG)

Degradation and Deterioration of Cellulose. The degradation products of cellulose are either glucose or polysaccharides based on anhydro-glucose units. The hydrolysis of cellulose by mineral acids is an almost continuous process of change. The series passes from hydrocelluloses of Cu number below 5, which retain the fibrous form, to those of increasing Cu number (5 or above), which are powders; thence to the cellulose-dextrins & oligo-saccharides, composed of $(C_6)_{25}$ units downwards, and finally to cellobiose & glucose. Modification of hydrolysis in which acetylation takes place simultaneoualy leads to acetylated derivs based on glucose. Hydrolysis of methylated cellulose gives simple methylated sugars. The thermal decompn of cellulose gives B-glucosan (Refs 1 & 2)

See also under Cellulose, Vol 2, p C96; Cellulose Derivatives, Vol 2, p 98-L; and Degradation of Nitrocellulose Solutions in this volume

Refs: 1)Dorée (1947), 195ff 2)T.Urbański, "Chemistry and Technology of Explosives", Pergamon Press, Long Island, NY, Vol **2** (1965), 226-28

Degradation and Deterioration of Explosive Jellies. Studies by Ficai of some jellied expls and proplnts showed that alteration and separation of components of jellies contg AN is particularly noticeable if Na nitrate is also one of the components. This is attributed to the presence of impurities in Na nitrate, such as Na hydroxide and Na nitrite. Na hydroxide reacts with AN giving ammonia

Ref: C.Ficai, Chimica (Milano), **5**, 167-69 (1950) & CA **45**, 2209 (1951)

Degradation and Deterioration of Explosives in Storage. See: Ageing of Dynamites in Vol 1, p A110-R; Ageing of Mine Explosives Containing Ammonium Nitrate and Chlorides in Vol 1, p A112-L; and McAdam & Westwater (1958), 22-3

Degradation and Deterioration of Propellants in Storage. See Ageing of Propellants, in Vol 1, p A112-L and also the following refs:

Refs: 1)C.A.Parker, JSCI **67**, 434 (1948) (The aging mechanism of Cordite stabilized with sym-diethyldiphenylurea) 2)Admiralty Material Laboratory (Brit), Report No A/12 (M) (1948) (Transformation products of Carbamite obtd on ageing of Service Propellants) 3)W.A.Schroeder et al, IEC **42**, 539-46 (1950) (Chromatographic investigation of smokeless propellants. Derivatives of Centralite formed in Double Base Propellants during accelerated aging) 4)Astrodyne, Inc, "Solid Propellant Aging Studies", Report AR(752) -1-58, Oct 1958; Project 3059; Contract AF 33(616)-5813 (Conf) 5)N.Fishman, Ordn **43**, 681-83 (Jan-Feb 1959) (Solid proplnt aging) 6)P.E.Gagnon et al, "Solubility of Aged Propellants", CARDE Technical Memorandum **229/59**, Valcartier, Quebec (1959) 7)Stanford Research Institute, "Aging Properties of Solid Propellants for Rocket Motors", Progress Reports 69 to 74 incl, April to Sept 1959; Contract AF 33 (038)-28634; Project SD 2708 (Conf) 8)Standford Research Institute "Solid Propellant Aging Studies", Reports 14 to 17, May to Oct 1959; Contract AF 33(616)-5806; Project SU-2547 (Conf) 9)North American Aviation, Inc, "Solid Propellants Aging Studies", Final Report of Activities R-4007, Nov 1959; Contract AF 33(616) (Conf)

Degradation of Nitrocellulose Solutions. It has been observed that the viscosity of NC solns (such as in acetone, ether-alcohol, etc) diminishes on standing. This may be partly due to changes in the degree of the of the specific interaction betw the dispersed particles, and is frequently a sign of lack of equilibrium in the soln, but

principally it is due to degradation of the NC

It has also been observed that:
a) High viscosity NC's degrade more rapidly than low viscosity NC's
b) Degradation seems to cease after standing of NC for a certain time at room temp, but at higher temp the reduction in viscosity might continue indefinitely
c) The rate of degradation of NC in a given solvent seems to be independent of concn
d) Oxygen is not a factor in NC degradation
e) Organic peroxides hasten degradation, and the usual oxidation & polymerization inhibitors of the neutral type (such as hydroquinone) slow down the degradation of NC
f) Inorganic acids and bases accelerate degradation, but organic bases lead to an even more rapid degration of NC
g) Some solvents may be set up in series of gradually increasing the tendency to degrade NC as follows: butyl acetate 40, plus toluene 60%; straight butyl acetate, ethyl acetoacetate, butyl lactate, cellosolve acetate, cellosolve and cyclohexane

It seems to be quite probable that the drope in viscosity of NC solns is due to a shortening of the average chain length of NC. The chance of a chain break, is, of course, greater as the chain becomes longer, and the effect of a given reduction in molecular weight on viscosity is much greater if the original chain is very long. A kinetic study of the process showed that the rate of breaking of bonds was the same in all viscosity ranges of NC

It seems also likely that the chain reaction of NC is catalyzed by bases and that the variation in activity of solvents in causing degradation is due to a variation in their basisity. The base- and solvent-catalyzed degradations of NC in solns appear to be internal oxidation-reduction reactions involving splitting of the chain

If NC solns contain suspended matter, changes in viscosity due to settling or flocculation may be anticipated. One source of such suspended matter is the irreversible change that often occurs on the surface of precipitated or dried products. For example, if a soln of NC in acetone is pptd by pouring into water, and if the ppt is dried and then redissolved, a white haze is observed. This is not encountered if a hydrocarbon is used for pptn. Another occasional source of change in viscosity is encountered if an attempt is made to work under conditions where the solvent power is low enough to allow the existance of two phases. In some cases, the two phases remain intermixed as in an emulsion. The viscosity of such a system depends greatly on the time of standing and on the temp. A rise in temp sometimes results in an increase in viscosity due to soln of more of the derivative in the continuous phase

Refs: 1)W.Wehr, KolloidZts **88**, 185 & 290 (1939) 2)Ott **5** Part 3 (1955), 703, 1046, 1048 & 1095 3)T.Urbański, "Chemistry and Technology of Explosives", Pergamon Press, Long Island, NY, Vol **2** (1965), 258-78

Degressive Burning Propellants. To the brief description given in Vol 2, p B360-L, under Burning, Regressive, and in Vol 2, p C31-L, under Cannon Propellant, the following may be added:

The burning characteristics of proplnts are classified as "degressive", "neutral" and "progressive". The degressive type for use in firearms is usually in the form of grains, thick flakes, thick strips or nonperforated cylinders (such as cords of Cordite). The initial total surface is large but it gradually decreases as the burning progresses, thus causing a continual decrease in the rate of burning and therefore in the volume and pressure of gases evolved. Since the peak pressure in the weapons using such proplnts develops earlier than when using progressive burning proplnts, there is less muzzle flash produced. However, inasmuch as the peak pressure is usually very high, especially when it is desired to develop high muzzle velocities (as in modern guns), such proplnts are usually suitable only for small arms or for small caliber cannons

A brief description of "neutral-" and "progressive-" burning proplnts is given in Vol 2, p C31, under Cannon Propellant

Refs: 1)Hayes (1938), 12 2)Davis (1943), 317 3)Ohart (1946), 22-23 4)Glossary of Ordn (1959), 91

Degtiarëv, V. A. (1880-1950?). One of the best known Russian designers of firearms. For example, he designed the 7.62mm 1938 Light Machine Gun, the 12.7mm Gun (equiv to US .50 caliber gun), and the M-1943 7.62mm Heavy Machine Gun (Ref 1, 2 & 4)
Refs: 1)M.M.Johnson & C.T.Haven, "Automatic Arms", Morrow, NY (1941), pp 62, 70, 72 & 229 2)W.H.B.Smith, "Small Arms of the World", MilServPubCo, Harrisburg, Pa, 4th ed (1948), pp 261, 265 & 480 3)G.M.Chinn, "The Machine Gun", US Navy Bureau of Ord, Washington, DC, Vol **2** (1952) (Conf, not used as a source of info) 4)B.T.Fedoroff, et al, PATR **2145** (1953)

Dehydrating and Drying Agents. Dehydration is a process of removing water from compds and crysts, usually by heating. Drying is a process of removing moisture or water by heat, vacuum or chemical agents. More generally drying refers to the removal of a liq from a solid by evaporation, the necessary heat being provided by conduction from a solid surface, convection from a hot gas, or by radiation. Drying agents then are heat & chemicals, the latter of which are also called *desiccants*

Some of the common desiccants are as follows:
Aluminum oxide, Al_2O_3, used for absorbing gases & water vapors
"Ascarite", a proprietary absorbent made from NaOH & asbestos and used for the absorption of carbon dioxide
Barium perchlorate, $Ba(ClO_4)_2$, used as a regenerable drying agent & absorbent for ammonia
Calcium bromide, $CaBr_2$, used as a dehydrating agent
Calcium chloride, $CaCl_2$; $CaCl_2.H_2O$; $CaCl_2.6H_2O$, used as cheap drying agent
Calcium oxide, CaO, used in water treatment
Copper sulfate, $CuSO_4$, used as a dehydrating agent
Magnesium oxide, MgO, used for adsorption of various gases & liqs
Magnesium perchlorate, $Mg(ClO_4)_2$, used as a regenerable drying agent for gases
Phosphorous pentoxide, P_2O_5, used as a dehydrating agent
Potassium hydroxide, KOH, has a wide variety of uses
Sodium hydroxide, NaOH, is the most important coml caustic
Sulfuric acid, H_2SO_4, is generally considered to be the most important industrial dehydrating chemical
Zinc bromide, $ZnBr_2$, used as a desiccant
Zinc chloride, $ZnCl_2$, used as a dehydrating & condensing agent in org synthesis
Commercial dehydration operations are best performed on solids by an apparatus that provides air to carry off water vapor on its release. Some of the equipment is convertible from heat removal to heat supply by simply changing the temp level of the fluid or air (Ref 3)
Refs: 1)Hackh's (1944) (found under individual compound) 2)Cond Chem Dict (1961) (found under individual compound) 3)Perry (1963), Sect 11-45

Dehydrating Value of Sulfuric (DVS) in Mixed Acids. In the process of nitration of aliphatic alcohols (such as glycerin, glycols, etc), it is necessary to know the exact composition of the nitrating acid. In the analysis of such acid, known as *mixed acid* (MA), the vollowing values must be detd: TA (total acidity), TS (total sulfuric), TN (total nitric), AS (actual sulfuric), AN (actual nitric) and N-so (nitroso). A brief description of procedures for detg these values is given under Acidity in Acids in Vol 1, pp A88-A90

In setting up optimum conditions for nitration so as to obtain maximum productivity, it is important to fulfill also the following requirements:
a) The amt of 100% HNO_3 present in MA must not only be sufficient to satisfy the stoichiometric requirements of the nitration reaction, such as in the nitration of benzene to Dinitrobenzene:

$$C_6H_6 + 2HNO_3 = C_6H_4(NO_2)_2 + 2H_2O,$$

but also must be in some excess in order to maintain a reasonably fast over-all reaction rate. The ratio of the wt of 100% HNO_3 to the wt of the material being nitrated is usually designated as R
b) The amt of 100% H_2SO_4 (with, if necessary, SO_3 which comes from added oleum) must be sufficient to bind the water formed during the nitration, thus promoting the desired reaction. The accumulation of water as a result of the nitration process is objectionable because:

1) In case of dealing with aliphatic alcohols (such as glycerin), it exerts an unfavorable influence on the equilibrium of the reaction and 2) In case of aromatic nitrations, it represses the formation of nitryl or nitromium ion, NO_2^+

In order to judge if the MA has a sufficient amt of H_2SO_4 to bind the water formed during the nitration to the extent necessary to fulfill the optimum conditions of nitration, the so-called DVS (Dehydrating Value of Sulfuric) can be calcd from the following equation (Ref 1);

$$DVS = \frac{\text{Wt of 100\% } H_2SO_4 \text{ in MA}}{\text{Wt of } H_2O \text{ at the end of Nitration}} \qquad (I)$$

If the compn of MA is known, the DVS may be calcd from the equation given by Groggins (Ref 2, p 105):

$$DVS = \frac{S}{EN/R + W} \qquad (II)$$

where:

S = % Actual H_2SO_4

E = Water equivalent of material to be nitrated (parts of w resulting from the nitration of 1 part of material, plus w present in material, if any

N = % Actual HNO_3

R = Ratio of HNO_3 to starting material

W = % Water in MA

If the calcn is based upon 100 lbs of MA, then the figures for percentages are in pounds. If the wt of starting material per 100 lbs of MA is designated x, then: R = N/x & x = N/R. Hence, the EN/R = Ex represents the pounds of w resulting from the wt of starting material requiring 100 lbs of MA. This means the denominator EN/R + W represents the total w present at the end of nitration

For the MA listed in Ref 2, p 105: Total H_2SO_4 49.99, Total HNO_3 52.44, Actual H_2SO_4 49.90, Actual HNO_3 52.38, Nitroso ($HNOSO_4$) 0.12 & H_2O minus 2.40%, the DVS may be calcd as follows:

Equation of nitration is:

$$C_3H_5(OH)_3 + 3HNO_3 = C_3H_5(ONO_2)_3 + 3H_2O$$
$$92.06 \qquad 189.06 \qquad 227.06 \qquad 54.06$$

If moisture content of glycerin is 1%, then the water equivalent for glycerin is calcd as follows:

$(54.06/92.06) \times 0.99 = 0.581$ lb per lb from nitration
plus 0.010 lb from moisture content
E = 0.591 lb per lb

If stoichiometric ratio nitric to glycerin is equal to 189.06/92.06 = 2.05, then the value R = 2.30, usually used in nitration of glycerin, provides an excess of ca 13%

On replacing S, E, N, R, & W of formula (II) with numerical values for the above acid, the following formula is obtd:

$$DVS = \frac{49.90}{(0.591 \times 52.38)/2.30 - 2.40} = \frac{49.90}{11.06} = 4.51 \qquad (III)$$

For optimum results, the DVS must be betw 4.35 & 5.00

It is important to understand that DVS is not the property of the acid alone, but applies also to the situation which exists in the nitrator at the end of nitration when the acid is used under certain specified conditions (Ref 2, p 107)

In the manuf of some expls, such as TNT, it is not required to calc DVS for MA, while for NG and Nitroglycol it is of great importance to know this value. For some other expls, the calcn of DVS value is optional. For example in the prepn of Dinitrobenzene by nitrating benzene with MA contg H_2SO_4 80, HNO_3 18 & H_2O 2% and R = 1.1, the DVS is 7.0, while nitration of benzene with MA contg H_2SO_4 60, HNO_3 32 & H_2O 8 and R = 0.81 gives only Mononitrobenzene and DVS 3.51

Refs: 1)Clift & Fedoroff, Vol 1, Chap XI, pp 6-7 (1942) 2)Groggins (1958), 104-07

Dehydration or Drying of Nitrocelluloses. In the manuf of NC, the product after nitration, preliminary boiling, poaching, screening, blending and wringing (See Vol 2, pp C107 & C108), is in the form of a white powdery material contg ca 30% moisture. It was discovered in 1869 by Brit chemist E. Brown that NC contg ca 13%N (such as Guncotton), could be detonated by a strong MF cap, if the NC was in compressed form. Better results were obtd with Guncotton contg less water (18-20%). This gave the idea to use compressed wet Guncotton for loading shells, warheads of torpedoes and demolition charges. This practice continued as late as WWI, especially in Russia. It was discontinued in favor of PA and TNT which are more brisant and powerful than NC. Brown also was the first

to point out (in 1868), that dry NC could be detonated even with a weak MF cap. This gave the idea to load shells with compressed dry NC, but this experiment was a failure because most of such shells exploded inside the gun barrels on firing. This, however, did not prevent the use of Guncotton in boosters

In early 1880's, it was discovered, nearly simultaneously by Duttenhofer (in Germany), Vieille (in France) and Volkmann (in Austria) that dry NC can be made to deflagrate slowly without deton and to be fairly insensitive to impact if it is made into a compact form by gelatinization with volatile liquids, such as acetone, ether-alcohol, ethyl acetate, etc, and then drying to remove the bulk of the liquid. Such NC was found to be suitable for the manuf of smokeless proplnts. It was also found that if NC contained more than 1% moisture, it did not gelatinize as easily as did the dry material and that the presence of moisture was undesirable in smokeless proplnts because it diminished their ballistic potentials

Laboratory Methods of Dehydration of NC. The following methods may be used for water-wet NC:

a) Spread 50-100g of completely stabilized sample in a layer not thicker than 1 inch on a tray made from several sheets of filter paper and leave for several hours (preferrably over night) in a dry place at a temp below 40°. No expls or combustibles, (especially volatiles like ether or alcohol), should be present in immediate vicinity. Turn over NC occasionally with a wooden or hard rubber spatula and, when required for analysis, remove a small portion (10-15g) to a flat, porcelain evaporating dish for final drying. Place the dish either in an expln-proof electric oven and leave for 1-1½ hrs at 100-105°, with the door slightly open, or in a vacuum oven maintained at ca 55° with a vacuum of 18 to 25 inches

b) A more rapid method consists of spreading a small sample (10-15g) in a layer ca 1/4 inch thick on a tray made of several sheets of filter paper and placing it for ca 2 hours either on a closed steam bath or on a hot radiator. A still more rapid method is to place the tray for 1-1½ hours either in an oven at 100-105° or in a vacuum oven at 50° with a vacuum of 18 to 25 inches. The rapid method is not as safe as the slow method, as occasional fires have been reported where this procedure was in use. The rapid method should never be used for incompletely stabilized NC, because it might cause decompn. Heating in an oven at 100-105° is not as desirable as heating at 55° in a vacuum oven (Ref 9)

Plant Methods of Dehydration of NC. As was mentioned above, wringing which is accomplished in centrifugals, reduces the percentage of water in NC to ca 30%. A slightly better dehydration is achieved by compressing NC in hydraulic presses. The resulting blocks of NC, containing 20-25% moisture can be used as demolition charges (Ref 2, pp 2161-63)

As the percentage of water in NC used for the manuf of smokeless proplnts should not exceed ca 1%, the centrifuged or hydraulically compressed NC should be further dehydrated, which can be achieved by the following methods:

a) In the "stove" method, as formerly practiced in Gt Britain (Ref 1), the loose NC was spread on brass gauze trays with wooden rims and placed in chambers (stoves). The trays were grounded to prevent accumulation of static electricity. The air, at a temp not higher than 40°, was blown thru inlets at top of the stove and removed thru outlets, of slightly larger size, located near the floor. The reason for this arrangement is that when the hot air arriving thru the top picks up the moisture, it cools off, becomes heavier and sinks to the floor

More rapid drying could be achieved by raising the temp to a higher degree (such as 70°), but this method is more dangerous (Ref 2, p 2152)

b) Quinan's method consisted of spreading the NC in a thin layer on a canvas and blowing hot air thru it. This method was quicker than the stove method (Ref 3)

c) Drying at reduced pressures was quicker and could be conducted at lower temps. Several proposed methods are described in Ref 2, pp 2154-55

d) Liquid method of dehydration. The water remaining after wringing can be removed by treating the NC directly in the centrifugal with a liquid which is missible with water and a non-solvent for military grades of NC's. The most suitable liquid was found to be ethyl alcohol. Among the centrifugals con-

structed for that purpose may be mentioned those of Selwig & Lang (Ref 2, p 2126, Fig 126) and of Gentiou (Ref 2, p 2145, Fig 245)

e) Hydraulic displacement method consists of treating NC (previously dehydrated to 20-25% moisture content by compressing it in a hydraulic press) with ethyl alcohol and then squeezing the alcohol out in an open cylinder with top and bottom plungers hydraulically operated (Ref 2, pp 2142-43, Fig 243 & 244)

Treatment with alc is superior to drying with air (see methods a, b, c), not only because it is safer, but also because alcohol removes water and some undesirable impurities as well. These impurities consist mostly of cellulose nitrates of low nitrogen content, which are unstable and have a tendency to reduce the ballistic potential of proplnts manufd from NC contg them (Ref 2, pp 2134 & 2142-43). This method is based on USP 516924 (1894) of F. DuPont (cited in Ref 2, p 2134)

f) Instead of centrifugal or hydraulic dehydration, the pulped NC may be passed thru two rollers of the so-called *wet machine* from which it emerges with ca 30% moisture. It may be completely dehydrated either by drying or by displacement with alc (Ref 2, p 2140ff)

g) Solvent displacement process without pressure, proposed in 1903 by F.Nathan and J. & W. Thomson, consists in placing pulped wet NC on the cloth of a large Büchner type funnel (similar to present *Nutsch,* but not connected to a vacuum system), and covered with a layer of water to remove the air. After this, alcohol is slowly and carefully run down the sides of Büchner in such a manner as to form a layer above the water and with a sharp line of demarcation betw the two. The w is then drawn off slowly by partially opening the valve at the bottom of the Büchner and as the level of w falls, the alc follows and gradually replaces it in the interstices of NC. This treatment is continued until the density of effluent drops to that of alcohol (Ref 2, p 2141)

h) Drying of NC by heating it with a water-immissible hydrocarbon, which is a non-solvent for NC, (such as kerosene) in a specially constructed still (Ref 2, pp 2156-57)

i) In the method of R.Müller (cited in Ref 2, p 2141), a pulped NC contg not more than 10% moisture is treated in an apparatus which combines a rotary suction machine with a device for the counter current washing of the material with alcohol of progressively increasing strengths

j) The French methods for dehydration of NC's intended for the manuf of Poudre B are described in Refs 4, 5 & 8

k) The American method of dehydration of NC of Pyrocotton type is described in Refs 6 & 7. It is conducted in a press consisting of a cylinder closed at the top and a movable piston operated from below by hydraulic pressure. The press is charged with pulped Pyrocotton which was previously dehydrated in wringers to ca 30% water content and kept in warehouses heated in winter to prevent freezing. A preliminary pressure of ca 250psi is applied to the piston for a few moments in order to squeeze a portion of the excess water. This opn transforms the material into a porous cake (block). After releasing the pressure, a predetermined amount of alcohol (usually ca 1.25 lb for every lb of dry wt NC) is forced by a pump into the upper part of the press. This causes the alc to be absorbed by the porous cake and then the pressure is applied and gradually increased to ca 3500psi. The alc functions first as an agent for displacing the water and finally as a dehydrating agent. Its initial entrance into the block forces out a small portion of the remaining w; later, however, it combines with the residual w and produces a block of Pyrocellulose nearly free of w. The first portion of liquid forced out of the press contains relatively large amts of w and is transferred to the spent alcohol tank. The remaining liquid, however, is ca 90% alcohol. The spent alc is pumped into a storage tank and eventually rectified with the addn of caustic soda to decompose dissolved and suspended NC. It is then used again either in the dehydrating process or in the manuf of ether

The resulting Pyrocellulose block contains ca 33% of alc, by wt, and this amt is sufficient to make, when mixed with 2 parts of ether, a gelatinizer for Pyrocellulose (Ref 6, pp 21-2 & Ref 7, pp 20-21)

Further opns in the manuf of Amer Pyrocellulose include Block Breaking, Mixing, Preliminary Blocking, Macaroni Pressing, Final Blocking, Graining, Cutting, Solvent Recover, Drying, Blending and Packing (See Refs 6 & 7)

Dehydration of NC is not required prior to

its incorporation in Solventless Double-Base Propellants. Here water-wet NC is blended with NG and the resulting dough is dehydrated as described under Dehydration and Drying of Smokeless Propellants

Refs: 1)Marshall **1** (1917), 289 2)Worden Vol 1, Part 3 (1921) 3)Marshall **3** (1932), 85 4)Vennin, Burlot & Lécorché (1932), 582 5)Pepin Lehalleur (1935), 294 6)Hayes (1938), 21-22 7)Anon, "Military Explosives", War Dept Tech Manual **TM 9-2900** (1940), 20-21 8)L. Brissaud, MP **31**, 145-60 (1949) & CA **46**, 11683 (1952) 9)Laboratory Method, practiced at Picatinny Arsenal

Dehydration (Drying) of Smokeless Propellants. All proplnts manufd with the aid of volatile solvents (such as 2:1-ether/alcohol) must be dryed in order to remove residual solvent. This is necessary because the presence of solvent would make the ballistic property of a proplnt too weak and uncertain. As the solvent would evaporate during storage of an incompletely dried proplnt, it would become more and more powerful, but in a manner which is unpredictable *Drying of Single-Base Propellants.* Removal of solvent should not be too fast in order to prevent the deformation of grains

The following description is taken from Marshall (Ref 1): As the cords, strips, tubes, etc of a single-base proplnt emerge from the die-press, they are usually wound on frames provided with springs which allow the cords, etc to contract as the solvent evaporates. In case of cords, etc of large diam, they are nailed to the frames. The frames are then placed in a "stove" (similar to that briefly described under Dehydration and Drying of Nitrocelluloses) where the air, heated not above 40°, is circulated from top to bottom until the bulk of the solvent is removed. The spent air is passed thru an apparatus for solvent recovery and the incompletely dried proplnt cords, etc are removed from the stove. After cutting the cords, etc to the desired lengths, they are subjected to final drying. This could be done either by heating in vacuum ovens or by digesting with hot water. Drying by heating in ovens is rather dangerous because of possible ignition of proplnt and gases evolved

In the wet method, the grains of proplnt are digested with water, heated to ca 60°. The process lasts from a few days, for small grains, to weeks or even months, for grains used in large caliber cannons. After completion of wet drying the proplnt is dehydrated in stoves in the manner similar to that used for NC

The United States methods of solvent recovery and drying of single-base propellants are described in detail in Ref 2, pp 26-32. These operations are conducted with proplnts in the shape of final grains. Prior to this, the colloided mass of proplnt is subjected, in sequence, to action of a preliminary blocking press, a macaroni press, final blocking press, graining and cutting opns (Ref 2, pp 23-25)

The initial process of drying is known as *solvent recovery,* although the recovery is only incidental to the proplnt manufg process. There are three types of equipment that have been used successfully in recovery of solvent (ether-alcohol) and initial drying of proplnt: first, the chamber type in which the proplnt is placed in a metal -lined box provided with a water sealed lid; second, the car type, which utilizes a solvent recovery car as a drying chamber, the car being loaded directly from the proplnt cutters; and third, the tank type in which the proplnt is transferred from the cutter room to a large metal tank. In all of these types, the operation of removing the solvent from the proplnt is the same and consists of the following:

A) Warm air is circulated thru the proplnt and is then forced over cold coils. The change in temp causes pptn of the solvent upon the coils and produces a miniature fog. The saturated air after passing over these coils loses its surplus solvent. It is then reheated and forced thru the proplnt in a continuous cycle, completing in this manner the closed system of air circulation. As the solvent is removed, the grains partially collapse or shrink and it is therefore necessary to control the rate at which the solvent is removed in order to produce a uniformly finished proplnt. Furthermore, if the solvent is removed too rapidly the surface of the grains becomes hard, thus making difficult the removal of the solvent from the interior of the grains. Definite rules are laid down governing the length of time necessary for proplnts to remain in the solvent recovery bldg, the larger the grains, the more time is required

B) Temperature and times of heating in solvent

recovery process vary somewhat at different plants. A schedule which has been generally followed is outlined below:

a) Proplnts having web thickness up to 0.095 inch are treated by raising temp to 30° and holding for 24 hrs; raising to 38° and holding for 24 hrs and raising to 45° and holding for 5 days

b) Proplnts of web thickness above 0.095 inch must be kept for 48 hrs w/o heating; then the temp is raised to 25° and held for 24 hrs; raised to 30° and held for 24 hrs; raised to 35° and held for 24 hrs; raised to 40° and held for 24 hrs; and finally raised to 45° and held for 48 hrs

It is not practicable from an economic standpoint to reduce the solvent content of the proplnt to the desired degree in one opn, such as solvent recovery; hence, the final drying is accomplished as a separate opn

The final process of solvent removal, known as *drying,* can be conducted by two methods: "air-dry" and "water-dry". There is also a "continuous air-dry method", which is used in conjunction with "water-dry method"

I) Air-dry treatment (batch process). Here the proplnt, transferred directly from the solvent recovery house, is placed in narrow bins of the dry house where air, heated to 43°, is circulated during at least 30 days. The temp may then be raised to 55°. The time required to drive off the volatile matter to the desired content varies from 30 days for 75mm cannon proplnts to 90 or more days for 16-inch cannon proplnts. For proplnts having a web thickness of more than 0.095 inch, a period of at least 2 weeks in the dry house without heating must procede any heating

During drying opn, samples of proplnt are sent to the lab to det if the required external moisture and volatile solvent contents have been reached. The desired external moist content for air-dried proplnts varies inversely as the size of the grain from ca 0.1 to 1.0%, while the total volatile content of the dried proplnt may vary from ca 3.5 to 7.5%, depending upon the web thickness - the heavier web proplnts having the greater solvent contents

II) Water-dry treatment. Here water is kept circulating thruout the proplnt grains at temps, which vary by 10° increments, from 25° to 55° for ca 4 days for proplnts having a web thickness of ca 0.020 inch. Then the proplnt is removed and air-dried for ca 48 hrs either by batch process or by continuous method described below. The external moisture content of water-dried proplnts varies inversely with the web size of the proplnt from 0.9 to 1.4%

III) Air-dry treatment (continuous process). Here the proplnt arriving from the water-dry house is hoisted to a hopper and then slides by gravity into a shaking screen. This removes foreign material and drops the proplnt onto a bucket conveyor. The proplnt is then hoisted to the top of the bldg where it is thrown into a drying chute. The chute is ca 6 inches thick, 5 ft wide and 10 ft long, being equipped with baffles running crosswise in the direction of its smallest dimensions. A series of screens on the side wall provides the means for the passage of warm air thru the proplnt, and a temp of 50° is maintained by this means at the bottom of the chute. A shaking device, near the bottom, is regulated in such a manner that the rate of discharge and consequently the volatile content of the proplnt, is controlled by the speed at which this shaker is operated and by the amt of clearance betw the shaker and the bottom of the shute. The time required for drying is betw 4 and 5 hrs. One of the installations operating in the US before WWII was capable of drying to correct moisture and volatile contents 100000 lbs of 75-mm proplnt in 24 hrs

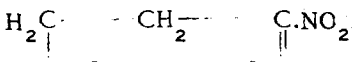
Drying of Double-Base Solvent Propellants. If a volatile solvent, such as acetone, has been used to facilitate the gelatinization of NC with NG, the solvent must be removed for the same reason as described under Drying of Single-Base Propellants. Here the air-drying method is preferred to water-drying because the loss of NG, on evaporation in heated air, is not as great as the loss due to solubility of NG in water (Ref 1)

Drying of Solventless Double-Base Propellants. Since the manuf of solventless double-base proplnts is potentially hazardous, particularly rolling, drying, extruding & machine operations, all such opns are performed by remote control. Following the final mixing, wringing & screen opns, the water-wet paste is preheated to 90°F and fed by remote control into differential rollers in sufficient quantity to make two sheets. Hot water or steam is used to control

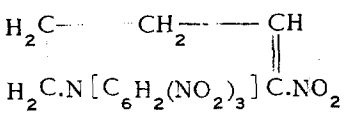
& maintain a relatively constant rolling temp. The sheets of proplnt are air dried in separate drying rooms which are so constructed that condensation & entrapment of NG is prevented. Air supplied to the drying rooms is not recirculated (Ref 6)
Refs: 1)Marshall **3** (1932), 89 2)Anon, "Military Explosives", War Dept Tech Manual TM **9-2900** (1940), 26-32 3)US Ord Corps Manual, "Safety Requirements for Manufacturing Small -Arms Ammunition", ORDM **7-228** (1955) 4)Ibid, "Safety Requirements for the Manufacture of Single-Base Solid Propellants", ORDM **7-229** (1959) 5)Ibid, "Safety Requirements for the Manufacture and Loading of Castable Solid Propellants", ORDM **7-230** (1960) 6)Ibid, "Safety Requirements for Manufacturing Double-Base Solventless Propellants", ORDM **7-227** (1960) 7)Urbański, "Chemistry and Technology of Explosives", Vol **2** (1965), 406

Dehydrogenation. The process whereby hydrogen is removed from compds by chemical means (Ref 2). Reducing or oxidizing agents in the presence of catalysts may be used (Ref 1)
Refs: 1)Hackhs' (1944), 256 2)Cond Chem-Dict (1961), 329

Dehydropicrylpiperidine, Nitro Deriv (called Nitrodehydropikrylpiperidin or 1-Pikryl-5 oder 6-nitro-1.2.3.4-tetrahydro-pyridin in Ger),



or



mw 339.22, N 20.65%; red ndls (from alc), mp 195°; prepd by treating an acetic acid soln of N-picryl-piperidin with ice-cold concd HNO_3; on warming with KOH, Picric acid is split off (Refs 1 & 2)
Refs: 1)Beil **20**, 23 2)A.P.N.Franchimont & H.J.Taverne, Rec **15**, 74 (1896)

Deissler patented in 1897 in Belgium primary compns contg as a base finely divided, easily oxidizable metals (such as Mg) mixed with a sulfide (such as Sb_2S_3) and an oxidizing agent (such as $KClO_3$)
Ref: Daniel (1902), 183

Deissler & Kuhnt patented in 1888 in England a safety mining expl consisting of Amm carbonate or chloride mixed with NG
Ref: Daniel (1902), 183

Delattre Explosive. The Société anonyme pour la fabrication des cartouches et explosifs, Bruxelles, Belgium patented in 1898 the following compn: Ba nitrate 78, aniline nitrate 12 & Amm oxalate 10%
Ref: Daniel (1902), 184

Delay. A mechanical, electronic, or explosive train component which introduces a controlled time delay in some phase of the arming or functioning of a blasting cap or fuze
Ref: Glossary of Ord (1959), 91-L

Delay Action Bomb. See Vol 2, p B239-R and All & En Exps (1946), 162

Delay Action Fuze. See Delay Fuze

Delay, Arming. It signifies either: a)The interval expressed in time or distance betw the instant a piece of ammo carrying the fuze is launched and the instant the fuze becomes armed (See Arming in Vol 1, p A481-R) or b) The time interval required for the arming process to be completed in a non-launched piece of ammo
Ref: Glossary of Ord (1959), 91-L

Delay Blasting Cap. It is a blasting cap (See Vol 2, pp B185-B189) in which a delay element is inserted betw the primary and secondary (base) charges. In case of electric blasting caps, the delay element is inserted betw the electrical firing element and the base chge

As an example of a non-electric cap may be given that of Nash (Ref 1). It includes a rigidly and permanently sealed ventless casing, an igniter chge, a fuse and a detonating chge. The powder train of the fuse comprising in admixture: a substance from the group consisting of S, Se or Te; an oxidizing agent which when burned with said substance will react without the evolution of gas in quantity sufficient to rupture the casing; and an inert sub-

stance which will exert a control on the burning rate of the mixture

Berliner (Ref 2) describes delay electric blasting caps contg a length of slow burning fuse inserted betw the bridge wire and the base chge. The application of current ignites the fuse and, in due course of time, the spit of the fuse sets off the base chge and the blasting expl. Delay elec blasting caps are usually made with 10 different lengths of fuse so that in connecting the caps in the same circuit with a standard elec cap (which fires practically instantaneously) it is possible to shoot 11 series of blasts one after the other with one application of current. These caps find wide use in tunnel, shaft and drift work

The DuPont Co manufactures two different types of delay electric blasting caps: 1)ordinary or regular type (made in 10 std periods of delay) and 2)millisecond (MS) type (made in 19 periods of time). Regular delays are used principally in underground mining for driving tunnels, drifts & raises, and for sinking shafts. They are also used for blasting coal in underground anthracite mines and to a limited extent for stoping in metal mines. MS delay caps are used principally to detonate chges in small diam drill holes. Above ground, this includes blasting on rock excavation projects & quarries. They are also used in larger holes either for direct initiation of chges or for initiation of *Primacord* down lines at the top. They are widely used in nonmetallic mines and their use is increasing in metal mines (Refs 3 & 4)

Refs: 1)H.E.Nash, USP 1999820(1935) & CA (not found) 2)J.J.Berliner et al, "Explosives", New York (19??), p 36 (pamphlet) 3)Blasters' Hdb (1952), pp 97-98 and (1958), pp 107-10 4)Cook (1958), 16 & 350 5)Blasters' Hdb(1966), 91-93 [Three types are now manufd: a)"Acudet" Mark V b)MS and c)Coal Mining Delays]

Delay Charges; Delay Compositions or Delay Powders (Verzögerungssätze, in Ger). Delay charges of the nongaseous type are usually inorg mixts of oxidants and powdered metals which yield nongaseous products on combustion. They are used to ignite the rest of a powder train at a definite predetermined time within a certain allowable precision. Such a compn must burn for a definite time so as to produce a delay, and also supply sufficient heat at the end of the delay time to ignite the powder train in contact with it. A typical delay powder consists of Pb chromate 89, silicon 10 & linseed oil 1%. This compn yields 406 cals & 18mls of gas/g, whereas, BkPdr gives ca 700 cal & 300 ml of gas/g (Ref 7a)

A US military specification (Ref 8) describes zirconium-nickel alloy delay compns, for use in the delay element of time fuzes, as follows:

	Delay Types		
Composition, %	**I**	**II**	**III**
Barium chromate	60	60	60
Potassium perchlorate	14	14	14
Zr-Ni alloy 70/30	26	9	3
Zr-Ni alloy 30/70	-	17	23
Av burning rate, sec/inch	2.0	5.0	12.0
Std deviation, max sec	0.1	0.1	0.2

The proportions of the fuel ingredients may be varied within specified tolerences to meet the burning rate requirement

According to Ellern (Ref 15), the best delay compns, at present, are binary or ternary mixts of one fuel with one or two oxidizers. Some of the modern delay compns listed by Ellern (Ref 15, pp 283-84) are as follows:

Zr-Ni Type Delay Composition

Zr-Ni alloy, %	54
Ba chromate, %	31
K perchlorate, %	15

Boron Type Delay Composition, %

Amorphous boron, %	5	10
Ba chromate, %	95	90

Manganese Delay Composition

Manganese, %	44	37	33
Ba chromate, %	3	20	31
Pb chromate, %	53	43	36

"Exotic" Delay Mixtures

Niobium (Columbium), %	15	18	50	-	-
Tantalum, %	-	-	-	29	50
Ba chromate, %	85	82	50	71	50

The latter group above shows experimental mixtures representing the most modern trends in gasless delay compns

Some addnl info on delay chges, compns & pdrs will be found in the refs below and in the selected list of refs to the patent literature
Refs: 1)D.Hart, "Gasless Powders for Delay Elements of Fuzes", PATR **1239** (1943) 2)D.Hart, "Investigation of Use of Nitroindene Polymer in Powder for M54 Time Fuze", PATR **1296** (1943) 3)D.Hart, "Gasless Powders for Delay Elements of Fuzes", PATR **1406** (1944) 4)Ibid, PATR **1513** (1945) 5)D.Hart, "Investigation of the Use of Nitroindine Polymer in Powder for M54 Time Fuze", PATR **1525** (1945) 6)M.C.Epton, "Long Range Development of Delay Powders for Ammunition Fuze Application (Bomb Fuzes)", PATR **1686** (1948) 7)D.Hart, Ibid, PATR **1733** (1949) 7a)Anon, "Ordnance Explosive Train Designers' Handbook", NOLR **1111** (April 1952), pp 2-17ff 7b)Kirk & Othmer **11**, 325 (1953) 8)US Military Specification **MIL-C-13739** (Ord) (1954) 9)Y.Wakazono, JIndExpls Soc-Japan **15**, 156-61 (1954) & CA **49**, 11279-81 (1955)(The combustion mechanism of siliconminium Pb_3O_4 delay compn) 10)I.Ito & Y. Wakazono, JIndExplsSoc Japan **16**, 214-18 (1955) & CA **50**, 17453-54 (1956) (Study of size & shape of components in relation to burning time of a silicon - Pb_3O_4 delay compn) 11)B.Werbel, "Development of Delay Powders", PATR **2249** (1955) 12)B. Werber & S.Lopatin, Ibid, PATR **2477** (1958) 13)S.Nakahara & T.Hikita, KôgyôKayakuKyôkaishi **20**, 275-79 & 356-60 (1959) & CA **54**, 20207 (1960) (Measurements of combustion temp & combustion pressure of delay powders) 14)Ibid, **21**, 2-8, 86-92, 238-43 & 363-74 (1960) & CA **55**, 11847, 20433-34 (1961) (Mechanism of combustion, oscillatory burning, effect of pressure on burning velocity, and theory of burning of delay powder) 15)Ellern, Pyrotechnics (1961)

Selected List of References to the Patent Literature on Delay Compositions

F.Olsen, USP 1748455 (1930) & CA **24**, 1983 (1930) (A slow-burning powder suitable as a delay in fuzes contains KNO_3 74.1, charcoal 15.3, sulfur 8.4 & Triphenylphosphate 2.2%)
G.C.Hale, USP 1805214 (1931) & CA **25**, 3839 (1931) (A delay powder suitable for time fuzes is made of Pb oxide, Si and a substance such as glycerin capable of reacting with Pb oxide & forming a binder)
G.C.Hale, USP 1877127 (1932) & CA **27**, 191 (1933) (A delay powder which produces very little gas and suitable for fuzes is composed of $PbCrO_4$ 88, pulverized Al or Si 11 & linseed oil 1%)
G.C.Hale, USP 2450892 (1948) & CA **42**, 408 (1949) (Improved gasless delay powders: 1)$BaCrO_4$ 90, Zr 5 & S 5% 2)$SrCrO_4$ 80, Mn 16 & S 4% 3)$BaCrO_4$ 80, MN 13 & S 7% and 4)$BaCrO_4$ 74, Mn 24 & P 2%. The chromates & metals should pass a 250-mesh screen. The burning times of these powders in a train 2 inches long & 0.22 inch diam are as follows: 1)11 secs 2)13 secs 3)16 secs and 4)17 secs. None of the compns evolves gas)
G.C.Hale & D.Hart, USP 2461544 (1949) & CA **43**, 3620 (1949) [A fuze powder for use in delay trains to provide a definite burning-time interval consists of the following mixts: 1)Zr 75 & S 25% 2)Ti 60 & Te 40% 3)Ti 80 & S 20% and 4)Mn 70 & S 30%. These ingredients may be incorporated dry or mixed with water and then granulated]
G.C.Hale & D.Hart, USP 2467334 (1949) & CA **43**, 4856 (1949) (Gasless delay fuze powder is comprised of a mixt of Mn-Ti alloy 90 & sulfur 10%, both ingredients fine enough to pass a 250-mesh screen)
G.C.Hale & D.Hart, USP 2468061 (1949) & CA **43**, 5189-90 (1949) (A gasless fuze powder easy to ignite contains $BaCrO_4$ 96 & red P 4%)
I.O.Lewis, USP 2749226 (1956) & CA **50**, 13444 (1956) (A delay compn for elec detonators consists of Pb_3O_4 91-95% & Si 9-95% of particle size 5-20 microns. This mixt when pressed at 15 tons/sq inch and loaded into 0.130 inch diam tubes burns at 10 millisec/mm permitting its use in 25 MS delay detonators)
K.Hino et al, JapP 1142 (1957) & CA **53**, 719 (1959) (A safety delay compn for expls is made by adding Sb_2O_3 or Sb_2S_3 to the compn to prevent a large evolution of heat)
M.Yamada et al, JapP 6294 (1957) & CA **53**, 720 (1959) (Sparks & flames from the delay compn of blasting caps are inhibited by the use of a Cu powd which has absorbed alkali ions on its surface. Such a delay powd consisting of Cu powd 30, Pb_3O_4 70 & ferrosilicon 5 parts burns at the rate of 528.5± 10.5 milli-

sec per gram with no CH_4 ignition in 10 tests. The sorption of water by this compn in 1.5% after 100 hrs at 90% RH)
P.Becker & H.Mencke, GerP 1061245 (1959) & CA **55**, 25256(1961) (A short-time delay compn for use in elec fuses consists of Pb_3O_4 60 -50 & Si of particle size 2-10 microns 40-50%)
R.H.Comyn, USP 2974029 (1961) & CA **55**, 19246 (1961) (A nongaseous delay mixt, suitable for initiation of expl or proplnt chges, and having improved stability, sensitivity and a uniform burning rate w/o noticeable gas evolution consists of Co 41, $PbCrO_4$ 47 & $KClO_4$ 12%)
R.H.Comyn, USP 2990264 (1961) & CA **55**, 25256 (1961) (A delay mixt for use in ordnance missiles or app which forms solid combustion products consists of Mn 1/3, $PbCrO_4$ 1/3 & $BaCrO_4$ 1/3 parts)
G.A.Noddin, USP 2991714 (1961) & CA **55**, 25256 (1961) [A delay compn for elec detonators of uniform burning rate is prepd by mixing boron 1.4, red Pb 45.5 & 2PbO.PbHPO.-$\frac{1}{2}H_2O$ 53.1%. Conventional graining agents (Thiokol LP-2 or neoprene) in amts 0.5 to 1.5% of compn are added. An Al shell of pressed 99.5/0.5 PETN/graphite as a base chge, and a primer of LA contains the delay compn within a swaged Pb-tube carrier]

Delay Detonators. See under Initiators (Detonators, Primers and Initial Detonating Agents).

Delayed Action Bomb. See Bombs, Delayed Action in Vol 2, p B239-R

Delayed Contact Fire. Firing system arranged to explode a mine at a set time after it has been touched or disturbed
Ref: Glossary of Ord (1959), 91-L

Delay Element. A component which provides a specific delay between actuation of the CAD (Cartridge Actuated Device) and ignition of the proplnt
Refs: 1)Glossary of Ord (1959), 91-L 2)Encyclopedia of Explosives, Vol 2, p C72-L, under Cartridges 3)R.A.Kaufmann & J.H. Daniels, "Development and Qualification of Delay Elements, Propellant Actuated Device XM48, XM49, and XM50", FA (Frankford Arsenal) Report **R-1740** (1964), SEG TDR 64-39 4)US Military Spec MIL-D-13512 (Ord) (Delay elements, loading and packing)

Delay Element. This term may also be applied to a component which provides a specific delay in a blasting cap, detonator, fuze, etc
Refs: 1)Ohart (1946), 20 & 278 2)H.M.Kerr & C.R.Hall USP 2560452 (1951) & CA **46**, 1259 (1952) [Delay elements for electric blasting caps consist of homogeneously mixed powdered metals (such as Mg, Al, Ni, Zn), fuels (such as Si), oxidizers (such as Pb_3O_4) and "Misch -metall". By varying the compns, burning times of 15 to 579 milliseconds were obtained] [The Misch-metall (Ger for "mixed metal") is an alloy of rare earths of the following compn: Ce 49.0, La 25.6, Nd 16.0, Pr 4.6, Sm 2.0, Tb 1.0, Y 1.0 & Fe 0.8%] 3)McAdam & Westwater (1958), 57 4)M.Yamada & J.Yonezawa, Kôgyô-Kayaku Kyôkaishi **19**, 118-21 (1958) & CA **53**, 11980 (1959) (Combustion mechanism of the delay element) 5)Addnl Ref: Collective, "Ordnance Explosive Train Designers' Handbook", NOLR **1111**, US Naval Ordnance Lab, Washington, DC (1952) [Characteristics of Delays and Delay Elements. This includes, pp 5-2 to 5-18, Black Powder Delay Elements, mostly obturated column types with or w/o baffles. Some vented column types, as well as pressure type and ring type are also described. Construction of components are described on pp 5-18 to 5-33 and effects of variables on burning time of delay elements are discussed on pp 5-33 to 5-49. Gasless Delay Elements are described on pp 5-49 to 5-58 and these include Electric Delay Fuze Primers MK 115, 116 & 117; Electric Delay Detonator MK 35, Mod 1; Experimental Obturated Percussion Delay Detonator and Non-Obturated Delay Element. Other Delay Elements, such as Millisecond Lead Styphnate Delay, 250 Millisecond Nitrostarch Delay, Flame Initiated 0.10 Second Flash Delay Detonator and Stab Initiated 0.02 Second Delay Detonator. Numerous refs on Delays and Delay Elements are included in this book]

Delayer. A substance mixed with the proplnt of a solid fuel rocket to decrease the rate of combustion (Ref 2) Delayers may also be called "ballistic agents" (such as carbon

black, DNT, Nitroguanidine & wax) which alter the burning props of the proplnt, and "inhibitors" (such as strips of plastic cemented to the grain surface or an inert fuel w/o oxidizer applied by dripping, painting or spraying) which prevents burning on selected areas of the proplnt surface (Ref 1). See also "deterrents", p C381-L in Vol 3
Refs: 1)Warren (1958), 32839 2)Ord Tech Term (1962), 94

Delay, Explosive Train. An expl train incorporating some delay. The train consists of combustible and expl elements arranged in the order of decreasing sensitivity inside a fuze, projectile, bomb, gun chamber or the like. The function of the expl train is to accomplish the controlled augmentation of a small impulse into one of suitable energy to cause the main chge of ammo to function. A "fuze explosive train" may consist of a primer, a detonator, a delay, a relay, a lead and booster chge, one or more of which may be omitted or combined. If the bursting chge is added to the foregoing train, it becomes a "bursting charge explosive train". A "propelling charge explosive train" may consist of a primer, igniter and a proplnt (Refs 1, 2 & 3)

Explosive trains for bombs, with/or without delays are described in Vol 2, pp B223-L & B224 of this Encycl and in Ref 1, p 20
Refs: 1)Ohart (1946), 20, 123, 138 & 277 2)Glossary of Ord (1959), 91-R & 110-R 3)Ellern (1961), 141-53

Delay, Functioning. The interval expressed in time or distance betw initiation of the fuze and detonation of the bursting chge
Ref: Glossary of Ord (1959), 91-L

Delay Fuze or Delayed Action Fuze. A fuze incorporating a delay element. See under Fuzes

Delay Igniters (Zeitzünder, in Ger). Various chemical and mechanical delay igniters are described by A.Stettbacher in Nitrocellulose **6**, 202-04 & 220-25 (1935) and **9**, 75-7, 100-01 & 138 (1938)

Delay to Ignition and Its Temperature Coefficient. When an expl or proplnt is heated to a sufficiently high temp, expln or ignition takes place after a certain delay, known as the *induction period*

Ubbelohde et al (Ref) describe a method of detg this period for initiating expls, such as LA (Lead Azide), MF (Mercuric Fulminate), LSt (Lead Styphnate), BaSt (Barium Styphnate) and Basic LDNR (Lead Dinitroresorcinate)

The heat sensitiveness of an initiator can be characterized by the following Arrhenius type equation:

$$\mathrm{Log}_{10}\, Y = E/4.57T+B,$$

where Y is the induction period; E the activation energy of the physico-chemical process controlling it, in kcal/mol; B is a constant, expressed in kcal/mol; and T is absolute temp of ignition

Ubbelohde also investigated some initiating mixts, such as ASA (Azide-Styphnate-Aluminum) - the standard British Service initiator (See Vol 1, p A493-R). He found that in this mixt LSt plays the predominant rôle in the heat sensitiveness, improving the thermal pick-up and lessening the tendency of LA to fail to detonate on heating

He also examined some initiators by X-rays and found, for example, that the grains of British Service LA (undextrinated) consisted largely of single crystals, whereas the grains of dextrinated LA consisted of round polycrystalline formations

The table which follows gives the activation energy E (as calcd from the above Arrhenius type equation), as well as the value B for various initiating agents and extrapolated temperatures in °C, such as: a) t°_{0} -maximum temp for 0% ignitions; b) t°_{100}-minimum temp for 100% ignitions; and c) $t^{\circ}_{5\ \mathrm{sec}}$-ignition temp when delay was 5 sec

Table
Thermochemical Properties of Initiating Agents

Initiator	E	B	t°_{0}	t°_{100}	t°_{5} sec
LA(Service)	41.3	-13.97	343	326	328
LA(Dextrinated)	23.4	-8.2	-	283	304
MF	25.3	-10.76	208	-	-
Basic LDNR	22.9	-7.90	308	-	-
Ba St	35.4	-11.26	373	-	-
LSt	61.6	-21.91	324	262	271
ASA(Service)	62.6	-	-	275	296
ASA(Commercial)	57.9	-	-	270	296

Notes: a) ASA(Service) contains undextrinated LA, whereas ASA(Commercial) contains dextrinated LA b) Ignition temps were detd by heating a sample (0.02g for dextrinated LA and 0.01g for other expls) in a suitable container starting from room temp, and using a controlled rate of rise of temp. In the 2nd series of tests, the cold container with sample was suddenly plunged into a bath maintained at constant temp which was close to or slightly above t°_{100}, and the time interval before the ignition takes place (known as delay to ignition or induction period) was measured and the temp exprapolated to 5 sec period. Detailed description of procedures is given in Ref, pp 206-07
(See also Compression, Effect on Sensitiveness to Initiation in Vol 3, p C 492-L)
Ref: A.R.Ubbelohde et al, TrRoySoc **A241**, 204-22 (1948)

Delay Miniature Initiators. See W.R.Peterson, "Development of Miniature Delay Initiators", FA (Frankford Arsenal) Report **R-1690** (1963); ASD-TDR-63-649

Delay-Relay Element. It is a component of HE train which introduces a delay in the time of functioning of the fuze. The *delay* element is always followed by a *relay* and the two are really one unit. The relay is an expl (such as BkPdr) designed to provide continuity of the expl train by picking up the weak flash from the delay element and augmenting it sufficiently to initiate, by detonation, a succeding component. The delay-relay element is located in the fuze of a projectile betw the percussion primer and the detonator (Ref 1)

The compn of Delay Charges (qv) has already been discussed. Black Powder (See Vol 2, p B171-L) is sometimes used as both a delay & relay chge, in pellet form, in military applications (Ref 2)

See also Ref 3

Refs: 1)Ohart (1946), 19, 20, 123, 138 & 277 2)D.J.Zauder & M.C.Epton, "Characteristics of M204 Hand Grenade Fuzes Loaded with Nickle-Zirconium Type Delay Powder", PATR **2178** (1955) 3)S.Ordierno, "Information Pertaining to Fuzes", Ammo Engrg Directorate, PicArsn, Dover, NJ, Vol **4** (1964), Sections IV A-IVF, VII A - VII C, VII F, and IX A - IX B

Delay Release Assembly. An item which releases a retardant device on a bomb, to reduce the velocity of the bomb in flight.
Ref: Glossary of Ord (1959), 91-R

Delay Train. See Delay Explosive Train

Delépine, Marcel (1871-??). French chemist & professor at École de Pharmacie and at the Collège de France. Professor Delépine's published researches have been many & varied in the fields of organic, inorganic & optical chemistry. Also many honors have come to him including president of the French Chemical Society and the rank of Officer in the Legion of Honor
Ref: R.E.Oesper, JChemEduc **27**, 567-68 (1950) & CA **45**, 7 (1951)

Delhorbe (Explosif). Accdg to Daniel (Ref 1), it was prepd by dissolving 50 parts of K chlorate in boiling water and adding it to 5 parts of flour (sawdust or starch), 1 part of paraffin and 1 part of carbon black. Accdg to Giua (Ref 2), it was prepd by adding gradually to a concd soln of 87 parts of K chlorate in boiling w the following ingredients: 8 parts of flour (sawdust or starch), 3 parts of paraffin and 2 parts of carbon black. This expl was tested in 1898 by the Commission Française de Substances Explosives and was not approved for use in France
Refs: 1)Daniel (1902), 184 2)Giua, Trattato **6** (I) (1959), 396

Deliquescence of Explosives. It is the gradual liquefaction of expls, which are hygroscopic, caused by the absorption of atmospheric mois-

ture. Deliquescence may be determined by gain of weight at desired relative humidity. See Hygroscopicity and Its Determination
Refs: 1)Marshall **2** (1917), 417-19 2)Hackh's (1946), 257

Delivered or Characteristic Velocity. This is a term frequently employed to compare the performance of different rocket engines. It measures the effectiveness with which the chemical reaction is accomplished in the combustion chamber. The characteristic velocity, denoted by C*i*, is calcd from the formula:

$$C^* = Vj/C_F = gI_s/C_F = gF/\dot{W}\ (1/C_F)$$

where Vj = effective exhaust velocity
C_F = thrust coefficient
g = gravitational acceleration (general)
I_s = specific impulse
F = thrust
and $\dot{W}$ = wt rate of proplnt consumption
The exptl detn of C* can also be accomplished by means other than measurement of the thrust developed by the rocket engine (Ref 3)
Refs: 1)Warren (1958), 81 2)US Ord Corps Pamphlet ORDP **20-175**, "Solid Propellants", Part **1** (1961), p 11 (now designated AMCP **706-175**) 3)US Army Materiel Command Pamphlet AMCP **706-282**, "Propulsion and Propellants" (1963), 32

Delivery Error. The dispersion of burst points around a desired point due to the ballistic characteristics of an atomic projectile, atmospheric conditions and other factors
Ref: Glossary of Ord (1959), 91-R

Delivery System. The means of delivering atomic ammunition to the target
Ref: Glossary of Ord (1959), 91-R

Delmhorst Moisture Indicator. This instrument and its suitability for detg the moisture content of AN were discussed by:
L.H.Eriksen, PATR **1214** (Nov 1942)

Delta Rocket. A launch vehicle or booster or booster system consisting of one to three stages. Depending upon the thrust desired, the fuel may be LOX (liquid oxygen explosives or oxyliquits) & RJ-1 or RP-1, unsym- dimethyl hydrazine (UDMH) & white inhibited fuming nitric acid (WIFNA), or a solid proplnt.
Ref: C.Butler, Missiles & Rockets, **11**, No 13 (24 Sept 1962), 28

Delvigne Explosive. A Belgian expl consisting of AN 78-95, Ca silicide 2.5 to 20.0 and TNT (PA or NG) 2.5 to 8.0 parts
Ref: M.Delvigne, USP 977076 (1910 & CA **5**, 786 (1911)

DeMacar Explosives or Macarites. A series of expls based on lead nitrate, patented in Belgium in 1899 & 1900. Other ingredients were (one or several) derivatives of phthalic acid, nitrated hydrocarbons, azocompounds, compds formed as a result of reactions between nitrated hydrocarbons & azocompds, and NC's

One of the varieties was similar to the expl patented in 1895 in US by Divine (qv)
Ref: Daniel (1902), 184

DeMarre Formula. A formula expressing the relationship between the projectile characteristics and armor plate penetration capabilities
Ref: Glossary of Ord (1959), 91-R

Demétriade, Jonesen & William Explosive. A Sprengel-type expl, patented in 1905, consisted of K chlorate 61, nitrobenzene 24, PA 16 & cellulose 5%
Ref: Giua, Trattato **6** (I), 398

Demi-Culverin. See under Culverin in Vol 3, p C573-L

Demilitarization. It is the taking away of all military organizations and installations. Also the disassembly, destruction or any other action which renders munitions, weapons and other matériel which is lethal, hazardous, classified, or of a specialized nature, harmless and ineffectual for military purposes
Ref: Glossary of Ord (1959), 91-R

Demolition. Act or process of demolishing (destroying) or making unusable objects either belonging to the enemy or abandoned by him; or to make gaps thru enemy obstacles in order to facilitate military operations. Demolition may be accomplished by the use of expls, fire, bulldozers, etc. The objects to be de-

stroyed might include fortifications, buildings, railroad tracks, locomotives, cars, trucks, bridges, industrial installations, water supply towers, electric power plants, airfields, roads, cannons, tanks, boats, utility lines, trees, fences, etc

Demolitions may be divided into two classes: a) *Deliberate Demolitions* (Démolitions préméditées, in Fr.) When planning a retreat, it is the usual practice to mine in advance the structures of any strategical importance so that they can be destroyed on retreating

As an example, when the French were retreating during WWII, they destroyed in their country all the bridges, industrial installations, oil storage tanks, all electrical lines including concrete poles, etc. When the Germans retreated from occupied territories they mined not only the objects of strategic importance, but also some churches, castles, palaces, monuments, etc

Deliberate demolitions permit economies in the use of expls because time permits judicious placing and thorough tamping of charges
b) *Hasty Demolitions* (Démolitions précipitées, in Fr). When it is necessary to destroy something in a hurry, as for example in operating in enemy territory, the time is limited and larger quantities of expls are required than in deliberate demolitions

Generally, in demolitions, structures are destroyed at their most vulnerable points so that a minimum quantity of expl will cause maximum damage

Refs: 1)Daniel (1902), 185-87 2)Pepin Lehalleur (1935), 448-53 3)Anon, "Explosives and Demolitions", US Dept of the Army Field Manual FM **5-25** (1954) 4)Anon, "Demolition Materials", US Dept of the Army Tech Manual **9-1946** (1955), 153-55 and TM **9-1375-200** (1964), 16, 80, & 130 5)Glossary of Ord (1959), 91-R 6)Anon, "Care, Handling, Preservation and Destruction of Ammunition", US Dept of the Army Tech Manual TM **9-1300-206** (1964), 177 7)Blasters' Hdb (1966), 475 (Demolishing bridge piers)

Demolition Bangalore Torpedoes. See Vol 2, p B16-R & GP (General Purpose Bomb), pp B227-R & B228-L

Demolition Blocks. See under Demolition Explosives

Demolition Bombs. See Vol 2, p 239-R

Demolition Charges. See under Demolition Explosives

Demolition Clam. A small plastic case designed to hold approx 0.5 lb of plastic HE. Another design of the demolition clam w/o case consists of a retangular block of PEX (1.7 lb) provided with 4 permanent magnets and connections for 2 delay-type firing devices. The demolition clam is used to destroy steel structures such as gasoline storage tanks, pipe lines, vehicles, etc (Refs 1 & 2)

Refs: 1)F.B.Sando, "The T41 High Explosive Demolition Clam", PATR **2310** (July 1956) 2)F.B.Sando, "T41E1 High Explosive Demolition Clam", SFAL Notes on Development Type Material No **175**, Picatinny Arsenal (Feb 1958)

Demolition Devices. See under Demolition Explosives

Demolition Explosives (Demolition Blocks, Demolition Charges and Demolition Devices) (See also Demolition Hoses, Demolition Kits and Demolition Snakes). Demolition expls are those used for demolition purposes. They are mostly HE's (such as TNT, PA, Tetryl, PETN, Dynamite, etc), made in the shape of blocks, cartridges, etc. Some LE's, such as compressed wet Guncotton, can also be used. Under the term *Demolition Devices,* Ohart (Ref 8) described Bangalore Torpedoes (See Vol 2, p 16-R of this Encycl) and various demolition blocks, including shaped charges. In Ref 12a, the same devices are listed as *Demolition Materials* (qv)

During WWII a wide use was made in demolition practice of the so-called *Shaped Charges,* which utilize the Munroe-Neumann Effect. In these chges the expl energy is concentrated in one direction and is greatly increased if the cone of the shaped charge is lined with a thin sheet of metal, such as Cu, Zn, steel, etc. The concn of energy in one direction permits greater penetration than would be obtd with ordinary expl chges, consequently they are

used for blasting holes thru enemy fortification. For example, when a shaped chge of Pentolite (50/50 PETN/TNT), is exploded against a concrete "pill box", it will blow a hole 2 to 6" in diameter thru the wall, depending upon the design and wt of the shaped chge. If the pill box is occupied by soldiers, they would be subjected to the disastrous effects of: a) a large number of high velocity missiles; b) high temperature flame; and c) a blast effect. Once an opening has been made in the wall, the sticks of Dynamite or other demolition expl could be inserted and detonated to complete the destruction of the fortification and personnel

In the above mentioned case, the shaped chge serves only as an auxiliary demolition expl, but it can also be used alone for complete destruction without resorting to the use of other expl chges. For example, in destroying a bridge, it is possible to obtain sufficient weakening of its supporting columns, by firing a group of shaped charges against them

In order to increase the penetration of a shaped charge, it is placed at a distance of several inches from a target. This distance is known as *stand-off*

The following demolition expls, including some shaped charges were used during WWII by the **US Forces** (Refs 5, 8, 9, 11, 12, 13, 15 & 17):

Table

U.S. Demolition Explosives

(Except Shaped Charges)

Explosive	Detonation Velocity, m/sec	Relative Effectivness TNT = 1.00	Principal Uses
TNT, rectangular blocks ¼, ½ & 1 lb	6400	1.00	General military use in forward areas; destruction of mines, booby traps, ammo
AN Cratering Charge	3350	0.42 (See also Vol 1, p A355)	Cratering
Nitrostarch in ¼ lb blocks	4570	0.86	Same as TNT
Tetrytol (75% Tetryl). M2 Block (2.5 lb) (See Note below)	7020	1.20	Same as TNT
Composition C-2 (RDX-C2), M3 Block (2.5 lb)	7930	1.34 (See also Vol 3, p C484-R)	Same as TNT
Composition C-4 (RDX-C4), M4 Block (plastic expl packed in a box 6¾x1½x1 inch)	8040	1.30 (See also Vol 3, p C485-L)	Same as TNT
DYNAMITES:			
Straight, 60%	5900	0.83	Land clearing, cratering, and general use in rear areas
Ammonia, 60%	4600	0.53	
Gelatin, 60%	6200	0.76	
Blasting Gelatin	8000	0.92	

Note: Eight M2 blocks connected by primacord comprise the M1 chain of demolition blocks

Table
US Demolition Explosives
(Shaped Charges)

Designation of Charge & Name of Explosive	Weight of Charge and Description of Arrangement	Penetration in Steel Armor Plate and Reinforced Concrete, inches
M3; Pentolite, 50/50	30 lb. enclosed in a metal container 9" diam x 12.5" high. Total weight 40 lb. Stand-off support is provided by 3-15 inch legs	Armor plate; hole 2.5 diam to a depth of 20. Concrete: 3.5 hole to a depth of 60
M2A3; Pentolite, 50/50	11.5 lb enclosed in a fiber container Total weight 15 lb. The fiber cylinder extends below the base of the chge to give the "stand-off" at the required distance	Armor plate; hole 2.5 to a depth of 12. Concrete: 2.75 hole to a depth of 36
M2; M2A1; and M2A2; Pentolite 50/50	10 lb enclosed in a fiber container arranged similarly to M2A3	Armor plate: 1.25 hole & depth 8. Concrete 2 hole & depth 25 to 30
M1; Pentolite 50/50	10 lb encased in a metal container provided with three "stand-off" legs	Same penetration and diameter of holes as above
M3E2	See Ref 17D	
M2A3E1	See Ref 17 E	

The following are some **British** demolition expls used during WWII (Refs 5, 9 & 11):
a) Guncotton (wet); 15 oz or 1 lb block in tin, internal & external uses
b) Slab TNT-TNT (Trotyl) 70 & Tetryl (CE) 30%; 1 lb block in cardboard; external use
c) Slab TNT (Australian), M II; 1.75 lb block in cardboard container; 1/3 TNT at each end and 1/3 - 70/30 Tetryl/TNT in center; internal & external uses
d) Golignite (commercial) - 60% Gelatine Dynamite; 1.6 oz & 4 oz cartridges; internal uses
e) Gelignite (commercial Australian) - NG, plus AN; 5.5 oz cartridges; internal & cratering uses
f) Monobel (commercial Australian) - AN, sensitized; 5 oz cartridges; same uses as above
g) Ammonal - AN, TNT & Al; 25 & 50-lb blocks; cratering uses
h) Explosive 808 - Desensitized Polar Blasting Gelatin; 4 oz cartridges; internal & external uses
i) Plastic HE - compn not given; 4 oz cartridges; internal & external uses
j) Beehive, 6in MI - TNT 75 & PETN 25%; 6.5 lb shaped charge in sheet metal container, 6" diam & 7" high placed on three 4.5 in legs (stand-off)
k) Beehive, 50lb - TNT 75 & PETN 25%; 30 lb shaped chge in sheet metal container; total wt 50 lb
l) Arched Demolition Charge; 26 lb shaped chge of Pentolite in arched linear, semicylindrical tin plate container of the following dimensions in inches: 9x17x5 or 9x12.75x6.37; the radius of inner arch was 2.25

The following **French** demolition expls are listed in Ref 5, p 35:
a) Small Cylindrical. A brass cartridge, ca 4.9" long and 1.1" diam, filled with Mélinite

(Picric Acid); total wt 2 lb 3/4 oz; internal & external uses
b) Large Cylindrical. A brass cartridge, ca 6.4" long & 2.5" diam filled with 2 lb 3/4 ox of PA; total wt not given; same uses as above
c) Model 28, 135g. A block of PA in a brass case 5.7x1.3x0.8" ; same uses as above
d) Model 28, 1 kg. A block of PA in a brass case 10.75x2.4x1.5"; same uses as above
e) Model 28, 10kg. A block of PA in a brass case 10.75x6.6x6.6"; same uses as above
f) Model 28, 20kg. A block of PA in a brass case 8.6x2.5x2.5"; same uses as above
g) Model 31A, 1kg. A block of cast PA in a brass case 8.8x2.5x2.5"; same uses as above

The following **German** demolition expls are listed in Refs 5, 11 & 16:
a) Bohrpatrone 28. A cartridge 3.9" long & 1.2" diam consisting of 3.5 oz TNT or PA wrapped in waxed paper; internal & external uses
b) Sprengkörper 28. A block 2.75x2x1.5" consisting of 7 oz TNT or PA wrapped in waxed or varnished paper; same uses as above
c) Sprengbüchse 24. A block of TNT, PA or 90/10 PETN/Wax, weighing 2 lb 3 oz placed in a zinc case 7.9x2.9x2.2"; cratering and internal & external uses
d) Gebalteladung 3kg. Several blocks of TNT or PA, total wt 6.5lb, placed in a zinc case, 7.7x6.5x3.0"; provided with carrying handle; same uses as above
e) Gebalteladung 10kg. Blocks of TNT, total wt 22 lb, in zinc case 10.4x7.6x5.75"; same uses as above
f) Demolition Charge, 12.3kg. A triangular block of 27 lb RDX/TNT in a seamless steel container; external uses only
g) Plastit. A block of plastic expl RDX/Oil, weighing 1 lb 1.5 oz; internal & external uses
h) Hohlladung, 400g. A shaped charge of PETN/Wax, weighing 14 oz, in an aluminum cylinder 3.1" high & 2.8" diam
i) Hohlladung, 12.5kg. A shaped chge consisting of 28 lb of TNT (weighed with container), in a sheet iron cylinder 8.1" high & 11" diam
j) Hohlladung, 13.5kg. A shaped chge consisting of 21 lb 3 oz of 50/50-RDX/TNT (weighed without container) in a mild steel cylinder 9" high and 13.5 diam
k) Hohlladung, 50kg. A shaped chge consisting of 110 lb TNT (weighed with container) in a sheet iron cylinder 10.2" high and 20" diam, provided with a carrying handle
l) Hafhohlladung, 500g. A magnetic antitank shaped chge of 50/50-RDX/TNT weighing 1 lb 1.25 oz in metallic container 7.7" high & 6.2" diam, provided with plywood base and 3 pairs of magnets
m) Hafthohlladung, 3.0kg. A magnetic antitank shaped chge of 50/50-RDX/TNT mixt in metal cylinder 7.7" high & 6.2" diam, provided with 3 pairs of magnets
n) Hohlladung, 3.6kg A magnetic antitank shaped chge of TNT in aluminum cylinder 10.75" high & 6.2" diam, provided with 3 pairs of magnets

The following **Italian** demolition expls are listed in Refs 5 & 11:
a) Charge, 100g. A block or cartridge of Tritolo (TNT), weighing 3.5 oz; cratering and internal & external uses
b) Charge, 150g. A block or cartridge of TNT weighing 5.25 oz; same uses as above
c) Charge, 200g. A block of Pentrite (PETN) weighing 7 oz; internal & external uses
d) Charge, 500g. A block of PETN or TNT weighing 1 lb 1.5 oz; same uses as above

The following **Japanese** demolition expls are listed in Refs 5, 11 & 12, p 211-12
a) Prepared Charge. A block in varnished paper consisting of Oshokuyaku (pressed PA), weighing 2 lb 3 oz; internal & external uses
b) Plastic Explosive. A cartridge in parchment paper consisting of 4 oz of Oshitsuyaku (80/20-RDX/Vegetable oil); same uses as above
c) "97" Explosive. A block or cartridge in paper consisting of 3.3 oz of Shimose (cast PA) or Haishokuyaku (RDX 17, Amm perchlorate 77, Si carbide 1.5 & paraffin 4.5%; same uses as above
d) Demolition Can, 1 kg. A block in can consisting of 2 lb 3 oz Shimose (cast PA); underwater uses
e) Demolition Can, 5 kg. A block in can consisting of 11 lb cast PA; internal & external uses
f) Tanoyaku, 30kg. A block consisting of 32.3 lb of compressed RDX; same uses as above
g) Shoanbakuyaku Charge consists of AN 79,

Dinitronaphtalene 10, NaCl 10 & sawdust 1%
h) Entoyaku Charge consists of K chlorate 80, DNT 16 & castor oil 4%
i) Ennayaku Charge consists of K chlorate 80, MNT 15 & castor oil 5%

The following **Russian** demolition expls are listed in Ref 14:
A) Podryvnaya Shashka (Blasting Sword) or Piroksilinovaya Shaska (Guncotton Sword). A cartridge consisting of compressed 13% N Nitrocellulose, called in Rus Piroksilin, contg ca 18% moisture. It was made since 1890 in various sizes. Its use was discontinued after WWI in favor of Trotil (TNT) demolition charges
B) Trotil (TNT) demolition charges used during WWII were as follows:

a) Large rectangular, 5x5x10 cm, weighing 400g

b) Small rectangular, 5x2.5x10 cm, weighing 200g

c) Cylindrical, 3 cm diam & 7 cm long, weighing 75g

Refs: 1)Marshall **2** (1917), 551-55 (Demolition expls) 2)Pepin Lehalleur (1935), 448-53 (Formulas for calcn of amts of demolition expls required for destruction of various objects, such as fortifications, railroad tracks, tunnels, weapons, vehicles, etc; methods of attaching some demo chges to various objects in order to achieve the best results with minimum amt of expls) 3)J.D.Hopper, "Investigation of Demolition Explosive Developed by Trojan Powder Company", PATR **848** (1937) 4)Hayes (1938), 600-603 (Demolition bombs) 5)Anon, "Explosives and Demolitions", US War Dept Field Manual FM **5-25** (1945) 6)Kay Brothers Ltd & H.K.Hartley, Brit P 572994 (1945) & CA **43**, 9536 (1949) (Demolition charge adhesive prepd by dissolving butadiene-styrene in a solv and incorporating a mineral oil & hydroquinone) 7)Pérez Ara (1945), 696-98 (Application of expls for demolition of military objects) 8)Ohart (1946), 369-70 (Demolition-type antipersonnel mines); 375-76 (Demolition devices) 9)All & En Expls (1946), 25 (Beehive demolition charges); 84 (Brit demolition and cratering chges contg 50/50-Amatol and Ammonals, such as AN 65, TNT 15, Al 17 & charcoal 3%) 9a)S.Fleischnick, "Demolition Charges", PA Tech Div Lecture (25 April 1947) 10)J.E.Riley, USP 2594996 (1952) & CA **46**, 7332 (1952) (A demolition expl of high strength and of low sensitivity to impact and high order of detonation is composed of NG 4-10% and cellulose acetate having a viscosity of less than 80 secs & combined AcOH content 54-56% plus other ingredients) 11)Anon, "Explosives and Demolitions", US Dept of the Army Field Manual FM **5-25** (1954) 12)Anon, "Military Explosives", US Dept of the Army Tech Manual TM **9-1910** (1955), 204, 206-07 & 211-12 (An Amer expl for demolition purposes standardized shortly before WWII consisted of Nitrostarch 34.5±2.5, Ba nitrate 43.5±1.5, TNT 15.0±2.0, Al 3.0 ±1.0, graphite 2.0±1.0, coal dust 1.5±0.5, paraffin 0.6±0.6, dicyandiamide 1.0±0.5 & moisture 0.75±0.75%. The Ba nitrate was coated with paraffin before the ingredients were pressed into 1/4-lb pellets, of d 1.75± 0.10. Four of these were covered with paper to form 1-lb packages. The compn was required to undergo no ignition or expln when heated to 100° for 48 hrs. It crumbled on breaking action

Its other props were: Brisance, by Sand Test 90% of TNT; Friction, by Pendulum Test - no explns; Hygroscopicity - 2.1%, when exposed to air of 90% RH at 30°; Power, by Ballistic Pendulum 96% of TNT; Rifle Bullet Impact Test - explns; Sensitivity to Initiation - more sensitive than TNT; Stability as packed, as shown by long term storage tests - satisfactory; Stability in the 75° International Test - loses 1% of wt but no deterioration; Stability at 100° in 96 hrs - volatilization, but no decompn

Other expls used during WWII for demolition purposes were Dynamites of the types procured as commercial items, such as Straight Dynamites, Ammonia Dynamites, Gelatin Dynamites and Ammonia Gelatin Dynamites 13) Anon, "Demolition Materials", US Dept of the Army Tech Manual **TM9-1946**(1955) 14) B.T. Fedoroff et al "Dictionary of Russian Ammunition and Weapons" PATR **2145**(1955), p Rus 13-R 15) Anon, "Ammunition General ", US Dept of the Army Tech Manual TM **9-1900**(1956) 16) B.T. Fedoroff et al, "Dictionary of Explosives, Ammunition and Weapons" (German Section), PATR **2510**(1958), p Ger 34 17) US Military Specifications: A) MIL-B-20397 (Demolition Block M3) B) MIL-C-12918A(MU) (Demolition blocks M5 & M5A1)

C) MIL-C-46246C(MU) (Charge, Demolition, TNT, Block, 1/4 Pound, 1/2 Pound, and 1 Pound) D) MIL-C-46594 (4) (MU) (Charge, Demolition, Shaped, 40 lb, M3E2 and Inert, M3, Loading, Assembling and Packing) E) MIL-C-13139B (MU) (Charge, Demolition, Shaped, 15 lb, M2A3 and (M2A3E1, Parts for) F) MIL-C-46460A (MU) (Charge, Demolition, M91 Block, Parts for Loading, Assembling and Packing) G) MIL-C-2156A (Charge, Demolition, Shaped, 15 lb Practice M2A3 and HE M2A3E1, Loading, Assembling and Packing) H) MIL-D-23850 (WP) (Demolition Firing Device Mk 24, Mod O)
18) Bureau of Ordnance, "Ammunition Ashore, Production and Renovation", Vol **2** (1958), Articles 6606 & 6609

Demolition Hoses. See Vol 2, p B17-R, under BANGALORE TORPEDOES

Demolition Kit or Unit. Any of several groups of items of an expl nature, with the necessary nonexplosive accessories and tools, with specially designed containers and carrying attachments, to enable efficient performance of particularly designated demolition tasks (Refs 2,3,7 & 13)

Demolition kits include:

a) *Demolition Kit, Bangalore Torpedo,* which consists of a number of Bangalore Torpedoes (See Vol 2, p B16-R) together with connecting sleeves and a nose sleeve to facilitate assembly of an expl unit of varying length (Ref 13 p 90)

b) *Demolition Kit, Blasting,* which comprises a collection of blasting charges, initiating devices and the necessary nonexpl accessories and tools with specially designed containers and carrying attachments, to enable the accomplishment of suitable demolition assignments by personnel groups of various sizes (Refs 3,7 & 13, p 86)

c) *Demolition Kit M 37* consists of 8 demolition blocks of Composition C-4 (2¼ x 2¼ x 11¾ inches), 8 block hook assemblies and two priming assemblies, each of which is a 5 ft length of detonating cord. On each end of the detonating cord, an adapter & booster (a chge of RDX) with appropriate clips are attached to permit the forming of junctions on main lines of detonating cord in a demolition system (Ref 2)

d) *Demolition Kit, M157,"Snake".* The kit developed in 1963 by the Munitions Command at PicArsn, Dover, N J, jointly with Emtex Division,Missile Systems of Texas, is an improvement over previous models of snakes described briefly in Vol 2, pp B17-R & B18-L. The kit is built in five-foot sections and measures over 400 ft long when fully extended. It is towed to the edge of a known minefield, pushed the rest of the way in, then detonated by machine gun fire. The concussion of the linear-shaped chge sets off any mines in the area, thus clearing a safe path for vehicles and troops (Refs 9,11,12 & 13, p 11T)

e) *Demolition Kit, Petard Type.* This kit invented by Kessenrich (Ref 1) contains a petard type expl use to penetrate a barrier. An annular chamber contains the expl chge which can be initiated either axially at the ends or in the middle. In operation, the inner wall consists of a liner for a shaped charge effect, and from which the container may be hung on a nail or wire inserted thru the passage and suspended from the barrier that is to be blasted

f) *Demolition Kit, Projected Charge.* A group of items including demolition charges designed for assembly into a device to be positioned by an external force. The kit serves for clearing mine fields. The early designs were termed "snake demolitions" (Refs 3,4,7,11 & 13, p 93)

g) *Demolition Kit, Training.* These kits are for the training of personnel in the use of demolition materials. The kits are similar in every way to others except that the expl components are removed to render them inert and are painted blue rather than olive drab. All inerted items are employed in exactly the same manner and with the same precautions as line kits in order that personnel be fully acquainted & conversant with all procedures. Training kits are described more fully in the Refs (Refs 2, p 110-18; 6,10 & 13, p 127)

See also Demolition Explosives and Demolition Materials

Refs: 1) G.J. Kessenrich, USP 2706949 (1955); CA (Not found) & Ordn **40,** 304 (1955) 2) Anon, "Demolition Materials", US Dept of the Army Tech Manual TM **9-1946** (1955), 108-10 3) Glossary of Ord (1959), 92-L 4) US Military Specification MIL-D-45411 (Jan 1960) (Demolition kit, Projected Charge M1E1, Loading, Assembling and Packing) 5) Ibid, MIL-D-4512

(Jan 1960) (Demolition kit, M1E1, Container and Accessories) 6) Ibid, MIL-D-46517 (Apr 1961) (Demolition Container, Ex 1 & Mod O for Demolition Kit, Projected Charge, HE, XM125 and Inert, XM126) 7) OrdTechTerm (1962), 95 8) US Military Specification MIL-D-46686 (Aug 1962) (Demolition Kit, Projected Charge, M173 and Practice, M174, Loading, Assembling and Packing) 9) Picatinny Arsenal News (16 Dec 1963), p 2 10) Ibid, MIL-D-46558A (1) (Feb 1964) (Demolition Kit, Projected Charge, HE, M125 and Inert M126, Loading, Assembling and Packing) 11) Ibid, MIL-D-45473A (Feb 1964) (Demolition Kit Projected Charge, M157, Loading, Assembling and Packing) 12) Ibid, MIL-D-45472B (3) (Parts for) 13) Anon, "Demolition Materials", US Dept of the Army TechManual TM **9-1375-200** (1964), 86, 90, 93 & 127

Demolition of Prestressed Concrete Bridge Beams with Explosives is discussed by J.A. Dennis in Engineer Research and Development Labs Rept **1830**, Fort Belvoir, Va (Sept 1965)

Demolition Sled, M3. A boatlike sled, 12 ft in length & 5 ft wide, manufd for the Army from fiberglass by Martin Co, Baltimore, Md, to carry expls and to blast a safe path thru mine fields in a matter of minutes. The sled is divided into three sections. The front section contains a 2-ft long Jato rocket attached to a linear charge of a plastic expl coiled in a center section. The rear section carries accessories such as a firing & towing cable and tools. The fully loaded sled weighs ca 3000 lbs. It can be towed over land or water and behind tanks or other tracked vehicles

Ref: Anon, Ordn **51**, 308 (Nov-Dec, 1966)

Demolition Rocket, 7.2 inch. Amer surface-to-surface rocket developed in 1943 by NDRC at CalTech, Pasadena, Calif. It can be fired from "WhizBang" 20-tube and "Grand Slam" 24-tube tank-mounted launchers against strongly built concrete positions

The US Navy fired them from LCM (3)'s (landing craft, mechanized) furnished with Woofus 120-tube launchers during attach on Southern France in August 1944

A chemical-type payload for this rocket (total wt 58.1 lb, payload 20 lb, length 4.1 ft, range 2 miles & velocity 475 mph) was developed later

Ref: F.I. Ordway & R.C. Wakeford, "International Missile and Spacecraft Guide", McGraw-Hill, NY (1960), 79

Demolition Materials. These include expl equipment or devices intended for destruction of obstacles (such as by Bungalore Torpedoes, Demolition Hoses and Snakes), fortifications and armor (such as by shaped charges), general material (such as by demolition blocks or cartridges) and special equipment (such as by destructors). Demolition materials include also devices used in conjunction with demolition expls, such as blasting caps, squibs, Bickford fuses, detonators, detonating cords, blasting machines, etc (Ref 2)

Destructors are components for demolishing or destroying certain pieces of equipment, particularly confidential equipment, the nature and operation of which it is desired to keep from the enemy hands. Destructors may be classified as those intended for radio equipment and those for control equipment and radar. Both types can be used for destroying guided missiles and their equipment. A detailed description of Amer destructors used during and after WWII is given in Refs 2 & 3. Ohart (Ref 1, pp 370-75) describes destructors M1, M2, M3, M4, M5, M6 & M7 (See also under Demolition Explosives and Demolition Kits)

Refs: 1) Ohart (1946), 370-76 2) Anon, "Demolition Materials", US Dept of the Army TechManual TM **9-1946** (1955) 27-118 3) Ibid, TM **9-1375-200** (1964)

Demolition Snakes. See Vol 2, pp B17 & B18 under BANGALORE TORPEDOES

Denaby Powders. Brit permitted coal mining expls:

a) *No1* - AN 34, K nitrate 33.5, TNT 13 & Amm chloride 19.5%; ballistic pendulum swing 2.41" (amm chloride is added as a cooling agent; it is supposed to react with K nitrate as follows:

$NH_4Cl + KNO_3 \rightarrow KCl + NH_4NO_3$ (Ref 2)
b) *No 2* AN 57.5 - 60.5, Na nitrate 5.0 - 7.0, Na chloride 19.0 - 21.0, TNT 11.0 - 13.0 & carbonaceous material 1.5 - 3.5%; powder of density 0.95 g/cm^3; power 58% of BG (Blasting Gelatin) (Refs 3 & 4)
c) *Denaby Powder, Sheathed* with either 30% Na bicarbonate or NaCl to give a safety expl having a power 58% of BG (Ref 4)
d) *Older Denaby Powder* (1891-1893) listed in Ref 1 under "Sécurites" had the following compn: K or Ba nitrate 73.18, DNB (Dinitrobenzene) 21.49, NC, plus charcoal 5.07 & moisture 0.26%
Refs: 1) Daniel (1902), 711-12 2) Marshall **3** (1932), 119 3) Taylor (1952), 21 4) Taylor & Gay (1958) 94

Denatured (or Industrial) Alcohol (Denatured Spirits). Colorless liquid (which is sometimes artificially colored blue or red to distinguish it from ordinary alcohol) consisting of ethyl alcohol and "denaturants". Its toxicity depends on denaturants used, of which the principal one is methyl alcohol. This alc produces blindness. Other denaturants may produce contact dermatitis, or asthma upon ingestion (Refs 1 & 2). Its fire hazard, accordg to Sax (Ref 3) is dangerous
Refs: 1) Kirk & Othmer **1** (1947), 252-88 & **1** (1963), 531-41 2) US Office of Internal Revenue Regulations 3, Industrial Alcohol, US Govt Printing Office, Washington, DC (1942) 3) Sax (1957), 247

Denespex. One of the Brit permitted NG gelatin expls belonging to the class of EqS [Equivalent (in Safety) to Sheathed] expls. Its compn is not given in Ref
Ref: Taylor & Gay (1958), 100

Denigès, Georges, Doctor of Science (1859-1951). French professor of analytical chemistry who designed many methods of analysis, some of them used in the expls industry. He published 680 papers in his field (Refs 1 & 2)
Refs: 1) J.A. Sánchez, "Life and Work of Prof Georges Denigès", Rev Asoc Bioquím Argentina **15,** 182-88 (1948) & CA **43,** 5649 (1949) 2) R. E. Oesper, JChemEduc **28,** 194 (1951) (Biography with portrait)

Denitration. Removal of nitric acid, nitrates or nitrogen oxides from acids and other substances. A few selected examples illustrating denitration are the following:
NC Articles. They are denitrated, w/o degradation of the resulting cellulose hydrate, with an aq soln of an alk reducing agent [such as NaHS, KHS, NH_4HS or $CA(HS)_2$] at a pH below 12 (Ref 1b)
Nitroguanidine. Denitration of N-nitroguanidine is 1st order except for a reverse reaction interfering at an early stage and rate increase caused by high initial concn of the compd, which have the same effect as addn of more concd H_2SO_4. An increase in denitration is found upon addn of $KHSO_4$ & $(NH_4)_2SO_4$ salts (Ref 2)
D-Mannitolhexanitrate. This compd in pyridine evolved gases (NO, N_2O & N) and mannitol - 1,2 4,5,6 - pentanitrate and $C_5H_5N.HNO_3$, after dilution with water. Approx 2 moles of C_5H_5N suffered ring cleavage, 0.25 mole of the hexanitrate was completely denitrated, and 0.75 mole of pentanitrate was formed (Ref 3)
Sulfuric acid. Denitration is accomplished by blowing air thru H_2SO_4 and diluting with a soln contg NH_4 salts. The entire process is carried out in a continuous-flow app (Ref 4). Industrial-scale equipment, for denitrating H_2SO_4, of new design is illustrated by Ponomarenko (Ref 5). Purification of concd H_2SO_4 and denitration of nitrogen oxides present is accomplished by use of 40% formalin soln as a reducing agent (Ref 6)

In the TNT manufg industry, the acid recovery house is equipped to operate a separate process called "denitration" or "acid recovery". During the mono-nitration process, the mono-mix acid becomes progressively weaker in HNO_3 and richer in water, so that the mono-waste acid resulting after nitration is complete contains only 4% of actual HNO_3. The mono-waste acid also contains such a high percentage of water that it cannot be fortified economically as can the bi- and tri-spent acids. It is necessary, therefore, for economic reasons to recover H_2SO_4 & HNO_3 by distillation

Mono-waste acid is pumped from the storage tank thru an oil separator, then thru a preheater,

and introduced into the top of the denitrating tower. Here it meets in its descending fall a countercurrent steam jet which is fed into the bottom of the tower. Intimate contact between hot steam vapor & the acid mixt effectively disintegrates the nitric compds, and releases oxides of N into the steam vapor stream. Denitrated H_2SO_4 (DSA) flows from the bottom of the denitrating tower into Pb lined cooling tubes. After cooling, the DSA is pumped to a storage tank. DSA contains 65-68% H_2SO_4. When needed the DSA is transported to the Acid Manufg Area as a raw material for the production of 95% H_2SO_4 (Ref 1a)

Refs: 1a) B.T. Fedoroff, "The Manufacture of TNT", Picatinny Arsenal Lecture, Tech Div, Dover, NJ (7 Feb 1947), 17 1b) F.H. Reichel & R.T.K. Cornwell, USP 2421391 (1947) & CA **41**, 5306 (1947) 2) G. Williams & R.J.J. Simkins, JChemSoc **1953**, 1388-89 & CA 8001 (1954) 3) J.R. Brown & L.D. Hayward, CanJChem **33**, 1735-45 (1955) & CA **50**, 11946-47 (1956) 4) G. P. Koptev et al, RusP 137900 (Appl 9 March, 1960) & CA **56**, 3119 (1962) 5) V.G. Ponomarenko, Koks i Khim **1961**, No 11, 54-57 & CA **56**, 6897 **(1962)** 6) A.R. Baranova & N.M. Antonova, Rus P 149405 (1962) & CA **58**, 5288 (1963)

Denitration Test (L'epreuve de dénitration, in Fr) The test consists of heating an organic nitric ester (such as NG, NGc, etc) or a nitrocompound (such as NBz, DNT, TNT, etc) at a desired temp and determining periodically the nitrogen content

For example 1,3-Butanediol Dinitrate, contg 15.31% N, heated for 2 days at 60° had 15.29% N and after 4 days 15.25% N; and Nitroisobutane Trinitrate, originally contg 19.50% N, decreased to 19.43% N after 2 days heating at 60°, and to 19.43% N after 4 days heating at 60°

Ref: J. Tranchant, MP **32**, 316-17 (1950) & CA **47**, 9014 (1953)

Dense Propellant Ignition Composition. An expl train for elec detonators & blasting caps is described having a Cyclonite base chge, a LA initiating chge and an ignition chge. The ignition chge consists of loose, dry MF (40-90% thru 200-mesh sieve) 30-50% and ground dense proplnt (100% thru 60-mesh sieve), prepd from NC (12.0 - 12.7% N) 93.8 max, DPhA 0.3-1.2 & graphite 5.0 max, 70-50%. Requirements of the proplnt compn are: ash 3.0 max, moisture & volatiles 2.0 max & ether extractable compds 2.75% max

The firing times for caps charged with various mixts of the ignition chge are as follows:

Composition, %	I	II	III
MF	50	40	30
Propellant	50	60	70
Firing Time, millisec			
Average	3.6	3.9	5.3
Total	10.9	13.2	18.0

See also Blasting Cap Compositions, Vol 2, pp B189-Lff

Ref: T.J. Mulqueeny & F.R. Seavey, USP 2825, 639 (1958) & CA **52**, 7704 (1958)

Densimeters. Devices for measuring densities. See under DENSITY & SPECIFIC GRAVITY AND THEIR DETERMINATIONS

Densité. Fr. for Density

Densites. Belgian coal mining permissible expls on the list of "Explosifs SGP":

Composition, %	I	II	III
AN	49.80	81.10	82.34
Sr Nitrate	33.70	10.40	11.82
TNT	16.50	8.50	5.84

They were patented in 1894 and manufd by Ghinijonet at Ougrée, Belgium (Refs 1 & 2)

Marshall (Ref 3) lists: a) Densite 3 AN 74, Na nitrate 22 & TNT 4% and b) Densite 4 - AN 18, K nitrate 45.5, TNT 19 & Amm chloride 17.5%. The chge limits are 700g & 850g and Dynamite No 1 equiv wts 310 g & 549g respectively

Refs: 1) Daniel (1902), 188 2) Gody (1907), 701 3) Marshall **1** (1917), 390 4) Marshall Dict (1920), 28-29

DENSITY & SPECIFIC GRAVITY AND THEIR DETERMINATIONS (Densité in Fr; Dichte in Ger; Densità in Ital; Densidad in Span & Plotnost' in Rus). *Density* (abbr d) is the mass per unit volume of a substance at a defined temperature. This physical property might serve for identification of various materials (including expls & proplnts)

and is required in computing volumes of materials from known weights. *Specific Gravity* (abbr sp gr) is the ratio of density of a substance to the density of another substance chosen as a standard at defined temperatures. The usual std is water

Densities may be subdivided into the following types:

A. *Absolute Density,* also called *Ordinary or Intrinsic Density* or *True Specific Gravity* (abbr here as abs d). (Densité absolue ou réelle in Fr). It is the mass per unit volume (M/V) of a substance universally expressed, for solids and liquids, in grams per cubic centimeters (g/cm^3 or g/cc). In US industry abs d is usually expressed either in pounds per cubic feet or in pounds per gallon. The abs d is mainly a function of temperature and only slightly of pressure, while for gases it depends largely on pressure and to a smaller degree on temp. The abs d of a gas is usually referred to standard temperature (0° C) and pressure (760mm), abbr as STP. The abs d of a solid or liquid can be detd only for substances contg no impermeable voids and if all the air is removed from their permeable voids. The abs d of crystals is known as *Crystal Density*

Absolute Specific Gravity (Abs sp gr, sp gr, d^t_t or S) can be defined as the ratio of true weight of a substance (wt corrected to air buoyancy) to true wt of water contained in the same volume at the same temperature. For gases, the term sp gr refers to dry air at the same pressure & temp. In US industries, the term "specific gravity", without the prefix "absolute" may refer to wts not corrected to the effects of air buoyancy (apparent wts). The sp grs using the uncorrected wts are known in science as *Apparent Specific Gravities* and they do not differ more than 0.1% from the sbsolute values (See also Ref 2, p 411 & Ref 13, p 876

B. *Apparent Density and Apparent Specific Gravity.* Apparent density is the weight of a unit volume of a porous solid including any air which may be entrapped in the pores or interstices (impermeable voids) but not between the grains (permeable voids). This value would be identical with abs d of a material contg no voids (permeable or impermeable), but otherwise it is smaller than the abs d

Note 1: The MIL-STD-650 (Ref 21, Methods 201.1, 201.2 & 201.3) does not make any distinction betn Apparent and Bulk Densities (See item C)

Note 2: Accdg to Kirk & Othmer (Ref 13, p 877) the term *Apparent Specific Gravity* is used in technology to denote the sp gr of porous solids when the volume of the solid is considered to exclude the permeable voids

Note 3: The apparent densities of expls & proplnts range between 1.0 & 1.9, more commonly betn 1.5 & 1.7. These d's are always lower than absolute d's, especially for substances which are not cast or highly compressed because they contain intrinsic voids (pores). For example, the apparent d of BkPdr varies betw 1.65 & 1.85 while its absolute d is 2.03

C. *Bulk Density and Bulk Specific Gravity.* The bulk d is the wt of a unit volume, (at a defined temperature), occuppied by pieces (grains) of a substance including all the voides inside the grains (impermeable) and outside of them (permeable). It is usually lower than the apparent d. The *Bulk Specific Gravity* is the ratio of bulk density of a substance, to the density of water at defined temperatures (Ref 13, p 877)

Note 1: As was mentioned in Note 1 under item B the US MIL-STD-650 (Ref 21) does not make any distinction betw Bulk & Apparent Densities. It defines them in Method 201.1 as "weight per unit of outside volume, which may include voids"; in Method 201.2 as "weight per unit of apparent volume"; and in Method 201.3 as "weight per unit of packed volume", which means that the specimen is dropped and tapped to dispel voids

Note 2: Accdg to Ref 13, p 877, "It is not always possible to say whether a given measurement refers to bulk specific gravity, to apparent specific gravity, or to some intermediate value, since the determination of solid volume depends on the nature of the substance used to permeate the voids and on the nature of the pores in the solid"

Note 3: It seems that the French term "Densité de tassement" corresponds to Amer Packed Density because in both cases the expls are packed. The term must not be confused with the Fr term "Densité gravimétrique (See Gravimetric Density, item G)

D. *Cast Density.* It is the density of a material in the solidified state. See also item E

E. *Charge (or Loading) Density* (Densité de chargement in Fr). This term is applied to densities of expls, proplnts or pyrotechnics loaded

in items of ammunition, such as shells, bombs, grenades, cartridges, proplnt bags, mines, torpedoes, flares, etc. If an expl is loaded by melting, pouring into an item and allowing to solidify, it is known as "cast-loading" and the density as *cast density*. Examples: TNT, Composition B, Pentolite or Tetrytol cast in shells, bombs, etc. This d is usually slightly lower than abs d (See item A) and higher than apparent or bulk d's (See items B & C). If an expl is loaded by pressing (press-loaded), its d is higher than bulk d and slightly lower than apparent d. Examples: Composition A, Amatol 80/20 loaded in shells, bombs, etc. If a material is loaded losely, as is smokeless proplnts and powdery dynamites, its loading d is equal to the bulk d. The loose pyrotechnic items are usually loaded by packing (Ref 6, pp 41 & 99; Ref 7, p 50; Ref 9, p 267; Ref 12, p 176 & Ref 20, Method 510.1.1)

In most cases of loading ammunition, the higher the absolute density of an explosive, the higher is its loading density (See also Limiting Density and Maximum Loading Density)

F. *Crystal Density*. It is the absolute dof a crystalline substance. No voids, inside or outside, should be present

G. *Gravimetric Density* (Densité gravimétrique, in Fr). Accdg to Snelling & Storm (Ref 1, p 68) "gravimetric density" or "apparent specific gravity" of an explosive is the ratio of the weight of the powder contained in a given volume to the weight of water that would exactly fill the same volume. This density is not only a factor of the true (absolute) density, but is also influenced by the size and shape of the grains. Its detn for BkPdrs is described (See also below under Density and Specific Gravity Determinations of Solids). Accd to Marshall (Ref 2, p 414), it is the weight of a charge in a firearm divided by the wt of water that would be required to fill the chamber space. As applied to gunpowders, it is the wt that will fill a unit volume of a vessel when simply poured in and very gently shaken down. Accdg to Vennin et al (Ref 5, p 254), the "densité gravimétrique ou apparente" is the wt in kg of 1 liter of powder or propellant under the conditions of filling. It increases with the increase of "densité réelle" (see Absolute Density, item A) and varies with the size and form of the grains. It is the "Densité gravimétrique" which governs the conditions of loading the ammunition, especially for rifle powders. For detn of this density, the apparatus known as *gravimètre* (gravitometer) was used in France (See below under Density and Specific Gravity Determination of Solids, item k)

Accdg to Ref 2, pp 414-15, the difference betw the gravimetric and absolute densities shows that there is almost as much free air space betw the grains as is occupied by the grains themselves. There is no necessary connection betw the gravimetric and absolute densities. For example, the French BkPdrs of abs d 1.80 had gravimetric d's betw 0.940 & 0.970 and the Ger rifle powder M/71 of abs d 1.655 had gravimetric d of 0.915. Snelling found a number of Amer Blasting powders to have gravimetric d's from 1.09 to 1.20 (Ref 2, p 415)

H. *Limiting Density* (Densité Limite, in Fr). Dautriche et al (Addnl Ref C) observed that for certain expl mixts, such as Cheddites (See Vol 2, pp C157 & C159), Explosif P ($KClO_3$ 90 & paraffin 10%), Explosif S($NaClO_3$ 89 & paraffin 11%), Minélites (See Ref 9, p 361), etc, the detonation rate increases with increasing loading density to a certain maximum value, which is characteristic for each expl mixt. This value is called by Davis (Ref 9, p 361) the *optimum density*. Further increase of d causes the diminution of the deton rate, as well as of the sensitivity to initiation and a point could finally be reached at which no detonation is produced. The density at which the expl ceases to detonate was named in France *densité limite*

A table in Ref 9, pp 362-63, gives optimum and limiting densities for several expl mixts. For example, when "Explosif S" was exploded in a Cu tube 20-22 mm diam, the highest deton rate was 2960m/sec at d 1.33,at d 1.54 the rate dropped to 2688 and at d 1.56 there were failures (See also Ref 5, p 62)

Muraour (Addnl Ref D, p 895), who also investigated this problem, stated that the "densité limite" exists only for expl mixts and not for definite chem compds, such as PA, TNT, Tetryl, etc. He also stated that "densité limite" must not be confused with the density at which an expl, such as MF, is "dead-pressed"

I. *Loading Density*. See Charge Density

J. *Maximum Loading Density* (Densité maxima de chargement in Fr). This may be defined as the highest density obtainable, either by compression or casting, in the loading of expls in ammunition.

Pepin Lehalleur (Ref 5, p 54), gives maximum loading densities for the following expls: TNT 1.58, PA 1.69, Blasting Gelatin (92/8) 1.63, PETN 1.62 & RDX 1.70 (See also Optimum Density under Limiting Density)

K. *Optimum Loading Density.* See under Limiting Density

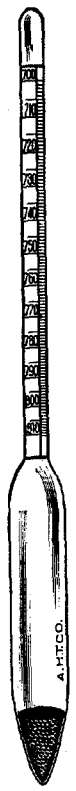
L. *Packed Density.* Accdg to MIL-STD-1233, Method 400, the packed d of a powdered material is the weight per unit volume of material which has been packed until it has attained the most compact form. Packed d indicates the loading d of loose pyrotechnic powders (See also item E)

M. *Relative Density* (d_4^t). It is the ratio of the absolute d at t°C to abs d of water at 3.98°C (0.999973 g/cu cm, its maximum d) and is expressed as g/ml. It is also called "density (or specific gravity) relative to water at 4°C"

The difference betw d_4^t and d_t^t is negligible and since 1cc = 0.999973 ml, the d_t^t = 0.999973 d_4^t. Most data found in the scientific literature are expressed as d_4^t and d_t^t; the relationship betw them is: $d_4^t = d_t^t \times d_4^t(H_2O)$. The values of the relative d of w, $d_4^t(H_2O)$, changes by less than 1% in the temp range 0-40° and about 5% in the range 0-100°, so that in most cases the difference betw abs sp gr and relative d is not great

Relation betw US industrial measurements and metric units are: Pounds/US gallon = 8.3452 g/ml and Pounds/cu ft = 62.427g/ml (Ref 13, p 876)

N. *Shaking Density.* It is a density of an explosive submitted in bulk after it undergoes shaking as prescribed by the BurMines Test (Ref 4, p 24). See under Density and Specific Gravity Determinations of Solids, item w)



Arthur H. Thomas Hydrometer for Liquids Lighter than Water

Density and Specific Gravity Determinations of Liquids

a) Hydrometer Method. It is used extensively in science and industry on account of its simplicity and rapidity, although the accuracy is no better than about 1%. It is based on the principle of Archimedes: the upward buoyant force exerted on a body immersed in a liquid is equal to the wt of the displaced liquid

A hydrometer is a hollow, tubular shaped vessel, provided at the bottom with a bulk contg some lead shot and at the top with a long thin tube contg a scale (See Fig). The scale may read either in specific gravity units or in degrees Baumé, Twaddell, etc. Unless otherwise marked on the stem, the scale of US hydrometers applies to a temp of 60°F (15.6°C) (Ref 13, p 886; Ref 23, Method D1298; Adnl Refs L & M).

Each laboratory at explosives plant should be provided with at least one "precision" hydrometer calibrated by the National Bureau of Standards in Washington. As these hydrometers are very expensive, it is advisable to use them only for calibration of other laboratory and plant hydrometers, which are much cheaper. A simple method of calibration, such as used during WWII, at Keystone Ordn Works is described in Ref 8. In this method the following liquids were used: kerosene for calibration of hydrometers, range 0.700 to 0.800; iso-butanol for 0.800 to 0.900; boiled distilled water for 0.900 to 1.000; 16% (by weight) soln of H_2SO_4 in w for 1.000 to 1.200; 41% H_2SO_4 soln for 1.200 to 1.400; 61% H_2SO_4 for

1.400 to 1.6200; and 95% H_2SO_4 for 1.800 to 2.000

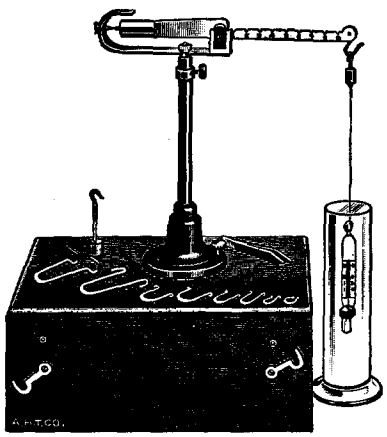
Calibration. Place the NatlBurStds hydrometer and the ones to be calibrate d in a tall beaker contg fresh cleaning soln ($K_2Cr_2O_7$ in H_2SO_4) and leave there for about 1 hour. Remove the hydrometers, rinse under tap water and place them in a tall beaker contg distd w maintained at 15.6°. Fill to about 3/4 a thoroughly cleaned and dried glass cylinder, ca 30cm long and 4cm diam, with one of the above liquids and insert a thermometer calibrated at the BurStds. Place the cylinder into a water bath maintained at 15.6° and wait until the temp of liquid in the cylinder reaches 15.6°. Remove the BurStds hydrometer from the beaker with distd w, wipe it off quickly with tissue paper and immerse slowly into liquid in the cylinder, without touching its walls and without touching the scale of hydrometer above the meniscus. Recheck the temp of liquid and, if it is still 15.6°, take the reading of hydrometer. For this, place your eye on the level with the surface of the liquid and observe its intersection with the scale. Remove the std hydrometer and replace it, in exactly the same manner, as above, with each of the hydrometers to be calibrated. Compare the reading of Std hydrometer with others and if they are not identical mark the correction on stem of each hydrometer

Procedure: For detn of sp gr of a liquid to test, such as benzene, toluene, nitric acid, sulfuric acid etc, select a hydrometer of corresponding range and proceed as above. For opaque liquids such as petroleum, readings must be taken at the top of meniscus, using a hydrometer calibrated to read in the same manner

b) Hydrostatic Weighing Method. It is also based on the buoyancy principle and consists in weighing a sinker of known volume, V, first in air (W_A) and then totally submerged in liquid to test (W_L). The apparent loss in weight, $W_A - W_L$, is equal to the mass of displaced liquid; therefore the density is:

$$d = (W_A - W_L) / V$$

Various types of balances may be employed with this method, such as ordinary analytical balance, chainomatic balance and Westphal balance. The most rapid of these is the *Westphal Balance* or its modifications. Its accuracy is 0.1 to 0.05% (Ref 13, p 886). The balance is listed

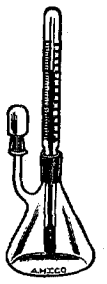


Westphal Specific Gravity Balance

in AHThCo Catalog as No 21500 (Addnl Ref L) or in Fisher Catalog as No 2-150 (Addnl Ref M) is provided on the left arm of the beam with a counter-weight and a pointer, while the right arm has 9 equally spaced notches and a plummet (with inserted thermometer), suspended from a hook at its end. The balance with no weights (riders) placed on notches, but with plummet freely swinging in the air, is set up on a horizontal surface and adjusted (by means of levelling screw) so that the two pointers meet. Place the cylinder (ca 150mm long & 25 mm in diameter), contg distd w, freshly boiled and cooled to ca 10°, under the plummet, so that it is completely immersed but not touching the walls. Place the unity wt on the beam and warm the cylinder by holding it in hand until the temp of w reaches 15.6°. Quickly readjust the wt, if necessary, so that the two pointers meet and take the reading. If the apparatus was calibrated properly by the manufacturer the reading for water should be 1.000

For detn of sp gr of a liquid such as toluene, benzene, acids, etc, fill the dry cylinder with a liquid to test cooled to ca 10° and place the rider to the approximate expected sp gr and when the temp reaches 15.6°, readjust the riders to balance the beam (Ref 8, Vol 1, Chap IV, p 7; Ref 10a, p 48; Ref 15a, p 746-R; Ref 20, Method 510.2.1

c) Pycnometer Method. A device most widely used consists of a small flask provided with a glass stopper with attached calibrated thermometer and a capillary side arm provided with a cap (See Fig). If a pycnometer is intended for



Pycnometer Specific Gravity Bottle

precise work (such as for measuring sp gr of toluene used in manuf of TNT), it should be calibrated in a laboratory and this can be done by the following method described in Ref 8, Vol 3, Chap II, pp 23-24:

Calibration: Clean both the pycnometer and stopper thoroughly with hot chromic-sulfuric acid soln, followed by rinsing with w, drying in an oven and cooling in a desiccator. Place pyc on the left pan of analytical balance and tare to the nearest 0.1 mg. Remove from the balance and fit over the capillary of side arm a piece of clean small-bore rubber tubing, ca 2.5" long. Fill the pyc thru the central opening with distd w, freshly boiled and cooled to room temp. Insert the stopper with thermometer firmly in place, taking care not to introduce air bubbles inside the pyc. Some w will be forced outside thru the side arm. Immerse the pyc up to ca 1" below the top of rubber tubing in a const temp both maintained at room temp and leave it there for 10 mins. During this time calc the appr wt of w in the pyc, by multiplying the vol, marked on pyc, by the density at t° of the bath. Put this wt together with the tare of pyc on the left balance pan. Raise the pyc slightly so that the upper part of the capillary is just out of the w. Disconnect the rubber tubing, quickly remove with a dry finger the excess of w on top of capillary, and wipe its ground portion with a piece of filter paper. Replace the cap quickly and, while holding the pyc by its capillary, remove pyc from the bath, and wipe it dry with a piece of cloth, followed by tissue paper. Place pyc on the left pan of the balance and weigh to the nearest 0.1 mg

The wt of w in pyc at 15.6° (60°F) is obtd from the following formula:

$$W_{15.6} = W_t \times \frac{d_{15.6}}{d_t} \times \frac{1}{[1 + a\,(t - 15.6)]} \text{ grams}$$

where: $W_{15.6}$ = Wt of w in pyc at 15.6°
W_t = Wt of w in pyc at t° (Temp of detn)
$d_{15.6}$ = Density of w at 15.6° (0.9990343)
d_t = Density of w at t° (Lange's or other handbook)
a = Coeff of thermal expansion of glass (0.000025 per 1° C)

Run a check and if duplicate wts agree within 0.005%, the calibration is satisfactory

Determination of Specific Gravity of a liquid, such as toluene, may be conducted as described in Ref 8, Vol 1, Chap IV, pp 7-8. For this fill the pyc, calibrated as described above, with toluene cooled to ca 10° and insert the stopper with thermometer leaving the capillary of side arm open. Allow pyc to stand at room temp, wiping occasionally the condensation with tissue paper. When the thermometer will read 15.6° remove with dry finger the excess of liquid from side arm and place on it the overflow cap. Weigh to the nearest 0.1 mg and calc as follows:

Sp gr of liq at $15.6^{\circ}/15.6^{\circ} = (W_3 - W_1)/(W_2 - W_1)$ g/ml,

where: W_1 = Wt of empty pycnometer
W_2 = Wt of pyc with water
W_3 = Wt of pyc with liquid to test

The above described method is rapid, but it is not, strictly speaking, very precise because no buoyancy correction was introduced. For example, one of the toluenes examined during WWII at Keystone OW, Meadville, Pa gave sp gr of 0.87070 when detd by this method, but when weighing was done by precision (*European*) method and the buoyancy correction introduced, the sp gr was 0.87086 giving the difference of 0.00016

The European method of weighing, described in Ref 8, Vol 1, Chap II, pp 7-8, has the advantage because it eliminates the error due to inequality in lengths of left and right sides of balance beams. The only requirement with European method is to have a precision set of weights, preferrably platinum plated, but the balance does not need to be of great precision. The weighing is conducted as follows: Adjust the balance to zero with both

pans empty. Place on each pan a 5(or 10g) brass weight, recently calibrated,preferrably by the Natl BurStds, and note the displacement of zero. Interchange the weights and if displacement of zero is the same, the weights are correct. Suppose now that we place the object on the left pan and weigh it by the method used in US and then place it on the right pan and the weights on the left. In most cases (except with expensive precision balances) the weights will not be the same and the difference is caused by the inequality in lengths of the left and right beams

Let us now determine sp gr of liquid (such as toluene), using precision (European) method of weighing and introducing a buoyancy correction, such as (described in Ref 8, Chap IV, pp 8-9

Procedure: Place on the left pan of the balance a calibrated 100 g brass weight and on the right pan the empty pycnometer, cleaned and dried as described above. Place on the right pan the calibrated brass weights until the pointer of the balance comes to zero. Suppose this gives the wt of 52.7879 g and the wt of pyc in air would be: 100.0000 - 52.7879 = 47.2121g. Its wt in vacuo can be calcd from the equation:

$$W_v = W_a + W_a \times d_a \left(\frac{1}{d_s} - \frac{1}{d_b}\right),$$

where: W_v = Wt of pyc in vacuo (true weight)
W_a = Wt of pyc in air (apparent weight)
d_a = Density of air (0.0012)
d_s = Density of substance (glass 2.5; brass 8.4; toluene 8.7)
d_b = Density of brass weights (8.4)

In order to make calcns of W_v easier for future use, determine the value of the expression: $d_a\left(\frac{1}{d_s} - \frac{1}{d_b}\right)$. It is for water - 0.001057, glass -0.0003372 and toluene -0.001237

If W_a for glass pyc is 47.2121, The W_v is equal to 47.2121 + (47.2121 x 0.0003372) = 47.2280g

Fill pyc with water and weigh it as above:
100 g = Pyc with w + 1.7673g

This gives the wt of w at 15.6° in air as: 100 - (47.2280 + 1.7673) = 51.0047 and its wt in vacuo is calcd as: 51.0047 + (51.0047 x 0.001057) = 51.0587g

Fill pyc with toluene and weight it as above:
100g = Pyc with toluene + 8.3620

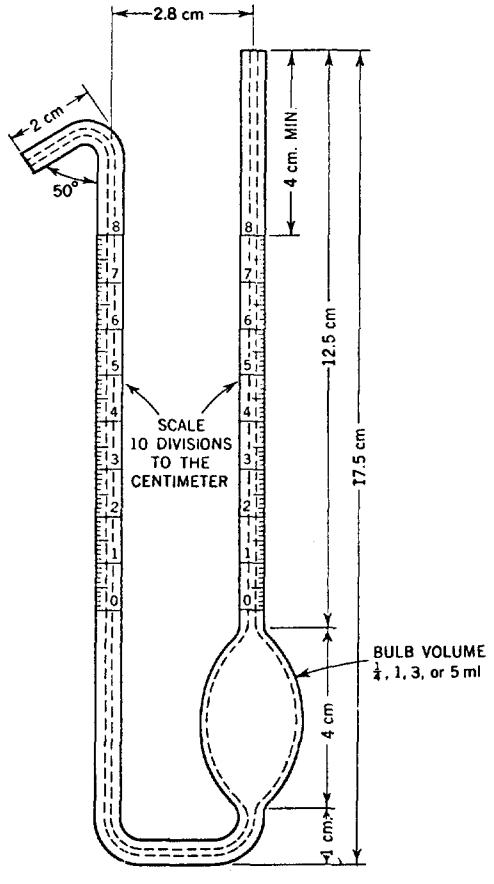
This gives the wt of toluene at 15.6° in air as: 100 - (47.2280 + 8.3680) = 44.4100 and its wt in vacuo is calcd as: 44.4100 + (44.4100 x 0.001237) = 44.4669g

Apparent sp gr of toluene at 15.6°/15.6° = 44.4100/51.0047 = 0.87070 g/ml

True or absolute sp gr of toluene at 15.6°/15.6° = 44.4649/51.0587 = 0.87086 g/ml

Specific gravity of liquids may also be detd at other temps, such as 4° (temp of maximum density of w = 1.0000), 20°, 25°, etc (See also Ref 23, Method D1217)

d) Lipkin Bicapillary Pycnometer. This device, briefly described in Ref 13, p 888 is widely used in industry. It is adopted by the ASTM (See Ref 23, Method D 941)



Lipkin Pycnometer

This method is intended for the measurement of the density of any liq at the specified temp of 20 & 25°. Its application is restricted to liqs having vapor pressures less than 600 mm of Hg and having viscosities less than 15 centistokes at 20°

Procedure. The liq sample is drawn into the pyc

and weighed. It is then equilibrated at the test temp, and the positions of the liq levels are observed. The d or sp gr of the sample is then calc d from its wt, a calibration factor proportioned to an equal vol of w, and a term which corrects for the buoyancy of air
Calculate d in gm/ml as follows:

$$d(\text{at } 20 \text{ or } 25^{\circ}) = \frac{W}{V} + C$$

where W = wt in grams in air of sample contd in pyc
V = apparent vol in ml corresponding to sum of scale on the two arms
C = air buoyance correction, obtd from a table

Density and Specific Gravity Determinations of Gases

Accdg to Kirk & Othmer (Ref 13, p 890), the direct detn of gas densities, by weighing a bulb before and after filling with the gas at a known pressure, is not frequently used because of the tedious operations and sources of error. Instead of this the *Gas Density Balances* employing the buoyancy principle are in common use. As an example of such balances is the *Edwards Portable Balance* developed at the National Bureau of Standards and the *ArcoBalance.* There are also other methods of measurement (Ref 13, pp 890-92 & Ref 23, Method D1070)

Density and Specific Gravity Determinations of Solids

a) *Suspension or Buoyancy Method.* The procedure consists of adding the lumps of the test solids to liquids of various densities until the one is found in which the solid remains suspended. It must not dissolve in the liquid. The density is equal to the density of liquid (Ref 10, p 49 & Ref 13, p 893)

b) *Balance Method.* For fairly accurate detns, suspend a lump of the solid by a fine non-corroding wire from the right beam of an analytical balance and weigh in air to the nearest 0.1mg. Install a small platform (bridge) over, but not touching, the right pan and place on the platform a beaker with liquid in which the sample is insol. The amt of liq should be sufficient to immerse the whole of the solid hanging by the wire and the temp should be recorded. Remove air bubbles clinging to the surface of solid by means of a small camel's-hair brush and weigh to the nearest 0.1mg. The difference betw the wt of sample in air and of that suspended in liq is equal to the wt of liq displaced by sample. On dividing this wt by sp gr of liquid at the temp of detn, the vol of solid is obtd. The density of sample is obtd by dividing its wt in air in grams by its vol in ml. If w is used as a medium and detn is made at 4° (at which d of w is 1.0000), the density of solid is calcd by dividing the wt of solid in air by the difference in weights of sample in air and when immersed in w (Ref 10, pp 48-49)

The procedure is more rapid if the *Westphal Balance* is used. This device is described as item b, under "Density and Specific Gravity Determinations of Liquid". For detn of sp gr of solids, the plummet is replaced by the sample (in the form of a lump), suspended by a non-corrosive wire

The hydrostatic balance principal has been used in *Bode's Densimeter* for density detns of large grain expls or proplnts, such as of prismatic BkPdr. Here the prism is weighed first in air and then suspended in mercury at known temp

Density = $(W \times 13.595)/[W + W_1(1+0.00018t)]$ gms/ml
where: W=Wt of sample in air
W_1=Wt to add in order to immerse the sample completely in mercury
13.595=Density of Hg
0.00018=Coeff of expansion of Hg
(Ref 2, p 413 & Ref 6, p 100)

c) *Pycnometer Method.* The US military procedure described in Ref 20 as Method 510.1.1, for detg sp gr of smokeless proplnts contg no water sol ingredients, is as follows:

Fill a clean 50ml pycnometer, similar to the one described as item c under "Determination of Densities and Specific Gravities of Liquids", with distd w, freshly boiled and cooled to 10° and insert the stopper provided with a thermometer. Take care not to introduce air bubbles and to have the capillary of side arm filled with water flush with the top. Allow to stand while holding the pyc in the hand to speed up warming to 15.6°. As soon as this temp is reached, remove with dry finger excess of w from the side arm and replace

the cap. Wipe off the moisture from outside of pyc and weigh the assembly to the nearest mg. Repeat the opn until two weighings agree to the nearest 3mg. Empty the pyc, introduce a 10g sample of proplnt (weighed to the nearest mg) and add hot (preferrably boiling) distd w until the pyc is about 3/4 full. Connect the neck by means of a thick wall rubber tubing to a source of vacuum (ca 20mm Hg) and immerse the pyc to 3/4 in hot water bath. With vacuum applied, close intermittently with a finger the top of side arm and continue until all air is removed from the proplnt grain perforations. Disconnect the tubing, add distd w to the top of pyc and cool to ca 10° in a cold w bath. Remove from the bath and insert the stopper with thermometer, as described above. Wipe off the w and hold the pyc in the hand until the temp reaches 15.6°. Remove the excess of w from the top of side arm and replace the cap. Wipe off the moisture from outside of pyc and weigh to the nearest mg

Sp gr of proplnt at 15.6°/15.6°= AF/(A+B-D) g/ml, where: A=Wt of sample in g
B=Wt of pyc with w
D=Wt of pyc with w & sample
F=Sp gr of w at 15.6° (0.9990343)

Note 1: If the sample to test is porous and it is desired to obtain its absolute d, it should be finely pulverized before placing it in the pycnometer

Note 2: If the sample to test contains water-sol ingredients, the w should be replaced with a liquid (of known sp gr), in which the sample is insol

Note 3: The above described method is not as accurate as the procedure described as item c, under "Density and Specific Gravity Determinations for Liquids", but it seems that this "precision" is sufficient in testing proplnts

d) *Density in Vacuo Method.* The US military procedures, described in Ref 21 as Method 202.2, for detg density of expls and proplnts in pellet form is as follows:

Weigh on an analytical balance without dampers a piece of Pyrex glass of appr the same volume and shape as the pellet to test. Place this glass, which will serve as a "standard", in a 250ml beaker contg a 0.01% aerosol soln satd with expl material to test and cooled to 71°F (21.11°). After the glass has become thoroughly wet, remove any adhering air bubbles by means of a camel's hair brush. Place another 250ml beaker on the platform (bridge) installed over, but not touching, the left balance pan and suspend a wire loop from the hook above that pan. Fill the beaker, cooled to 70°F, with 0.01% aq aerosol soln satd with material to test until the loop of the wire is completely immersed. Attach a tare wt to the right beam hook and adjust balance to zero. Install the "standard" in the loop and completely immerse it in soln. Weigh and calc the apparent density (d_{ag}) of glass standard as follows:

$$d_{ag} = Ag/(Ag-Bg)$$

where: Ag=Wt of the glass standard in air
Bg=Wt of the glass standard in aerosol soln

The true density of glass (density in vacuo) (d_g) is calcd from the equation

$$d_g = \{1-0.0012\ (\frac{1}{d_1} - \frac{1}{8.4})\}$$

where: d_1=App d of glass multiplied by the d of w at 70°F (d of w =0.9980)
0.0012=Density of air
8.4=Density of brass weights

The correction factor (F_c) which will be applied to the apparent densities of samples is equal to d_g/d_{ag}

Determine the apparent d of sample (d_{as}) in the same manner as described for the glass standard and calc as follows:

$$d_{as}=A_s/(A_s - B_s),$$

where: A_s=Wt of sample in air
B_s=Wt of sample in aerosol soln

The density of sample in vacuo (d_s) is equal to $d_{as} \times F_c$ g/cc

e) *Bottle Method.* The US military procedure described in Ref 21, as Method 203.2, for detg sp gr of powdered expls is as follows:

Fill with distd w, freshly boiled and cooled to desired temp (such as 25°), 500ml Erlenmeyer flask, which is provided with a ground glass stopper grooved to allow excess of w to escape on closing the flask. Insert the stopper making sure that no air bubbles are left in the top of flask. Wipe dry the outside of flask and weigh it on a torsion balance of 1000g capacity & 0.1g sensitivity. Empty the flask and introduce into it ca 100g of explosive to test, previously weighed on a torsion balance of 200g capacity & 0.02g sensi-

tivity. Add ca 400ml of freshly distd w at the same temp as above and stir vigorously with a wooden rod to remove the air bubbles. Fill the bottle completely, stopper it, wipe off the outside and weigh as above

Sp gr of expl at °C/°C=AD/A+B-C)g/cc,

where: A=Wt of sample
B=Wt of flask with w
C=Wt of flask with w & expl
D=Sp gr of w at °C/°C

Note: If an expl contains water-sol ingredients, the w should be replaced by a liquid of known sp gr in which the expl is insol

f) *Volumenometer Method.* It consists in placing a known wt of solid in a vessel, such as a narrow graduated cylinder, partly filled with a liquid which is not a solvent for the solid. The rise in level of the liquid is a direct measure of the solid's volume. Mercury is often employed as the liquid for measuring *bulk density* by this method, since mercury does not penetrate any but large holes (Ref 13, p 892)

g) *Mercury Displacement Method.* Procedure used in US military installations for detg the sp gr of expl pellets, is conducted accdg to Ref 21, Method 203.1, as follows:

Place into a tared (to the nearest 0.1g) evaporating dish a 250ml beaker provided with ground top and fill it with previously purified mercury. Insert the "immersing fixture" (which consists of semi-hard rubber perforated plate pierced at each corner with sharp pointed pins so that about 0.5 inch of pin protrudes from each side) and level off the mercury by pressing down with a flat glass plate until it is even with the top of the beaker. Remove the mercury which spilled into the dish and place the beaker back into dish. Place an accurately weighed pellet of expl or proplnt beneath the prongs of the rubber plate and level the mercury as before. The pellet should be of sufficient size to allow easy immersion in mercury. It should not contain sharp corners and should not displace more than 200g of mercury (See Fig)

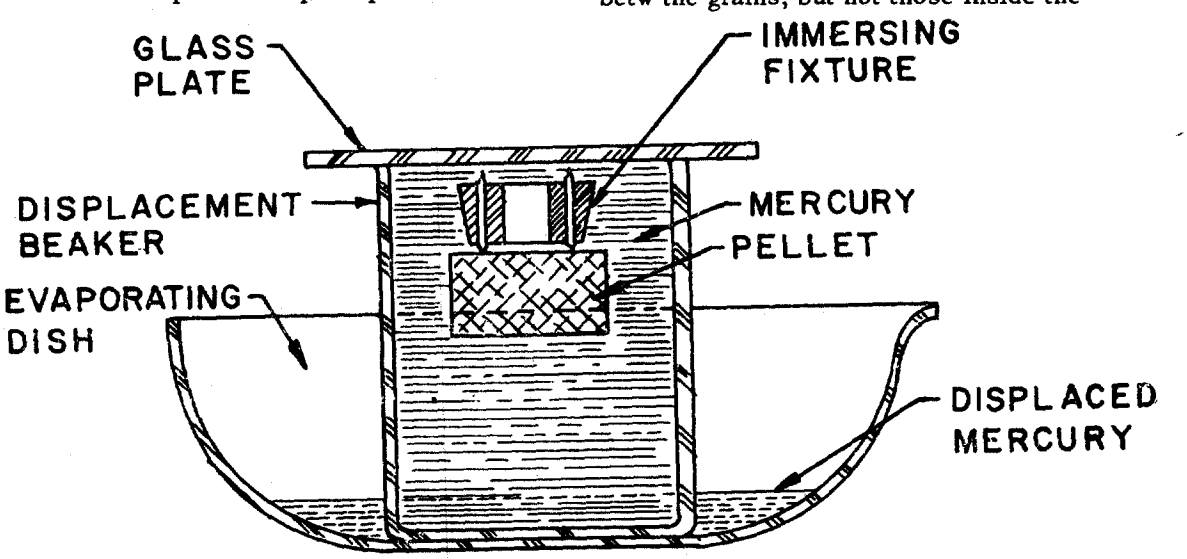
Weigh to the nearest 0.1g, the dish with mercury displaced by the pellet and calculate as follows:

Apparent sp gr of pellet = $13.54W/W_1$ g/cc

where W=Wt of sample in g
W_1=Wt of displaced mercury
13.54=Sp gr of mercury at room temp

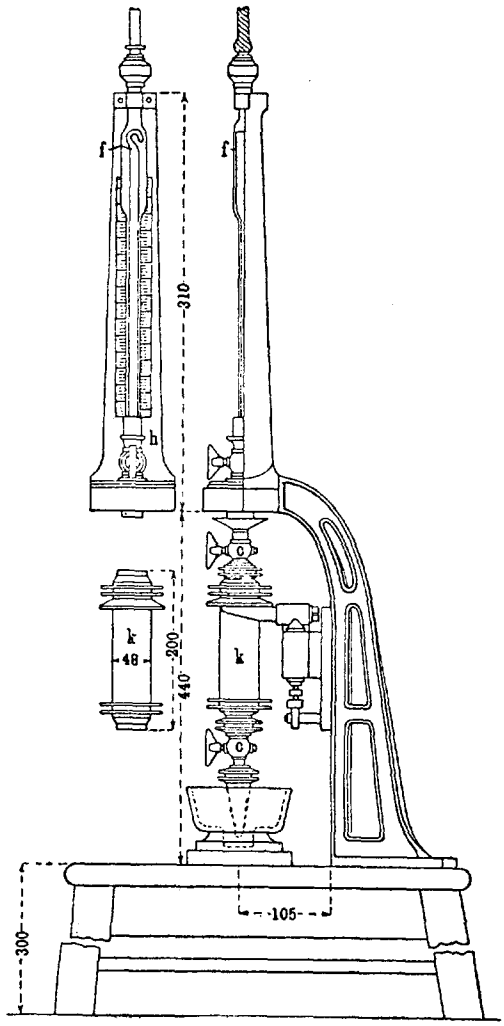
Note: As the US military terminology does not make any distinction betw apparent and bulk densities, the above procedure as well as the three US Military procedures, m, n & p, determine the same kind of density

h) *Bianchi's Densimeter.* The apparatus used extensively in Europe and Latin American countries, determines the displacement of mercury by a known wt of expl or proplnt (especially BkPdr), disregarding the air spaces betw the grains, but not those inside the



Density of Solids by Mercury Displacement Method

grains. The apparatus consists of a steel vessel k, which is screwed by means of a steel cock c to a vertical tube f, which is graduated. The bottom cock c of vessel k is provided with a tube drawn out to a point which dips into a cup with mercury. At either end inside of k there is a piece of iron wire gauze to prevent grains of powder from escaping (See Fig)



Bianchi's Densimeter

Procedure: Clean and dry k, connect its upper cock to f and plunge the tip of lower cock into cup with mercury. Connect the top of f to a vacuum pump and suck in the mercury of a known temperature, until it attains a steady level in f. Close the lower cock, break the vacuum and close the upper cock. Disconnect the upper cock from f, wipe k and weigh it. Run a check by repeating the previous opns. Empty k into cup, unscrew the upper cock and remove the upper wire gauze. Transfer into k an accurately weighed sample (6-7g), replace the gauze & cock and connect it to f. Fill k with mercury of known temp by suction in the same manner as described above and reweigh it

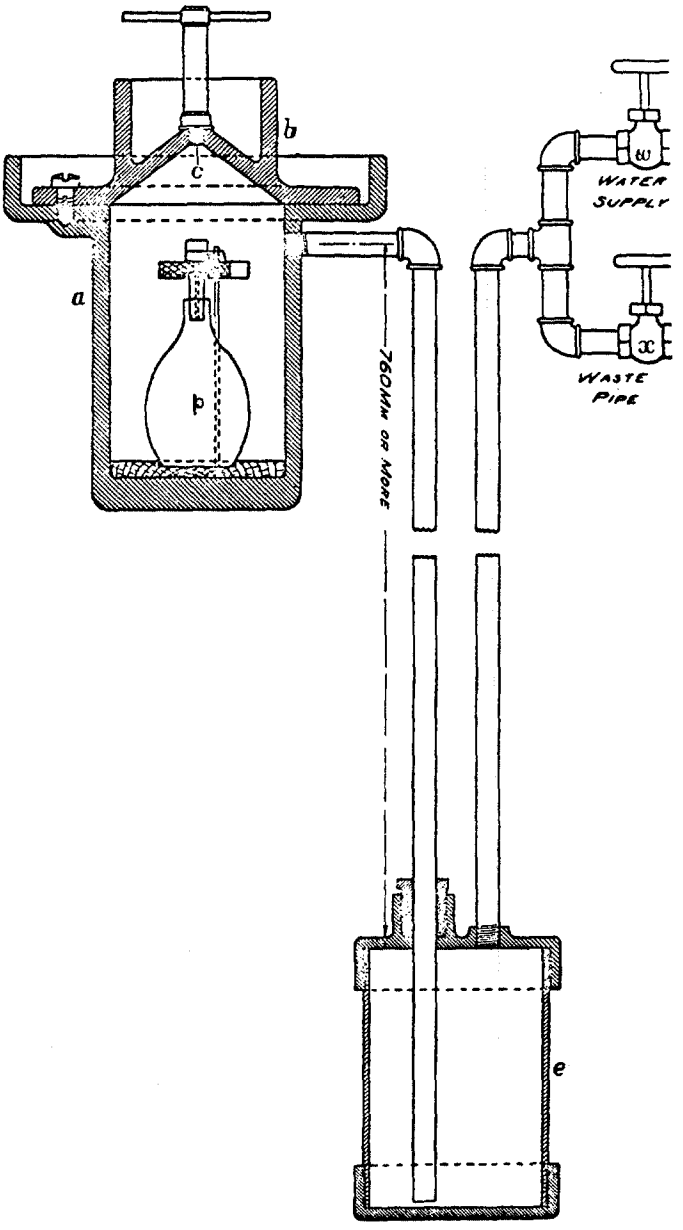
Density of sample=$(A\times 13.595)/[(A+W_1-W_2)(1+0.00018t)]$

where: A=Wt of sample in grams
W_1=Wt of k filled with Hg
W_2=Wt of k contg sample & Hg
t=Temp of mercury
13.595=Sp gr of Hg at 0°
0.00018=Coeff of expansion of Hg

(Ref 2, pp 412-13; Ref 6, p 100; Ref 11, p 179; and Ref 15, pp 16-17)

i) *Snelling's Densimeter.* The apparatus invented and used at the US Bur Mines, works somewhat on the same principle as the Bianchi but does not require a vacuum pump. It consists of vessel, e, connected by a pipe to reservoir, a, and a 2nd pipe which can be connected either to water supply or to waste pipe. Fill the pycnometer (p), with clean mercury, weigh it and then introduce in it, after emptying, an accurately weighed sample to test. Clamp down the pyc inside a, screw the lid on, open the valve, c, and the water stopcock w. The pressure of water entering the vessel, e, will force mercury to enter reservoir a and to chase the air thru valve c. As soon as mercury starts to come thru c, close c as well as the valve w. Open the waste-pipe x thus releasing the water pressure above mercury in e. As result of these opns part of mercury will flow back from a to e and a Torricellian vacuum will be created in the dome above the pycnometer. This will cause the removal of a large part of the air from pyc and its replacement with mercury. By repeating the above opns, all the air (except inside the grains) will be driven from the pyc thru valve c. Then the pyc is removed from a and weighed. This gives the wt of pyc with sample and mercury. As the wt of pyc filled with mercury, but without sample, is known, the density can be calcd by the equation used in Bianchi's method (See previous item)

(Ref 1, p 6 & Ref 2, pp 413-14)

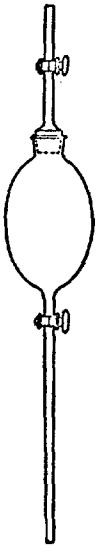


Snelling's Densimeter

Note: The densities of BkPdrs detd by Bianchi's and Snelling's methods are usually 1.65 to 1.85, but the "absolute" sp gr as calcd from its constituents runs higher, such as 2.03. This shows that under conditions of detns, many pores in the interior of the grains do not become filled with mercury. In detns of densities of commercial expls by Snelling's densimeter, the values were much higher than those obtained by the "sand method", described here as item v. For example the Amer 40% Dynamite has a d ca 1.6 when detd by Snelling's method and only 1.22 to 1.24 by "sand method" (Ref 2, p 414)

j) *Density of Black Powder by Bulb Method.* The procedure uses an elliptically shaped bulb of 50ml capacity as represented in Fig, which is taken from the "Ace" Catalog "40", No 5675 (See also Ref 8, Vol 1, Chap XVII, p 5)

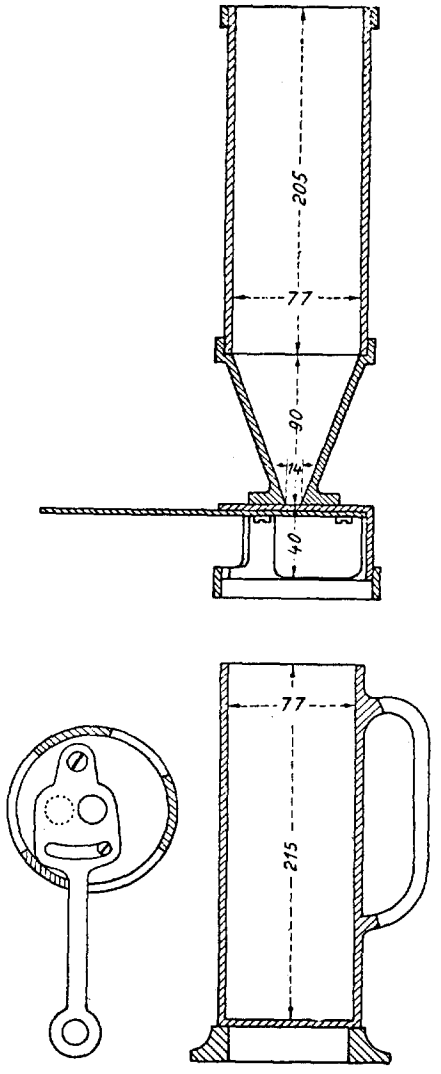
Grease all ground joints, assemble the apparatus and fill it with mercury. For this evacuate the bulb thru the upper stopcock, plunge the bottom tube into clean mercury of known temperature and open the lower cock, thus allowing the mercury to partly fill the bulb. Repeat these opns until the bulb will be filled as far as the upper cock. Then close both cocks, remove the mercury from tubes beyond the cock by means of pipe cleaners, wipe the bulb and weigh it. Empty the bulb, regrease ground glass joints. Close the lower cock and introduce thru the large opening at the top exactly 40g of BkPdr. Stopper the bulb, plunge the lower tube into mercury and, with both cocks open evacuate the bulb until it is filled with mercury as above. Close both stopcocks and reweigh it. In order to remove the air as completely as possible, a gentle tapping of the bulb is advisable during evacuation. Use for calcn the same formula as given in item f (Bianchi's method) (Ref 8, Vol 1, Chap XVII, pp 5-6; US Specification JAN-P-223A (12 Jan 1949)



ACE Company 50-ml Bulb

(See also under Black Powder, Analytical Procedures in Vol 2, p B177-R, Refs 10 & 11)

k) *Gravimetric Density Determination.* Accdg to Marshall (Ref 2, p 414), this density is the weight of substance that will fill a unit volume of a vessel when simply poured in and very gently shaken down. Accdg to Vennin et al (Ref 5, p 254) and Belgrano (Ref 15, pp 17-18), this density can be detd by the apparatus called in France *gravimetre* and it Italy *gravimetro* (See Fig). The apparatus consists of a brass or bronze cylindrical vessel which is connected to a funnel having an opening of 14mm ID. The opening can be closed by a sliding device as shown in Fig. Underneath the funnel is placed a brass or bronze cylindrical cup of 1 liter capacity. For detn of gravimetric d, close the opening of funnel, fill the vessel with sample to test and let it run freely into the cup until it overflows it. Remove the excess of sample by a straight edge and weigh the filled cup in grams. Subtract the wt of empty cup and divide the resulting value by 1000



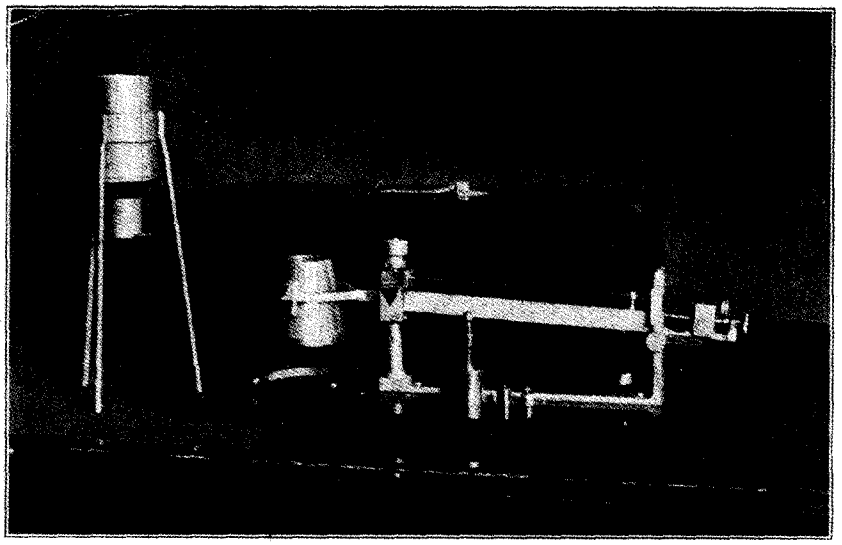
Italian Gravimetro

Note: The value obtained by this method is called by Belgrano "densità apparente o gravimetrica". In the US Military Method (see item l), which is quite similar to the above method, the same kind of density is called "bulk density"

l) *Gravimetric Density by Gravimetric Balance.* The balance used by the US Bur Mines gives direct readings of density. It contains at the end of the arm a vessel in the form of frustrum of a cone (See Fig, p D77) of known volume. The sample is run freely from the funnel, shown on the left of the balance, into tared vessel in the same manner as described in item k. Then the balance is brought to equilibrium and density detd by direct reading (Ref 1, p 67 & Plate II B)

m) *Bulk (or Apparent) Density by Standard Vessel Method.* The US military procedure described in Ref 21 as Method 201.1, determines the wt per unit of outside volume of low sensitivity powdered expls. The apparatus consists of a funnel and a glass cup, serving as a "standard". The funnel is made of sheet metal 1/32" thick and of dimensions represented in Fig, p D77. Approximately midway along the length of the funnel stem, a metal sheet is inserted to serve as support for metal slide, 2x1-1/8", which controls the flow of the expl from the standard cup. This has a capacity of ca 60ml, an inside diam ca 1.65", an inside height ca 1.73", and a maximum wt of ca 41g. The top of the cup is flat and the inside surface concave at the bottom to prevent bridging of the sample over sharp corners. The sample consists of ca 85ml of the explosive (weighed to 0.1g) which has been thoroughly mixed to obtain representative distribution of large and small grains

Place a glass plate over empty cup and weigh the assembly to the nearest 0.1g. Remove the plate and fill the cup with distd w at room temp to form a convex meniscus over the top. Slide the plate over the top to remove excess water without entrapping air bubbles



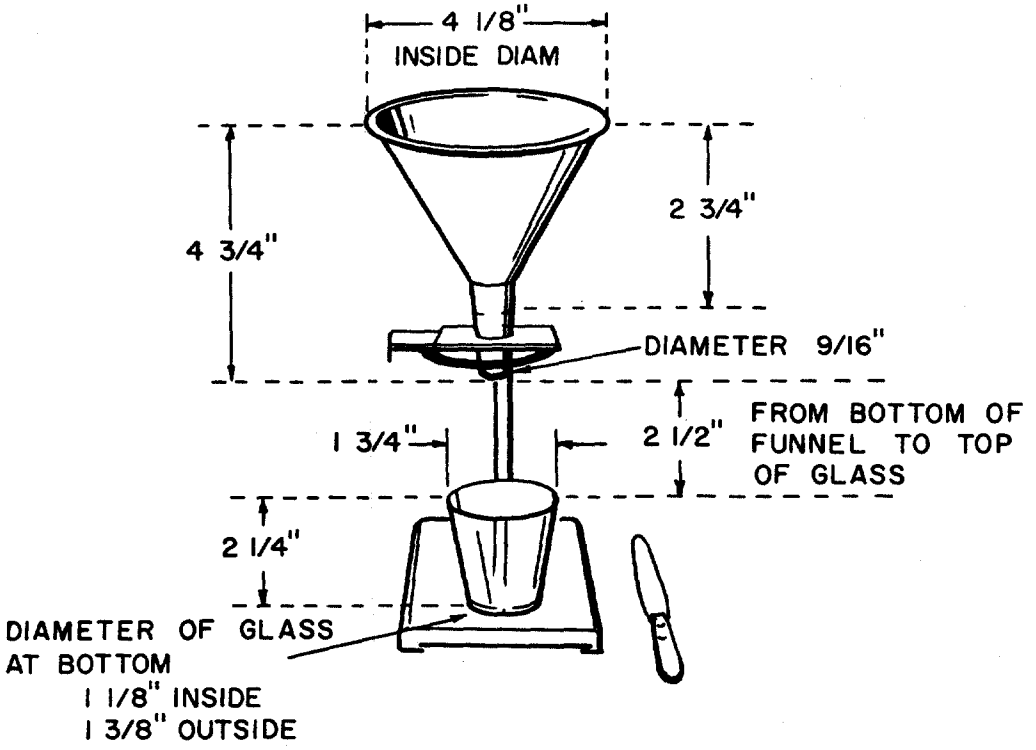
US Bureau of Mines Gravimetric Balance

under the plate. Weigh the assembly and calc the weight of w in the cup as

$$W=B-A,$$

where: W=Wt of water
A=Wt of cup with plate
B=Wt of cup with water & plate

Adjust the ring support of the funnel to make the distance from the bottom of the funnel stem to the top of the std cup exactly 2.5" and adjust the center of the cup to be directly under the funnel stem. Close the flow orifice of the funnel and pour there the previously weighed sample



Standard Vessel Method for Determing Bulk (Apparent) Density

Open the orifice to permit the free flow of sample into the cup until it is heaping full. Without tapping or jarring the cup (as this will cause the powdery material to settle thus giving high results), remove with a spatula the excess material from the top. Cover the cup with the plate avoiding to compress the material. Tap the full cup to settle the material in order to prevent spillage during weighing, but do not add more material. Weigh to the nearest 0.01g and calculate as follows:

Bulk or apparent d=(C-A)/W,

where: C=Wt of cup with plate & sample
A=Wt of cup with plate
W=Wt of water to fill the cup

n) *Bulk or Apparent Density by Dry Method.* The US military procedure described in Ref 21 as Method 201.3, determines the wt per unit vol of "packed" expls of low sensitivity (See Note below). The apparatus required consists of a 100ml, graduated in ml, cork stoppered, precision grade cylinder, meeting the requirements for accuracy as specified in Natl Bur Stds Circular C-434 (tolerance in 100ml is ± 0.20ml). Its length betw the 10ml and 100ml marks must be 145 ± 15ml

Tare the stoppered cylinder to the nearest 0.1g and transfer there a 50g sample. Weigh the stoppered cylinder with the sample and pack the material by dropping the cylinder vertically 50 times, from a height of 2.5" onto a piece of soft leather (or rubber). Level the surface of the sample by gently tapping and read the volume in ml

Density=W/V g/ml,

where: W=Wt of sample
V=Vol occupied by sample

Note: This density is called in Ref 21, Method 201.3 as "Bulk or Apparent Density", but actually it is "Packed Density", because it is detd after the material has been packed and tapped. Compare with "Bulk Density" detn described in previous item (m) and with "packed density" detn, described in the next item (o)

o) *Packed Density Determination of Powdered Materials.* The US military procedure described in Ref 22 as Method 400, has been used for detg the wt per unit vol of powdered material, which has been packed until it has attained its most compact form. Packed density indicates the loading density of loose pyrotechnic powders. The procedure is the same as in previous item (g), except that the sample size should be the wt of material filling the graduate cylinder to 100ml mark and packing is conducted by "instrumental method" instead of "hand-tapped" techniques. The graduate contg the sample should be stoppered and sealed with tape before inserting it into an apparatus which is represented on Drawing P-72534, and may be obtained on request from the US Dept of Defence, Washington, DC. In this apparatus, the motor revolves the cam at a constant rate of ca 60rpm, raising and simultaneously turning the graduate part way around before it drops to the rubber covered block. This should last exactly 10 mins

Packed density = W/V g/ml,

where: W=Wt of sample in the cylinder
V=Vol of sample in the cylinder

p) *Bulk or Apparent Density by the Wet Method.* The US military procedure described in Ref 21, as Method 201.2, for detg wt per unit of apparent volume of high sensitivity expls, is as follows:

Weigh to within 10mg 2 to 3g of dried expl to test and transfer it in small amts into 2ml of a liquid contained in a calibrated 5ml cylinder, having an inside diam of ca 9mm and graduated in 0.1ml divisions. Wash down any of the specimen which adheres to the walls of the cylinder with the same liquid until it fills the cylinder slightly above the 5ml mark. Allow to stand for 1 hr and note the vol occupied by the expl. If the vol exceeds 5ml, repeat the test using a smaller sample

Bulk d of expl = W/V g/ml,

where: W=Wt of sample
V=Vol occupied by sample

Note: In selecting the liquid to be used for a particular substance, three factors must be considered. The expl to be tested must not be sol in the liquid, the d of the sample must be greater than the liquid and the liquid must not be hazardous

q) Bulk (Gravimetric) Density by DuPont Densimeter. In the US military procedure described in Ref 20 as Method 502.1.1, the "bulk (gravimetric) density" of proplnt is defined as equal to the apparent sp gr multiplied by 1000. The apparent sp gr is defined as the ratio of the wt of a given vol of proplnt to the wt of the same vol of w at a temp of 70°F. (21.11°). The procedure is as follows:

Close the hopper valve of DuPont Densimeter and fill the hopper with 350-400cc of proplnt which was kept previous to test in storage for 24-36 hrs at a temp of 70°F. Quickly open the hopper valve completely permitting the proplnt to run thru the opening of 0.5" into the cup of vol 324.9 ml until it just overflows. Close the valve and remove the hopper. Run a straight edge across the top of the cup to remove the excess and then tap the cup gently to prevent spilling. Place the cup on the balance of the densimeter and adjust the wts. The reading on the scale is the bulk (gravimetric) density

Note: If the temp of proplnt is not 70°F, add 0.001 to the reading for every 2°F above 70°F, and subtract 0.001 for every 2° below 70°F.

r) Bulk Density in Pounds per Cubic Foot. The US military procedure described in Ref 20, as Method 502.1 for detg bulk d of proplnts, is as follows:

Tare on a trip balance, in grams, an empty cylinder 6" high & 2 5/16" ID, fill it with distd w at room temp and reweigh. Empty the cylinder dry it and fill completely with the proplnt permitting it to overflow. Run a straight edge across the top to remove the excess, tap the cylinder gently to settle the contents and prevent spilling in handling, and weigh

Bulk d = $[62.5(C-A)]/(B-A)$ lbs/cu ft,

where A = Wt of empty cylinder in grams

B = Wt of cylinder filled with water

C = Wt of cylinder filled with proplnt

s) Density of Loading of Explosives in Caps, The following procedure was developed at Picatinny Arsenal:

Mark each cap for identification and weigh on a analytical balance to the nearest 0.001g (W_0). Fill each cap with water to the top so that the surface of w is slightly convex, taking care not to overflow the cap. Weigh each cap (W_1) empty it and dry. Place in each cap exactly 0.400g of an expl to test and subject it to the pressure of 3000 psi acting for 3 mins. Remove the cap from the press and weigh to the nearest 0.001 g (W_2). Fill the upper part of each cap with water, as described above, and reweigh (W_3)

Calculations:

ld(loading density) = $0.400/(V_0-V_1) = 0.400/[(W_1-W_0) - (W_3 - W_2)]$ g/cm^3

where $V_0 = (W_1 - W_0)$ volume of water in each empty cap

$V_1 = (W_3 - W_2)$ volume of water in each cap contg expl

(Ref 106)

t) Density of Loading of High Explosives. The term "Density of Loading" (Densité de chargement in Fr) signifies the weight of a unit volume of an explosive when it is loaded in an item of ammunition, such a bomb, shell etc. In order to determine that the item is loaded at the proper density the following procedure may be used:

Select items, previously cleaned and dried, one from 100 of each type and weigh each of them empty, in pounds, on a rough balance (W_e). Fill them with water to the same levels as the explosives would occupy in loaded items and reweigh (W_w). Empty the items, dry them thoroughly and load with expls under conditions identical in all respects with those employed in loading other items of the same type. After loading reweigh each of the selected items (W_e) and calculate the loading density (ld) from the following formula:

$$ld = (W_l - W_e)/(W_w - W_e)\ g/cm^3$$

Following values are considered to be the minimum loading densities of expls: TNT (cast) 1.54; 50/50 - Amatol (cast) 1.50; 80/20 - Amatol (extrusion - loaded) 1.38; Explosive D (press-loaded in AP shells) 1.45; Explosive D (press-loaded in HE shells) 1.35; Tetrytol (cast) 1.60; 50/50 Pentolite (cast) 1.65; 60/40 Composition B (cast) 1.67; Trimonite (cast) 1.60

A list of US military explosives which are cast-loaded is given in Vol 2, pp C83-R & C84-L

Samples for the detn of loading density may also be taken from each increment of loading and should be sufficient in number to represent all parts of the loaded chge. These samples may be taken with a core drill or by using a metal bar of nonsparking material with a chisel point. Each sample should be cut to approx a 3/4 inch cube. The specific gravity should be detd by Mercury Displacement Method, given above as item g under Density and Specific Gravity Determinations of Solids

Note: When drilling sensitive expls, such as Tetryl, Comp B & PETN, it is advisable to direct a jet of cold w on the tip of drill to prevent heating

u) Density of Loading of Propellants. The US military procedure described in Ref 20, as Method 507.1, for detg the density of proplnts as loaded into cartridge cases, is as follows:

With a cartridge case in vertical position, place a funnel so that the bottom end of its stem is 2" above the mouth of the cartridge case. Pour the proplnt very slowly until it fills the case to the required height. Emty the case and weigh the proplnt chge. Fill the case with distd w at 70 ± 2°F from a burette until it fills the case to the same height as above and record the volume in ml:

Density of loading = W/Vg/ml,

where: W = Wt of sample in g

V = Vol of water in ml

(See also Ref 12, where an experimental formula for density of loading of proplnts is given)

v) Apparent Specific Gravity of Mining Explosives Cartridges by Sand Displacement. The opn, as conducted at the USBurMines, consists of filling a cylinder of known wt (W) and vol (V) with sand (Lake Michigan dune sand which passes thru a 30-mesh screen and is retained on a 80-mesh screen) and reweighed (W_s). The expression (W_s - W)/V gives density of sand (d_s) (usually 1.42). After pouring most of the sand into a beaker, insert in the cylinder the cartridge to test of known wt (W_c), with its smoother and flatter end embedded in the sand. While holding the top of the cartridge centrally in the cylinder by means of a pencil, fill the empty space in the cylinder with sand by pouring it from the beaker thru the spout. During this opn hold the spout as close as possible to the opening of the cylinder without touching it. Smooth off the surplus sand by a straight edge and weigh the cylinder contg cartridge & sand (W_{cs})

Volume of sand displaced by the cartridge:

$$V_c = (W_c + W_s - W_{cs})/d_s$$

Cartridge density:

$$d_c = (W_c \times d_s)/(W_c + W_s - W_{cs})\ g/ml$$

(Ref 3, p 14 & Ref 4, pp 22-3)

Note: Accdg to Marshall (Ref 2, p 414), the densities of Amer permissible expls detd by this method varied from 0.84 to 1.54 & those of European expls from 0.68 to 1.30

w) Shaking Density (USBurMines). The apparatus consists of a piece of Shelby tube 1¼" ID, 1 5/8" OD, and 12" inside length with a pipe cap on one end; a frame which allows the tube to be raised just 2" thereby obtg a constant height of drop to shake the tube; and a small plunger which measures the depth to which the expl has sunk after shaking. The base of the shaking frame is a piece of oak 2" thick which on a lead block. For calibrating the tube, weigh it first empty and then filled with water. For detg the density of powdery expl, allow it to run freely into dry tube until it fills it. Then tap the tube gently to obtain some space at the top for inserting the cork, and insert the tube into holder of shaking apparatus which operates by alternately raising (to the height of 2") and dropping the tube on the lead plate. Remove the cork and measure the depth to which the plunger sinks. This gives the shrinkage caused by shaking. Repeat the opn till constant volume is obtd. Pour the sample into a tared weighing bottle and weigh. The wt of sample divided by its vol in the tube, after shaking, is equal to "shaking density" (Ref 4, p 24)

Refs: (Density & Specific Gravity): 1) W.O. Snelling & C.G. Storm, "The Analysis of Black Powder and Dynamite", USBurMines Bull **51**, Washington, DC (1916), 67-8 [Determination of gravimetric and absolute densities; (Snelling's densimeter) plate II, gravimetric balance] 2) Marshall **2** (1917), 411 (Absolute specific gravity); 412 (Bianchi's densimeter); 413 (Snelling's densimeter); 414-5 (Gravimetric density) 3) S.P. Howell & J.E. Tiffany, "Methods for Routine Work in the Explosives Physical Laboratory of the Bureau of Mines", USBurMines Technical Paper **186,** Wash, DC (1918), 14-15 (Detn of apparent specific gravity by sand method) 4) C.E. Munroe & J.E. Tiffany, "Physical Testing of Explosives", USBurMines Bull **346,** Wash, DC (1931), 22-3 (Detn of apparent sp gr by sand test); 24 (Detn of shaking density) 5) Vennin, Burlot & Lécorché (1932), 154 (Gravimetric & apparent density) 6) Pepin Lehalleur (1935), 41, 54 & 62 (Densité de chargement); 99-100 (Densimeters of Bianchi and of Bode) 7) Hayes (1938), 50 (Loading density) 8) Clift & Fedoroff, Vol 1 (1942), Chap XVII, pp 5-6 (Detn of sp gr of BkPdr); Vol 3, Chap 2 (1943), pp 35-6 (Calibration of hydrometers) 9) Davis (1943), 267 & 360-63 (Density of loading of proplnts & expls) 10) J. Reilly & W.N. Rae, "Physico-Chemical Methods", Van Nostrand, NY (1943), vol 1, pp 113 & 465-502 (Density and its detn), 10a) F.D. Snell & F.M. Biffen, "Commercial Methods of Analysis", McGraw-Hill, NY (1944), 43-9 (Density detns)

10b) A.J. Clear, "Pruficiation of RDX with Cyclohexanone", PATR **1438** (1944), p 3 11) Pérez Ara (1945), 179-80 (Mallet-Bianchi's densimeter) 12) Ohart (1946), 176 (Density of loading, B, of a proplnt charge in a cartridge based on water, may be calcd from the formula:

$$B = 27.68\ W_c/V_c,$$

where: W_c is the wt of proplnt in pounds, V_c - capacity of the cartridge in cubic inches & 27.68 - the number of cubic inches per pound of water. B is usually 0.3 to 0.7) 13) Kirk & Othmer **4**(1949), 875-77 (Density and Specific gravity); 886-893 (Density determinations) 14) P. Tavernier, MP **31**, 198-99 (1949) & CA **46**, 11683) (Theoretical study of gravimetric and loading densities. Relation which exists betw the geometric dimensions of the usual forms of proplnts, such as cords, cylinders, tubes, balls & rods and the geometric dimensions of the charge containers. Discussion on the optimum charge density obtainable with each geometric form of proplnt) 15) Belgrano (1952), 15-17 (Detn of "Densità assolutà o reale" by Bianchi's densimeter and of "densità apparente o gravimetrica" by app called "gravimetro") 15a) Coll, "VanNostrand Chemists Dictionary", NY (1953), 756-R (Westphal balance) 16) W. Frey, "Specific Gravity of solids, Liquids and Gases", G. Hoehn, Zürich (1955), 75 pp 17) Blasters' Hdb (1958), 53 (Density) 17a) Rocket Encycl (1959), 49 (Bulk Density) 18) Giua, Trattato **6** (1) (1959), 17-18 (Densità di carica); 25 (Densità Limite); 451 (Densimetro di Bianchi) 19) J. Johnson, "Determination of Density" in Chap 6 of A. Weissberger, ed, "Physical Methods of Organic Chemistry", **3**, Wiley, NY (1959) 19a) A.B. Bofors, Nobelkrut, "Analytical Methods of Powder Analysis", Bofors, Sweden (1960), 22 (Mohr-Westphal Balance Method), 22-23 (Pycnometer Method), 23 (Bulk Density), 172 (Modified Bianchi Method for detg sp gr of smokeless proplnts), and 173 (Bulk Density detn of smokeless proplnts) 20) (US) Military Standard, "Propellants, Solid: Sampling, Examination and Testing", **MIL-STD-286A**, US Govt Ptg Off, Washington DC (1961) 21) Military Standard, "Explosive: Sampling, Inspection and Testing", **MIL-STD-650**, US Govt Ptg Off, Wash, DC (1962) 22) Military Standard, "Procedures for Determining Particle Size, Particle Size Distribution, and Packed Density of Powdered Materials", **MIL-STD-1233**, US Govt Ptg Off, Wash, DC (1962) 23) ASTMStds, Part **17** (Jan 1965), Method D941 Method D1217; Part **18**, (Jan 1965), Method D1298 and Part **19**, (March 1965), Method D1070 24)Blasters' Hdb (1966), 74 (Density of expls)

Addnl Refs (Density): A) Daneil (1902), 188 (Table of densities of some expls) B) Gody (1907), 146 (Detn of density of BkPdr as used in Belgium) C) H. Dautriche, MP **15**, 137 & 212 (1909-10) & **16**, 29 (1911-12)(Limiting density & optimum density) D) H. Muraour, MAF **18**, 895-97 (1937) (Sur la surcompression des explosifs d'amorçage) E) J.L. Culbertson et al, JACS **59**, 306 (1937) & **60**, 2695 (1938) (Densities of fine powders, determination) F) H.K. Alber, IEC (AnelEd) **12**, 764-67 (1940) (Detn of sp gr) G) P. Caldirola, RendIstLombardo Sci **77**, No 2, 12pp (1943-44) (Exptl data on the variation of the velocity of detonation as a function of the density can be used to perfect the method of calcn of pertinent parameters of detonation) H) A.F. Belyaev & A.E. Belyaeva, DoklAkadN **50**, 295-97 (1945) & CA **44**, 10321 (1950) (Relation betw the limiting density of expls and diameter of charge) I) H.A. Gardner & G.G. Sward, "Physical and Chemical Examination of Paints, Varnishes and Colors", H.A. Gardner Laboratory, Inc., Bethesda, Md (1950), 402-03 (Description of Scott's Volumeter, manufd by Fisher Scientific Co; it serves for detn of volume of powdered materials) J) A. M. Cummings, USP 2577110 (1951) & CA **46**, 3764 (1952) (Density of AN based expls can be controlled by the addn of vermiculite) K) R.L. Wagner et al, "Investigation of British Service Lead Azide", EDS (Explosives Development Section) Rept **457248**, PicArsn, Dover, N.J. (1957) (Loading density of British Service Lead Azide) L) A.H. Thomas Co. Philadelphia Pa, Catalog (1961), p 1005 (Volumeter Scott, Schaeffer & White which is improved Scott's Volumeter. M) Fisher Scientific Co., Pittsburg Pa, Catalog (1962), p 745 (Scott's Volumeter); p 66 (Westphal Balance); pp 632-38 (Hydrometers) & pp 26, 101-02 814 (Pycnometers) N)Blasters' Hdb (1966), 74 (Density of expls)

Density of Blasting Explosives; Density in Borehole; Location of Boreholes; Controlling Charge Densities and Stemming. These subjects are discussed in DuPont's publications entitled "Blas-

ters' Handbook" (Refs 2 & 3)

In the case of dynamites, the *density* is expressed in terms of the number of 1¼ x 8 inch cartridges contained in a 50 pound box. In the present line of DuPont expls this varies from 85 to 205 cartridge s per 50 lbs. The purpose of d variations in expls is to enable the miner to concentrate or distribute charges at will

The *density in borehole* is the weight of expl charge per unit length of a borehole. In US it is expressed in pounds per foot

The following table gives pounds per foot of DuPont blasting expls (Ref 3, p 189):

Table 1

Diameter Inches	DuPX-A "Gelex" 1 RCX	DuPX-B "Gelex" 2 F.R. 1	DuPX-C&D "Gelex" 3 F.R. 2, 3, 4	DuPX-E&F "Gelex" 4, 5 F.R. 5	DuPX-G&H	Str. Gel. 20–50 Sp. Gel. 20–50 "Hi-Velocity" 40	Str. Gel. 60–90 Sp. Gel. 60–90 "Hi-Velocity" 50–60	"Hi-Velocity" 70–80
1	0.4	0.4	0.4	0.3	0.3	0.5	0.5	0.4
1¼	0.7	0.6	0.6	0.5	0.4	0.8	0.7	0.6
1½	0.9	0.8	0.7	0.6	0.6	1.2	1.1	0.9
1¾	1.2	1.1	1.0	0.9	0.8	1.5	1.4	1.2
2	1.7	1.5	1.4	1.3	1.1	2.1	2.0	1.7
2¼	2.0	1.8	1.7	1.5	1.3	2.6	2.5	2.1
2½	2.4	2.2	2.0	1.8	1.6	3.3	3.0	2.6
2¾	3.1	2.8	2.6	2.3	2.0	4.0	3.8	3.2
3	3.9	3.5	3.3	2.9	2.5	4.6	4.4	3.7
3½	5.8	5.2	4.8	4.4	3.8	6.2	5.9	5.0
4	7.5	6.8	6.4	5.7	5.0	8.0	7.6	6.4
4½	9.5	8.6	8.0	7.2	6.3	10.1	9.6	8.0
5	10.9	9.8	9.2	8.2	7.1	12.5	11.8	10.0
5½	13.1	11.8	11.1	9.9	8.6	15.1	14.2	12.0
6	15.6	14.1	13.2	11.8	10.3	18.0	17.0	14.4
7	21.3	19.2	18.0	16.1	14.0	24.5	23.1	19.5
8	27.7	25.0	23.4	21.0	18.2	32.0	30.2	25.5
9	35.0	31.6	29.6	26.6	23.0	40.5	38.2	32.2
10	43.3	39.0	36.6	32.8	28.4	50.0	47.2	40.0
11	52.3	47.2	44.3	39.7	34.4	60.5	57.0	48.2
12	62.4	56.2	52.7	46.5	41.0	72.0	68.0	57.5

Table 2 gives pounds of typical US dynamites per foot when using 1¼ x 8 inch cartridges of various densities. The cartridges were slit and well tamped (Refs 1 & 2):

Table 2

Diameter of Borehole in inches	Number of Cartridges per 50 lb Box						
	90	100	120	140	160	180	200
1.0	0.54	0.48	0.40	0.34	0.30	0.27	0.24
1.5	1.20	1.08	0.90	0.77	0.68	0.60	0.54
2.0	2.14	1.92	1.60	1.37	1.20	1.07	0.96
2.5	3.34	3.00	2.50	2.12	1.87	1.66	1.50
3.0	4.81	4.32	3.60	3.08	2.70	2.40	2.16
3.5	6.50	5.85	4.88	4.17	3.66	3.25	2.92
4.0	8.50	7.65	6.37	5.46	4.78	4.25	3.82
4.5	10.73	9.67	8.06	6.90	6.04	5.36	4.83
5.0	13.35	12.00	10.00	8.58	7.50	6.68	6.00
6.0	19.01	17.25	14.38	12.31	10.75	9.58	8.63
8.0	34.1	30.75	25.61	21.99	19.20	17.08	15.38

The proper *location of boreholes,* known also as *hole placement,* involves several factors, one of the most important being density of the expl charge as loaded. As the cost of hole drilling is very expensive in the US (cost per lineal foot may run as high as 10 to 14 times the cost of one foot of expls loaded in a borehole), it is often preferrable to drill the holes far apart and use stronger (higher density) expls although they are more expensive than the weaker (lower density) ones. The high d expls, known also as *low count explosives* work fine in mining hard ore and in driving tunnels thru hard rock, but they are not suitable in coal mining when producing lump coal is required. Here low d expls must be used and the boreholes are drilled closer. In still other instances, as in quarrying, a high d expl is sometimes used in the bottom of the hole where the work to be done is the greatest, and a lower d expl at the top, where very little energy is ordinaryly required (Ref 3, p 188).

The purpose of *controlling the charge density* of blasting expls is to obtain the optimum results with the minimum amounts of cheaper (lower d) expls. With the wide variety of expls now available, it is possible to obtain any desired loading d by combining the grade and size of expl with the proper loading procedure. As in most types of blasting, more energy is required to break the burden near the bottom of the hole than toward the collar; the heavier grade of expl can be used at the bottom and lighter grade in the upper part. It is preferable, however, to use the same expl for each borehole, but to tamp the lower part of the charge solidly in the hole, allowing the balance of the chge to "build up" toward the collar. If none of the above methods achieves proper blasting action, it is probably more economical to use the denser expls and string out the load. In horizontal holes this can be done by the use of "spacers", which are rods 8-10 inches long, square in cross section with an area not over half that of the borehole, so as not to interfere with the propagation of the charge. They are usually made of wood or of incombustible materials, such as clay, tile or concrete. Plastic tubes 8-12 inches long also proved satisfactory. With this method, the lower part of the charge should contain the primer and one or more high d cartridges. Then the spacers are introduced, placing one betw each chge of one or two cartridges until the desired length of the column has been reached. Controlling the chge d in such manner is not, however, feasible in vertical holes since the space betw the cartridges may become entirely filled with fine "stemming" (see below) material which sifts down the hole, thus causing a partial failure. In this case, the so-called *deck loading* can be used. This method is similar to the use of spacers except that the sections of hole betw the several charges are completely filled with drill cuttings, rock chips, or other stemming material; each "deck charge", as well as the main charge at the bottom of the hole, is separately primed (Ref 3, p 187)

It is generally advisable to cover the charge of expl in a borehole by suitable inert material (such as clay, concrete, etc.), known as *stemming.* The main purpose of stemming is to confine the charge and thus obtain the best possible efficiency from the expl. Another purpose is to protect the loaded chge from accidental ignition or detonation. This is of importance where BkPdr is being used, especially when it is fired with safety fuse which is ignited by means of an open flame lamp. As BkPdr builds up its gas pressure slowly and is apt to blow out thru the borehole. The stemming should be tight. Although the dynamite chges cannot be ignited as readily as BkPdrs, they should also be protected with stemming especially in vertical boreholes where it is easier for fire or sparks to enter from a number of sources (Ref 3, p 188)

In selecting expls for blasting charges, it is important that they are *sensitive* enough to insure propagation of detonation thru a column of cartridges in a borehole, as well as between several charges in separate boreholes (Ref 3, p 53)

Refs: 1) Olin Industries Inc., "Explosives Products", Pamphlet, 5th ed, p 55 2) Blasters' Hdb (1949), 423 3) Blasters' Hdb (1958), 53 & 187-190; and (1966), 219 (Control of chge density), 490 (Densities of DuPont products) and 492 (Conversion of g/cc to lb/ft)

Density, Bulk (of Liquid Rocket Propellants). This is the density of a theoretical fluid that represents all the liq oxidizer and all of the liq fuel consumed during the time that *specific*

impulse is measured or detd. This term is of interest principally when *density impulse* is under consideration. Bulk density is calcd by the equation:

$$\rho_{bulk} = (1 + r)/(r/Poxid + r/Pf)$$

where ρ_{bulk} = build density in lb/in^3
r = ratio of the wt of oxidizer divided by the wt of fuel consumed
ρoxid = density of liq oxidizer in lb/in^3
and ρ_f = density of liq fuel in lb/in^3

Ref: Rocket Encycl (1959), 59

Density - Detonation Velocity Relationship. See under DETONATION AND EXPLOSION, Velocity of Detonation in Vol 4 of this Encycl

Density of Energy (Concentration of Energy). This is, accdg to Colver (Ref), the amount of energy stored in a unit volume of explosive charge. To obtain a maximum density of energy, the expl must be packed as tightly as possible, which means that its loading density must be at a maximum

Expl substances, not possessing sufficient density of energy (as for example gaseous mixts, such as H_2 plus O_2; C_2H_2 plus O_2, etc), cannot be used in practice as expls. Their initial volume is so great that the increase in volume due to the expln would be insignificant compared to that obtd from expls of high loading d

Ref: Colver (1918), 654

Density of Fragments (Splinters) Test or Fragment Concentration Test (Splitterdichterprobe in Ger). A series of tests was conducted during WWII by the German Ordnance Department (Waffenamt) under the direction of Dr Römer in order to determine the relation between: effective fragment (splinter) weight, fragment velocity, fragment velocity, distance of travel (range), the weight & type of expls used and the type & thickness of steel used in ammunition. Results of these tests were useful for designing new, more effective bombs and shells

One of the tests used for this purpose was conducted by detonating an item, to be tested, inside of an enclosure (fence) consisting of boards 2-inch thick. The number of fragments per square meter was counted and the average distance at which there would be one fragment per square meter was calculated from a specially constructed curve. In order to obtain reliable results it was required to detonate at least 10 bombs or shells of the same size for each expl to be tested

Following are some calcd values for the distance (average) at which one penetration per sq m is achieved on detonating a 105-mm shell, when using various expls: TNT 39-40m; 40/60 AN/TNT, 38-39m; 50/50 AN/TNT 35m; 60/40 AN/TNT 34m; 50/50 TNT/NaCl 26m; and 40/60 TNT/NaCl 23m

Refs: 1) G. Römer, PBL Rept 85160 (1946) & private communication, Dec 1953 2) B.T. Fedoroff et al, PATR **2510** (1958), p Ger 52-R

Density Impulse. The density impulse (I_d) equal to the product of specific impulse by mass density, is a useful parameter in liq - proplnt rocket engineering. It gives the performance on the basis of wt per unit vol of the proplnts which yielded the performance

Many liq-proplnt combinations yield a high value of *specific impulse* but often these combinations have a relatively low *bulk density*. This necessitates large proplnt tanks and increases the aerodynamic drag & the dry wt. The ideal liq-proplt combination is one which yields a very high specific impulse yet is also made up of a high-density liq fuel & oxidizer. A large amt of chem energy is thus packed into the smallest volume. The highest values are, of course, the most desirable

Ref: Rocket Encycl (1959), 126-27

Density of Loading of Cast Explosives, Increase of. Loading density of cast nitrocompounds may be increased 2 to 6%, if they are subjected, while in the molten state, to a strong vacuum in order to remove the occluded gases and then allowed to solidify while maintained under gaseous pressure (Ref 1). The density may also be increased by heating one end of a charge during casting in order to keep it semi-liquid, while gradually chilling the other portion of the charge. During this procedure, the gaseous pressure is applied to the heated portion of the charge. Finally the heat is removed and the fused portion is allowed to solidify (Ref 2)

Refs: 1) C.E. Bichel, USP 925419 (1909) & CA **3**, 3227 (1909) 2) G. Issler, USP 1074623 (1913) & CA **7**, 3842 (1913)

Density Values of Some Explosives as Determined by Stettbacher. Stettbacher detd in 1942 the true crystal densities of various expls, using as a medium paraffin oil, water, ether or benzene in the Bianchi's or Zeiss-Ikon apparatuses or in a pycnometer

Following are some density values as detd by pycnometer method

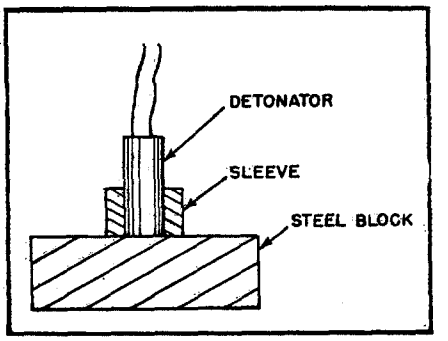
Explosive	Temperature °C	True Density g/cm^3	Liquid Used in Pycnometer
PETN	0°/4°	1.7653	Paraffin Oil
RDX	20°	1.816	Paraffin Oil
LA	15/4°	4.716	Ether
AgAzide	20°	5.1	Benzene
$AgC_2.AgNO_3$	20°	5.385	Benzene

Stettbacher also detd densities of some smokeless proplnts but he did not give any values. He recommended to use benzene as the medium and at least a 10g sample
Ref: A. Stettbacher, Nitrocellulose **13**, 23-26 (1942) & CA **37**, 4900 (1943)

Dentex. An aluminized expl (RDX 48, DNT 34 & Al 18%), developed in US during WWII to replace **Torpex 2** (RDX 42, TNT 40 & Al 18%), which was considered to be too sensitive for some ammunition. Dentex is less powerful and brisant than Torpex 2 (See Vol 2, pp B27) & B 291) or **HBX-1** (RDX 39, TNT 37.8, Al 17.1, Desensitizer D2 5.0 & Ca chloride 0.5%), and not as suitable for underwater explns
Refs: 1) Coll, "Summary Technical Report, Division 8, NDRC", Vol 1, Washington, DC (1946), 33 2) TM 1910 (1955), 198

Dent Tests. See Plate Denting Tests (PDT) in Vol 1, pp XIX-XX and under Brisance Test Methods in Vol 2, p B299-L. The values of PDT's for various expls are given in Table 1, pp B266-B295 in Vol 2

The test described by V.J. Menichelli in Ordn **48**, 663-66 (1964) is easily performed and is claimed to yield results which correlate with the ability of an expl to initiate the next component in an expl train (See Fig). The criterion



Typical Arrangement in Steel Dent Test

of the test is the depth of dent produced in a steel block. More exactly it is the vol of the dent. For a given device, however, the diam is uniform and the depth of dent may be simpler to measure. Although the steel dent test may be superior to other tests (such as Trauzl, Sand Bomb, Lead Disc & Gap Tests), precautions must be taken, since confinement, confining media, type of steel block, and hardness of block all affect the results

DEP or DETPh Abbr for Diethylphthalate

Depot. An establishment and installation for storing supplies or for maintaining equipment, or for any combination of these activities
Ref: Glossary of Ord (1959), 93-L

Dépôt d'explosifs or Magasinage des explosifs (Fr). Storage of Explosives

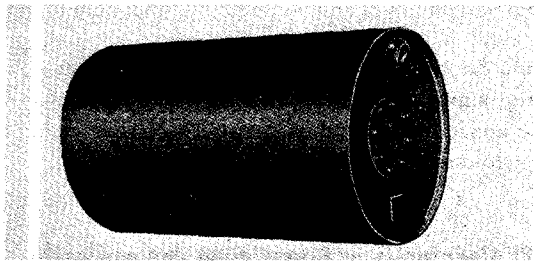
Depth Charges and Their Explosives. The sole purpose of a depth-charge is to destroy a submerged submarine and, as such, is still the most widely used weapon in spite of many new developments in anti-submarine warfare. A depth charge is essentially a thin-walled metal-

lic container filled with a powerful explosive. The most widely used expls are TNT and its mixts with Al, such as *Tritonal (UWE)* (TNT 80 & Al 20%) or with Al & RDX, such as *DBX* (TNT 40, RDX 21, Al 18 & AN 21%). The Germans used during WWI mixts of TNT with Hexanitrodiphenylamine, such as *Hexamit* (HNDPhA 60-70 & TNT 40-30%). During WWII the mixts contained Al (such as TNT 60, HNDPhA 24 & Al 16% or TNT 67, HNDPhA 8 & Al 25%).Some Ger mixts contd PETN, such as PETN 25, TNT 48 & Al 27% (Ref 4)

Each depth charge is provided either with a hydrostatic exploder (which detonates the chge at a predetermined depth below the surface of the sea), or a contact fuze. In order to sink or damage a submarine, the depth chge must explode in the immediate vicinity of the target, no matter what its depth is

The following types of depth chges were used during WWII:

a) *Cylindrical Depth Charge,* commonly known *Ash Can* of which MK 6 was the most popular. It contd 300-400 lbs of cast TNT. These charges were launched from a gun which was located at the stern of the ship. The K type of gun consisted of a smooth-bore barrel attached to an expansion chamber fitted with a breech mechanism. The gun was secured to the rail of the ship and could not be elevated or trained. Variations in the range were obtd by changing the weights of propelling charges

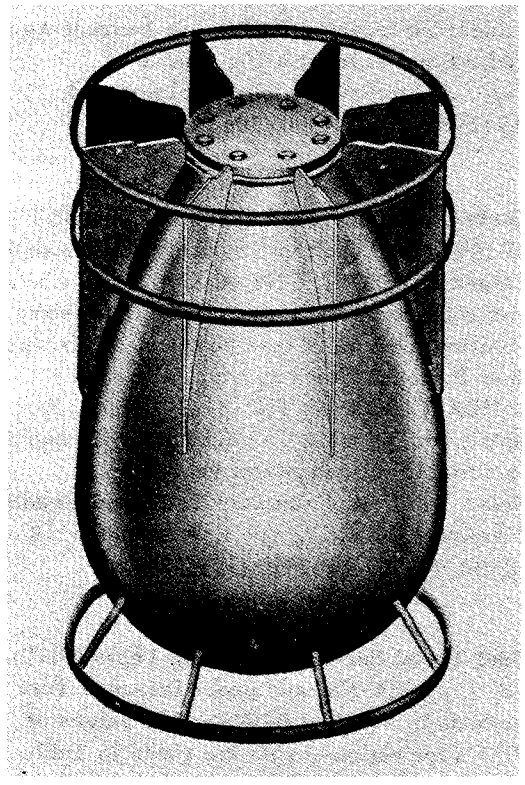


MK6 Cylindrical Depth Charge

b) *Teardrop Depth Charge.* This type designated as M-9 contd 200 lbs of TNT. Its streamlined case and rotation-inducing canted fins permitted the device to sink on a fairly straight line with a velocity of ca 20 ft/sec. It was usually launched from a K gun and was considered more effective than other charges. Much work was in progress at the end of WWII relative to improving its fuzing

c) *Projector Missiles.* These were cylindrical in shape charges contg ca 31 lbs TNT and provided with impact fuzes. They were propelled from projectors such as MK-10 based on the British "Hedgehog" projector. The MK-10 launched 24 charges at a time and was fired in pairs, in conjunction with cylindrical chges to increase the effectiveness of the attack. The disadvantage of this projector was strong recoil and this prevented the use of K-10 in small craft. The projectors known as "Mousetrap" and MK-20 & MK-22 were designed for use in small craft (Refs 2 & 5)

McDugall et al patented (Ref 6) depth charge expls which are more powerful than TNT alone. These mixts can be prepd by mixing TNT with Ba nitrate and aluminum. For example: TNT (granulated) 35, Ba nitrate (tech, powdered; which almost completely passes thru a 100-mesh screen and ca 75% passes a 200-mesh screen)



MK9 Teardrop Depth Charge

50 and Al (atomized of which at least 95% passes thru a 200-mesh screen) 15% are blended and cast to a slightly porous hard material of density 2.3. This mixt does not explode on being heated on a Wood's metal bath (360°C) and, on being heated for 2 hrs at 200° or 5 hrs at 135°, it does not decompose. Its sensitivity to impact is comparable to that of TNT, but it partially detonates in the rifle bullet test

Japanese Navy bombs designed for use against ships are called "ordinary". They include both GP & SAP types and are of smoother construction than land bombs. They are loaded by casting directly into the case with fillings, such as Type 9 (Trinitroanisole) or Type 98 (Picric Acid) (Ref 3b)

Accdg to Ref 1, the Japanese cylindrical charges were equipped with a device called "pistol" to detonate the chge at a definite depth. The pistol was housed in a central tube running along the axis of the cylindrical body. The pistol made use either of the ratio between water pressure and depth of sinking, or of the ratio betw time and depth to accomplish detonation

The Ger *Bomben Torpedo* (abbrn BT) is described in Vol 2, p B241-L under Bombs, Torpedo

Italian anti-submarine bombs closely resemble Japanese bombs in that they are usually more than one-piece in construction. They are normally filled thru the base with a TNT filler (Ref 3c, p 22)

Little info is available on French Navy bombs, except that the nature of the filling is TNT or Picric Acid (Ref 3c, p 178)

Refs: 1)OpNav **30-3M** (1945), 61 2)B.Rowland & W.Boyd, "US Bureau of Ordnance in WWII", Dept of the Navy, Washington, DC (1953), 136 3a)TM **9-1985-2** (1953), 44-45 3b)TM **9-1985-4** (1953), 50-58 3c)TM **9-1985-6** (1953), 22 & 178 4)PATR **2510** (1958), p Ger 88-R (Hexamit) & p Ger 212 (Under water expls) 5)Anon, "Ammunition Ashore. Production and Renovation", US Bur Ord, Dept of the Navy, Washington, DC, OP 5, Vol **2** (1958), p 31.1 6)D.P.McDugall et al, USP 2918362 (1959) & CA **54**, 8087 (1960) (Depth charge and sea mine expls)

Dermatitis and Dermatosis in War Industries. *Dermatitis* is an inflammation of the skin caused by direct action of a chemical substance and is characterized by redness, heat and swelling; whereas *dermatosis* is a disease of the skin usually associated with an occupational causal factor previously established to have produced similar changes in the skin of other workers

Occupational dermatitis and dermatosis may be caused in war industries in handling explosives (such as MNT's, DNT's, TNT's, MF, LA, PA, Tetryl, etc), acids (such as nitric, hydrochloric, sulfuric, chromic, etc), cutting oils, paints, degreasers, toluene, benzene, nitrous gases, etc. The diagnosis, prevention, treatment, protective clothing and protective ointments are discussed in the Refs

Refs: 1)W.F.von Oettingen, "Toxicity and Potential Dangers of Aliphatic and Aromatic Hydrocarbons. A Critical Review of the Literature", US Public Health Service Bulletin **225** (1940) 2)L.Schwartz & L.H.Warren, "Occupational and Related Dermatosis", US Public Health Service Bulletin **266** (1941) 3)W.F. von Oettingen, "The Aromatic Amino and Nitro Compounds, Their Toxicity and Potential Dangers. A Review of the Literature", US Public Health Service Bulletin **271** (1941) 4)L.Schwartz, Trans Natl Safety Congr **30**, 308-15 (1941) & CA **39**, 1481 (1945) 5)Anon, "Occupational Hazards to Young Workers", The Explosives-Manufacturing Industries Report No 1, US Dept of Labor (1942) 6)W.F. von Oettingen et al, "The Toxicity and Potential Dangers of Toluene, with Special Reference to its Maximum Permissible Concentration", US Public Health Service Bulletin **279** (1942) 7)L.Schwartz, Ind Med **11**, 457-62 (1942) & CA **37**, 969 (1943) 8)L.Schwartz, Proc 7th Ann Meeting, Ind Hyg Foundation Am, 36-47 (1942) & CA **37**, 2095 (1943) 9)C.Swanston, Proc Roy Soc Med **36**, 633-38 (1943) & CA **38**, 5672 (1944) (Effect on the skin of irritant expls) 9a)W.F. von Oettingen, "The Aliphatic Alcohols", USPHS Bull **281** (1943) 10)H.M.Primster & R.O.Anderson, Med J Australia **31**, 104-06 (1944) & CA **38**, 4805 (1944) (The incidence of Tetryl dermatitis, known as "CE Rash") 11)W.F. von Oettingen et al, "Toxicity and Potential Dangers of Penta-Erythritol-Tetranitrate (PETN)", US Public Health Service Bulletin **282** (1944) 12)R.F.Sievers et al, "A Medical Study of the Effect of TNT on Workers in a Bomb ans Shell Loading Plant", US Public Health Service Bulletin **291** (1945) 12a)W.F.

von Oettingen, "The Effects of Aliphatic Nitrous and Nitric Acid Esters on the Physiological Functions with Special Reference to their Chemical Constitution", US National Inst of Health Bull **186** (1946) 13)R.F.Sievers et al, "Toxic Effects of Tetranitromethane, A Contaminant in Crude TNT", US Public Health Reports **62**, No 29, 1048-61 (1947) (Reprint No 2793) 14)Kirk & Othmer **1** (1947), 552 (Dermatitis) 15)S.J.Hopkins, Mfg Chemist **18**, 389-93 (1947) & CA **42**, 1364 (1948) (Industrial dermatitis and the formation of barrier creams) 16)L.Schwartz et al, "Occupational Diseases of the Skin", Lea & Febiger, Philadelphia (1947) (Reviewed in Military Surgeon **101**, 356 (1947) 17)R.L.Sutton & R.L.Sutton, Jr, "Handbook of Diseases of the Skin", Mosby & Co, St Louis (1949), 719pp 18)S.Greenbaum, "Dermatotogy," Davis Co, Philadelphia (1949), 924pp 19)R.C.Low & A.G.Peterkin, "The Common Diseases of the Skin", Oliver & Boyd, Edinburgh, 4th ed (1950), 282pp (Reviewed in Bull Johns Hopkins Hospital **87**, 89 (1950) 20)H.O.Mackey, "A Handbook of Diseases of the Skin", Fallon, Dublin (1952), 216pp (Reviewed in J Am Med Assoc **150**, 1441 (1952) 21)S.Kubota, J Soc Org Synthetic Chem, Japan **14**, 351-52 (1956) & CA **51**, 3143 (1957) (TNT dermatitis) 22)G.H.Percival, "An Introduction to Dermatology", Williams & Wilkins, Baltimore, 12th ed (1956), 374pp 23)Sax (1957), 207-24 24)K.L.Golshmid, "Zabolevaniya Kozhi u Rabochikh Khimicheskikh Proizvodstv,Ikh Preduprezhdenie i Lechenie" (Skin Diseases of the Workers of Chemical Industries), Knizh-Izdatel, Perm (1960), 71pp 25)R.L.Baer & V. I.Witten, edits, "The Year Book of Dermatology", Year Book Medical Pubs, Chicago (1961), 478pp

Derne Mining Association Testing Gallery. See Vol 3, p C371 under Coal Mining Explosives, Testing for Permissibility **E.** *Germany*

Dervish. An Amer air-to-air missile manufd by the Northrop Corp, Beverly Hills, Calif. It consists of a small spin-stabilized rocket, 2.75" in diam, powered by a Thiokal solid proplnt
Ref: I.F.Ordway & R.Wakeford, "International Missile and Spacecraft Guide", McGraw-Hill, NY, (1960), p USA 31

Desensitization (Phlegmatization) of Explosives; Desensitizers and Desensitized Explosives. *Desensitization (or phlegmatization) of an explosive,* means rendering it insensitive or less sensitive to the following actions: heat, shock, impact, percussion, rifle bullet or friction *Desensitizer (or phlegmatizer)* is a substance which makes insensitive or reduces the sensitivity of an explosive. The term phlegmatizer is also used for a material added to a cannon proplnt to reduce its erosive effect in gun barrels *Desensitized explosives* are those which are made insensitive to the above listed actions. Such expls are safe to handle and to transport. Typical examples of such expls are RDX Compositions A (See Vol 3,p C474-L); PETN/Wax compns, such as Fillers Nos 32, 33 etc, described in Ref 18, p Ger 47-Rff (See also Refs 1, 2, 15, 18 & 19)

Most of the primary expls (MF, LA, Tetracene, etc) and many high expls (NG, Tetryl, PETN, RDX, etc) are too sensitive to be used per se and must be desensitized. For example LA may be desensitized by coating its crysts either with dextrin (See Dextrinated Lead Azide in Vol 1, pp A546 to A548) or with PVAlc (polyvinyl alcohol) (See Vol 1, p A558-R). It can also be desensitized by controlling the size and shape of its crysts [See Service Lead Azide (Brit) in Vol 1, p A557-R], or by pptg it in colloidal form (See Vol 1, p A558-L)

Liquid HE's (such as NG, NGc or some other nitric esters) can be desensitized by mixing them with substances absorbing or adsorbing them. Such substances can either be inactive (as formerly used kieselguhr) or active (as sawdust, meals, collodion cotton, etc). The resulting combinations, known as Dynamites, are weaker than NG and are not liquid but either powdery or gelatinous. As some industries (such as in blasting petroleum wells) require the use of powerful liquid expls (such as straight NG), it was necessary to devise safe methods for their delivery. This can be achieved by using a desensitizer which does not chemically attack NG and which can be easily removed before use of NG is required. One of the European methods is to mix NG with kieselguhr, ship it (such mixt was allowed for transportation) and then leach the product with warm water; the separated NG settled at the bottom of the tank and could be easily removed. In the US and some other

countries, NG has been mixed with methylol (or acetone), shipped and then treated with water. NG or other liq nitric esters may also be desensitized by addg water-sol, nonvolatile compds (such as diols contg 3-6C atoms, Cellosolves or Carbitols. For example, 60 parts of NG, 34ps of diethylene glycol & 1p of Et Centr are mixed in Cu cans for shipment. To recover the NG, 30parts of mixt and 60ps of water are agitated by air in a lead tank for 10 mins and, after allowing to settle, the aqueous layer is removed. This leaves at the bottom the NG with dissolved Et Centr which serves as a stabilizer. The recovery is 98% (Ref 16)

For desensitization of solid HE's, the usual procedures are to coat them with one or several of the following: paraffin, paraffin oil, waxes, stearic acid, stearates, PVAcet (polyvinyl acetate) DNT, etc. Wöhler (Ref 3) detd the maximum percentage of paraffin oil which could be incorporated in a HE without making it detonate incompletely. He named this value *Phlegmatizierungsgrenze* (Phlegmatization Limit), which probably may be called *Desensitization Coefficient.* These values were found to be: 6.75% (of paraffin oil) for TNT, 8.25 to 8.75% for Tetryl and 8.25% for PETN. The procedure was as follows:

About equal quantities (0.7 to 0.85g) of straight HE's were compressed at 100kg/cm^2 in Cu caps placed on a lead plate and initiated with a No 8 LA detonator. This produced complete detonations and indentations in lead plates which were measured and recorded. The tests were repeated using the same HE's but mixed with varying amts (5 to 9%) of paraffin oil. The tests were continued until the amt of oil started to cause incomplete detonation (See also Ref 4)

For other methods of desensitization, see Refs 5, 6, 7, 8, 9, 10, 11, 12, 13, 14 & 17)

Refs: 1)K.A.Gillespie, USP 1370015 (1921) & CA **15**, 1622 (1921) (A desensitized expl, prepd by mixing undried Nitrostarch with heavy petroleum lubricating oil and gum arabic, was insensitive to rifle bullet) 2)W.H.Ward, USP 1462753 (1923) & CA **17**, 3101 (1923) [A desensitized expl suitable for use in drop bombs and trench mortar shells consists of Nitrostarch 40-45, inorg nitrate (such as of Na or K) 32-40, heavy lubricating petroleum oil up to 5 and water 5-10%] 3)L.Wöhler, SS **20**, 166 (1925) (Desensitization of HE's with paraffin oil) (See in text) 4)L.Wöhler, SS **21**, 1-5, 35-8, 55-7, 97-9 & 121-23 (1926) (Desensitization of HE's with paraffin oil) 5)J.Rubin, "Desensitization of High Explosives by Waxes", PATR **1489** (1945) (Evaluation of commercially available synthetic waxes as desensitizing agents for PETN & RDX) 6)J.Rubin, Ibid, PATR **1499** (1945) [Evaluation of Swiss P Appl 439767 for desensitization of PETN with PETS (pentaerythritol tetrastearate.) Of the three compns described in patent, one appears of interest as a military HE] 7)J. Rubin, Ibid, PATR **1533** (1945) (Prepn of PETN -wax compns by slurry method studied on both laboratory and semi-plant scales) 8)E.G.Cockbain & J.L.Moilliet, Brit P 574271 (1945) & CA **42**, 7531 (1948) (Desensitization of PETN & RDX may be effected by adding, with stirring, to a hot aqueous slurry of the expl, a small amt of a water-sol salt of an aliphatic carboxylic acid of at least 8C atoms per mol or an aliphatic or aromatic amine, each of at least 8C atoms per mol and a sufficient amt of molten wax, followed by cooling, filtering, washing and drying of solid blend) 9)H.A.Aaronson, "Semi-Plastic RDX Composition", PATR **1634** (1946) (Investigation of RDX coated with polyvinylacetate & dibutylphthalate: RDX 90, PVAcet 8 & DBuPh 2%) 10)E.G.Cockbain & ICI Ltd, Brit P 574879 (1946) & CA **43**, 1984 (1949) (Coating of particles of expls, such as PETN with wax, followed by coating with an insolubilized protein or its derivs) 11)H.G.Hyatt & E.S.Shanley, USP 2453070 (1948) & CA **43**, 1806-07 (1949) [Solid org peroxides (such as Bz_2O_2) are rendered stable to impact and are made relatively slow -burning by pptg on the surface 2-10% by wt a water-insol aliphatic carboxylic acid (such as lauric acid)] 12)Ibid, USP 2453071 (1948) & CA **43**, 1807 (1949) [$CaSO_4$ & $Al(OH)_3$ may be used effectively to desensitize Bz_2O_2 if pptd directly on the particles from salt soln] 13)R. Knoch & E.S.Stanley, USP 2454254 (1948) & CA **43**, 1807 (1949) [The stability of org peroxides (such as acetyl benzoyl peroxide) is greatly increased by prepg a soln of the peroxide in at least 25% by wt of the esters of phthalic acid (such as di-Me or de-Et phthalate)] 14)H.A. Aaronson, "Desensitization of High Explosives by Waxes, Semi-Plastic RDX Compositions", PATR **1761** (1950) (A number of commercially

available waxes were studied as desensitizing binders for use in RDX A-3 Type compns) 15)A.L.Forchielli, Ibid, PATR **1787** (1950) [The following compns, representing modifications of standard Comp A-3 (91/9-RDX/wax) and Comp A-4 (93/7-RDX/wax), were developed: a)Comp A-3 97.5 & talc (or metallic stearate of Ca, Li, Co, Zn or Cr) 2.5% with 0.5% of artificial graphite added) b)Comp A-3 98.0 & talc or metallic stearate 2.0% c)Comp A-3 98.0 & talc or metallic stearate 2.0% with 0.5% artificial graphite added d)Comp A-4 98.0 & talc 2.0%] 16)R.F.Preckel, USP 2648698 (1953) & CA **47**, 10852 (1953) (Desensitization of NG & other liquid nitric esters) (See in text) 17)Dynamit AG, Ger P 876979 (1953) & CA **52**, 10582 (1958) (Sensitivity to impact and to percussion of desensitized expls can be decreased further by addg emulsifying plasticizers to these expls. For example, PETN, previously desensitized with 10% of a wax-like high-melting ketone, can be further desensitized by addg tritolylphosphate) 18)S.J.Lowell, "Desensitized Torpex-Type Bomb Fillers, Properties of", PATR **2355** (1956) (Conf) 19)Fedoroff et al, PATR **2510** (1958), p Ger 47-R [German desensitized expls of WWII, as, for example: Fillers No 32 (90/10-PETN/Wax), No 33 (85/15-PETN/Wax), No 34 (70/30-PETN/Wax), No 36 (60/40-PETN/Wax), No 37 (50/50-PETN/Wax), No 38 (35/65-PETN/Wax), No 43 (Plastic expl PETN/mineral oil), No 45 (50/35/15-RDX/PETN/Wax); p Ger 48-L: Fillers No 92-HIO (90/10-RDX/Montan wax), No 109 (85/15-RDX/Mont wax), No 109 (70/25/5-RDX/Al/Mont wax)]

Désignolle (Poudres). Proplnts and expls, based on K picrate and nitrate, were patented in France and England in 1867. Daniel (Ref 1) gives the following compns for powders manufd at Bouchet in 1869:

Ingredients	Uses: Bursting chges for torpedoes & shells	Cannon Proplnts: Small	Cannon Proplnts: Large	Rifle Proplnts
K picrate	50-55	16.4	9.6	22.9-28.6
K nitrate	50-45	74.4	79.7	63.4-65.0
Charcoal	-	9.2	10.7	7.7- 6.4

Pérez Ara (Ref 2) gives for cannon proplnt: K picrate 9.16, K nitrate 80.74 & carbon 11.90% and for rifle: K picrate 22.28, K nitrate 70.62 & carbon 7.10%

Refs: 1)Daniel (1902), 189 2)Pérez Ara (1945) 223

Désignolle & Casthelaz Powder. See Casthelaz & Désignolle Powder in Vol 2, C84-L

Desintegrating Bullets. These bullets have their interiors formed of a hard metal (such as Zn) interspersed in a core of soft metal (such as Pb) and the surfaces are a more densily compressed mixt of the metal particles capable of being worked

Compare with **Bullets, Dumdum** and **Bullets, Frangible** in Vol 2, p B331-R

Ref: G.A.Foisy, USP 2105528 (1938) & CA **32**, 2358 (1938)

Desmaroux Stability (Heat) Test. Place a 10g sample of expl or proplnt in a glass tube 10cm high & 3cm diam, provided with a ground glass stopper and a capillary outlet leading to a mercury manometer. Then the tube is evacuated and heated to 75° or 108.5°, depending on the substance under test. The rate of decomposition is measured by the rise of pressure in the manometer

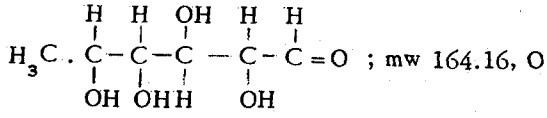
Refs: 1)J.Desmouroux, MP **22**, 245 (1926) 2)Reilly (1938), 90 3)Clift & Fedoroff, **4** (1946) 22

Desorption. The reverse of Adsorption which is described in Vol 1, p A105-L

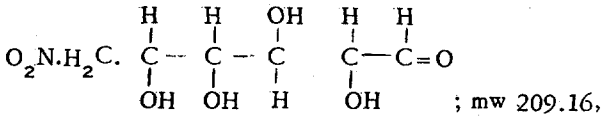
Desoxy. A prefix indicating the removal of oxygen. *A desoxy compound* is an org compd in which the hydroxyl group has been replaced by hydrogen. It has been proposed to give such

compds the ending -ane, thus desoxy-quinine becomes quinane, etc
Ref: Hackh's (1944), 260-L

6-Desoxy-D-glucose (called d-Glucomethylose or d-Chinovose in Ger),



; mw 164.16, O 48.73% (Ref 1); may be considered as the parent compd of its nitro deriv, although not used to prep it:
6-Nitro-6-desoxy-D-glucose,

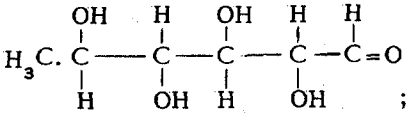


; mw 209.16, N 6.70%; wh crysts (from n-butanol + n-dibutyl ether), mp 156-57°; shows mutarotation; was prepd by condensation of 1, 2-acetone-D-xylo-trihydroxyglutaric dialdehyde (made from monoacetone -D-glucose by oxidation with Pb tetracetate) with nitromethane. The two diastereoisomers resulting were separated by a process, called desacetonation, with dil H_2SO_4 (Ref 2)

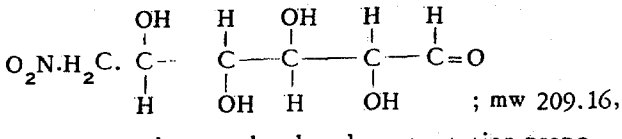
This new sugar deriv is of interest because of its remarkable tendency to form cyclic compds of the cyclohexane & benzene series

See also Mononitro deriv of Desoxyinositol
Refs: 1)Beil **31**, 63 2)J.M.Grosheintz & H.O. L.Fischer, JACS **70**, 1476-79 (1948) & CA **42**, 7253-54 (1948)

6-Desoxy-L-idose,



; mw 164.16, O 48.73% (Ref 3); may be considered as the patent compd of its nitro deriv, although not used to prep it
6-Nitro-6-desoxy-L-idose,



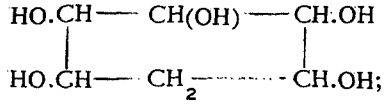
; mw 209.16, N 6.70%; resinous, sl colored mas; rotation props varied according to the method of drying; was prepd as described under the 6-desoxy-D-glucose nitro deriv and separated from the mixt by desacetonation with H_2SO_4 (Ref 2)

This new sugar deriv is of interest because of its tendency to form cyclic compds

See also Mononitro deriv of Desoxyinositol
Refs: 1)Beil - not found 2)J.M.Grosheintz & H.O.L.Fischer, JACS **70**, 1476-79 (1948) & CA **42**, 7254 (1948) 3)G.Charalambous & E.Percival, JCS **1954**, 2447 & CA **49**, 8820 (1955)

Desoxyinositol and Derivatives

Desoxyinositol; Quercitol; or 1, 2, 3, 4, 5-Cyclohexanepentol,



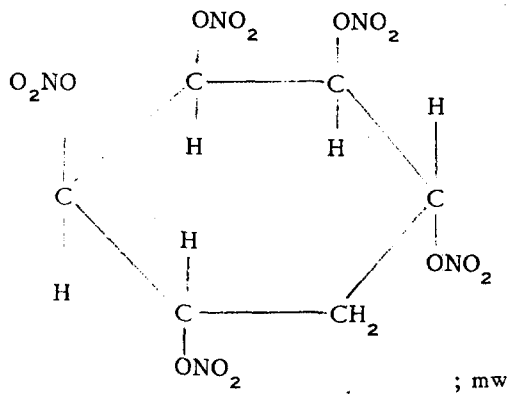
mw 164.16, O 48.73%; col crysts, mp 215.5-16.5°; can be synthesized from meso (or i) -inonsitol via rac-epi-inosose. Although the yield is low, the method is simple, direct, and uses relatively inexpensive materials. Details of the method of prepn are given in Ref. Posternak (Ref 2) reports the prepn by hydrogenation of inosose in the presence of H_2SO_4

Desoxyinositol pentaacetate & pentabenzoate were prepd as confirmation of structure
Refs: 1)Beil - not found 2)T.Posternak, Helv **24**, 1056-57 (1941) (in French) & CA **36**, 2257 (1942) 3)E.L.May & E.Mossetig, JOC **14**, 1137 (1949) & CA **44**, 3916 (1950) 4)ADL, Synthesis HE's, 2nd Rept (1951), 249-53 [Compd called DL-epi-quercitol by B.Magasanik & E.Chargaff, ACS 114th Meeting, Abstracts (1948), p 350]

Mononitrodesoxyinositols, $C_6H_{11}NO_7$; mw 209.16, N 6.70%. A mixt of nitrodesoxyinositols was obtd by cyclization of 6-nitro-6-desoxy-D-glucose, of 6-nitro-6-desoxy-L-idose; or of an equimolar mixt of the two using very small amts of alkali:
Nitrodesoxyinositol I, crysts (from dioxane), mp 147-48°; insol in acet
Nitrodesoxyinositol II, crysts (from abs alc + eth), mp 185-86° dec;
Nitrodesoxyinositol III, crysts , mp 215° dec; (from ethanol) 212-13° dec; and (from dioxane), mp 218-19° dec

Derivs of Mononitrodesoxyinositols are given in Ref 2
Refs: 1)J.M.Grosheintz & H.O.L.Fischer, JACS **70**, 1479-84 (1948) & CA **42**, 7254 (1948) 2)B.Iselin & H.O.L.Fischer, JACS **70**, 3946-47 (1948) & CA **43**, 1336 (1949)

Pentanitrodesoxyinositol or Desoxyinositol Pentanitrate,



; mw 389.52, N 18.93%; OB to CO_2 -2.1%; col crysts (from abs alc), mp 138-40° dec; was prepd by addg finely powdered desoxyinositol to constantly stirred mixed acid (2 parts by wt of concd H_2SO_4 and 1 p HNO_3 of 91% strength & d 1.50). The time of addn was 5 mins and time of reaction 30 mins. The reaction mixt was poured on ice and the solid was separated by filtration. Without drying, the thoroughly washed product was recrystallized from 95% alc. Further purification was accomplished by recrystn from abs alc

This compd is considered to be an expl of considerable power

Refs: 1)Beil - not found 2)ADL Synthesis HE's, 2nd Rept (1951), 22 & 195

Destruction of Ammunition, Explosives, Propellants and Pyrotechnics. Ammunition, expls, etc which has deteriorated and cannot be economically salvaged and ammo which is dangerous to unload or a menace to life or property must be destroyed. This can be done by burning, by chemical means, by explosion (detonation) or by dumping at sea or other large bodies of water. No dumping should be done into waste places, pits, marshes, shallow streams or inland waterways

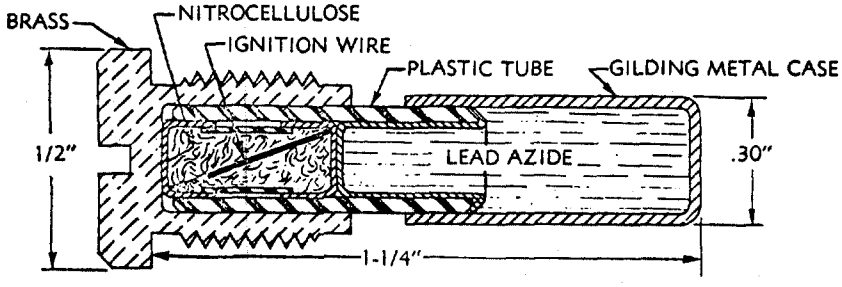
For more information on this subject, see:
1) Vol 2, pp B177-R to B178-R, "Black Powder Destruction"
2) Vol 2, pp B233-L to B234-R, "Bombs, Destruction of"
3) Vol 2, p B357-R, "Burning Ground or Destruction Site"
4) Vol 2, p B358-R, "Burning of Ammunition and Explosives for Destruction"
5) Vol 2, p C173-L, "Chemical Destruction of Explosives"
6) Vol 3, "DECOMPOSITION, DESTRUCTION and DISPOSAL of AMMUNITION, EXPLOSIVES, PROPELLANTS and PYROTECHNICS

Destructors of World War II. Destructors may be defined as explosive devices used for demolishing or destroying certain pieces of equipment under controlled condition, particularly confidential equipment (such as radio, radar, bomb sights, controls for glide bombs & torpedoes, guided missile controls, etc), the nature and operation of which it is desired to keep from the enemy. Destructors are tailor-made units, usually designed after the equipment to be destroyed is either already in production or practically ready for production. They are essentially metal cases contg an explosive or pyrotechnic charge, an initiating device (such as Bickford fuse, fuze, igniter or primacord), and certain auxiliary devices. There is a wide variation in sizes and shapes of destructors. Among about 20 US types existing in 1947 (15 of which were designed since 1940 at Pic Arsn), the smallest one consisted of a single unit about the size of a 1/4 inch fillister head machine screw, whereas the largest consisted of many parts and weighed 15 lbs. The smallest destructor was designed to mutilate the tubes and circuits in radio equipment, whereas the largest one to mutilate the vital parts of control equipment on guided missiles. Destructors can be operated either manually, so that the operator can close a switch, pull a friction wire, or push a lever at the proper time; or automatically, so that when a plane makes a crash landing with all occupants incapacitated, the destructor will respond by closing an inertia switch. Standard bomb fuzes, special bomb fuzes, and standard 2.5-lb Tetrytol Demolition Blocks are used for guided missile, control unit destructors. The fuzes are sensitive to impact and non-delay in action. These two features are required in order to eliminate the possibility of the fuze being disconnected from the expl chges on the first impact of the missile

The first destructor designed and manufd at Pic Arsn was used to destroy the Norden Bomb Sight to prevent its capture and reproduction by the enemy. The description of this destructor

and its schematic view are given in Ref 2, pp 2-3. It consisted of a 1/2-inch thick cylinder loaded with fast-burning pyrotechnic compn and a fuze which could be fired manually by trigger action or by inertia. The chge produced a hot flame jet of sufficient heat intensity to penetrate a 1/4-inch metal wall of the bomb sight housing and then melt in the inside the important units, thus destroying the device (Ref 3, p 2)

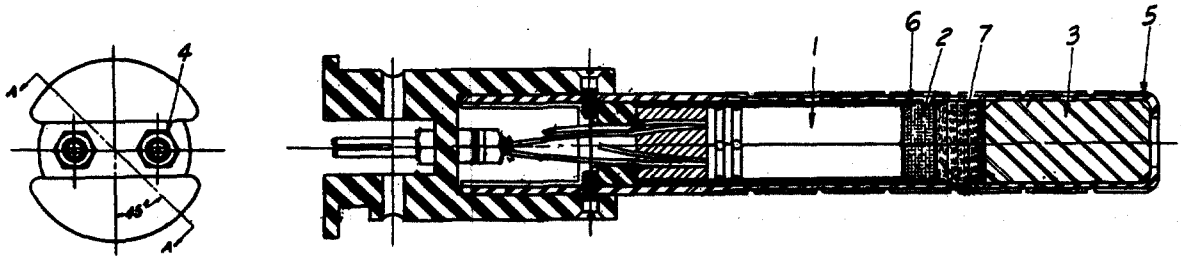
The first radio equipment destructor, known now as *ANM1 Destructor,* was developed in US during the latter part of 1940. It consisted of a small tubular-type metallic container, 1-1/4" long and 0.5" in diam which was screwed into a threaded adapter or well in certain radio



AN-M1 Destructor

equipment by means of the screw driver slot. The head or ignition end contained a NC chge. It was fired by a resistance wire, when the circuit was closed either manually, or by an inertia switch. The tube contained a LA chge having the exact amt of expl force required to destroy the vital parts of the radio without being harmful to nearby personnel. The head and expl chge case were separated by a plastic insulator tube in order to direct the flow of current thru the resistor wire. The radio equipment destroyed by this device was the one used in an airplane which received and/or transmitted an automatically coded signal to announce and guide the approach of the airplane to its home station (ship or aerodrome). Since knowledge of details of such radio equipment by an enemy would permit counter measures, it was necessary to provide means of destroying its identity, if a plane should be forced or shot down in enemy territory. Several destructors could be connected in parallel in each set (Ref 2, p 371, Fig 215; Ref 3, pp 3-4; Ref 1, p 206, Fig 145)

The *AN-M2 Destructor,* used for the same purpose as the AN-M1, consisted of a sheet-metal support 5-3/4 × 3-5/8 inches on which two commercial LA-Tetryl electric detonators M1 1.5" long and 0.25" in diam were mounted and connected in parallel so that they could operate simultaneously upon application of an electric current (Ref 2, p 371; Ref 3, pp 4-5; Ref 1, p 206)

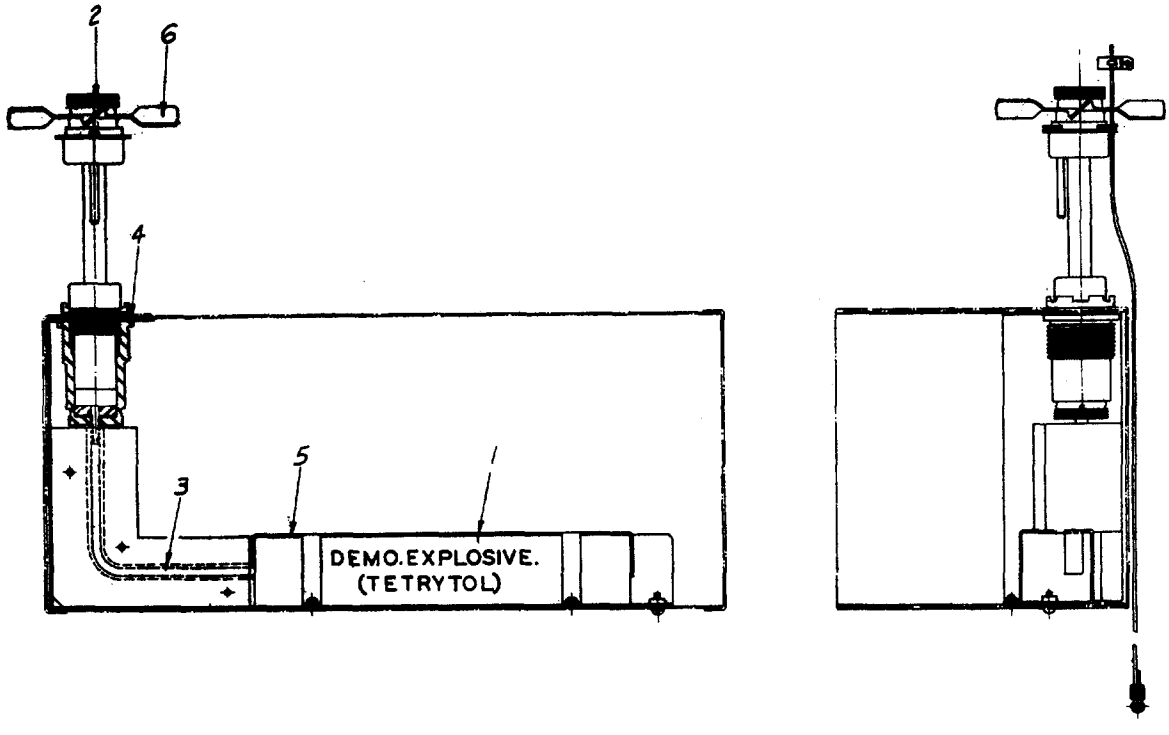


AN-M3A1 Destructor

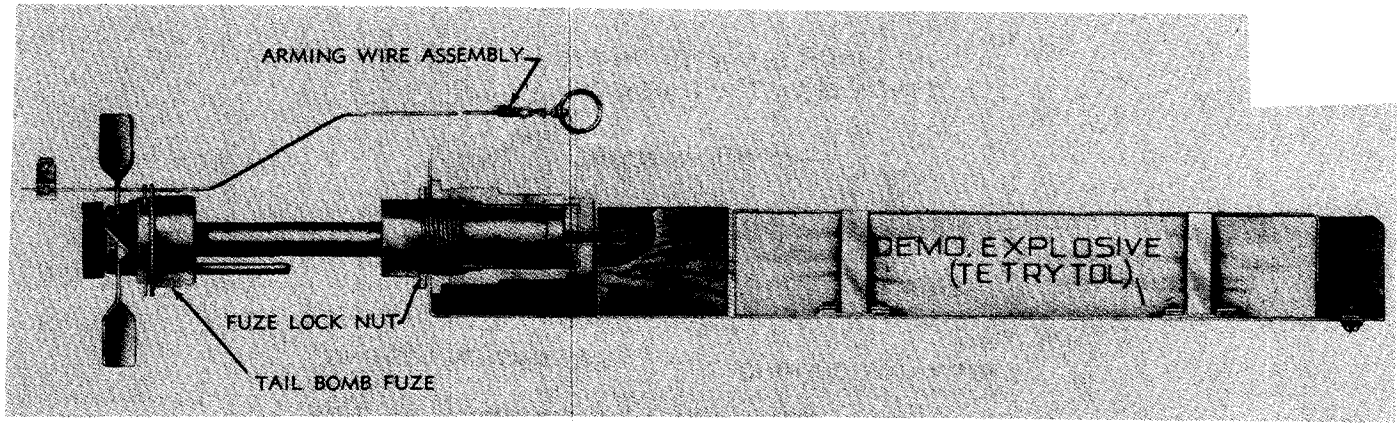
The *AN-M3A1 Destructor* was a large tubular-type device (6.2" long & 1-1/8" in diam) designed to demolish a greater number of secret parts which have more resistance than the radio parts in the devices intended to be destroyed by AN-M1 & AN-M2. Because of this, in addition to having two electric LA-Tetryl commercial detonators (1), with terminals (4), a Tetryl pellet (2), and a steel slug (3), were used. The exterior of the fragmentation tube was grooved both longitudinally and circumferentially so as to obtain the desired number and size of fragments. The slug (3) was staked in place to prevent it from damaging the Tetryl pellet during shipment, handling and vibrations of the airplane. As a further precaution against crushing and dusting of the pellet, it was encased in a plastic housing (6) with soft wads inserted betw the housing and slug (Ref 2, p 372; Ref 3, p 5 & Ref 1, p 207). See Fig on p D 93

The *M4 Destructor* was designed to destroy the equipment which controls the prefixed course of flight of a glide bomb as soon as it lands at the target, so that, if the main bomb chge does not detonate, the enemy will not learn the method of controlling the flight apparatus. The destructor consisted of a 2.5 lb Tetrytol demolition block (1) and a bomb fuze (2) mounted on a bracket (5) which positioned the destructor in the control unit. One end of primacord (3) was secured to the bottom of the fuze adapter (4) while the other end extended thru the block (1). In operation the arming wire was withdrawn from the fuze when the bomb was released from the airplane, thus allowing the fuze arming vane (6) to rotate and release the firing plunger. Upon impact, the plunger moved forward by inertia and fired a primer, which, in turn, initiated a LA relay. Then the detonator & the primacord were initiated and the block (1) was detonated, thus destroying the control equipment (Ref 2, pp 372-73p Ref 3, pp 7-8 & Ref 1, p 207)

The *M5-Destructor,* used for destruction of control equipment of a glide torpedo, consisted of a tubular-shaped Tetrytol demolition block and a tail bomb fuze both mounted on a bracket (Ref 2, p 373 & Ref 1, p 208). See Fig on p D95

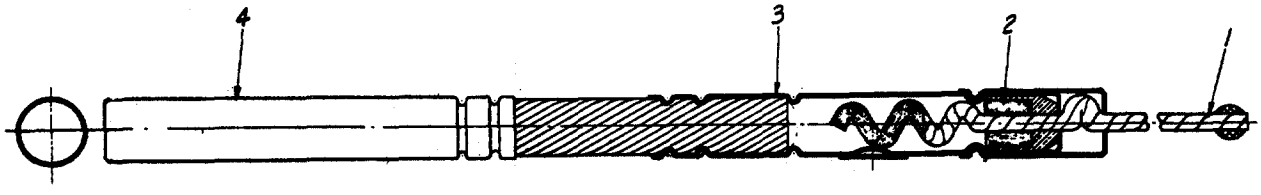


M4 Destructor



M5 Destructor

The *M6 & M7 Destructors,* used for destroying radio equipment carried by a paratrooper, if he were in danger and wished to dispose of his equipment, were identical in design, except that the M6 was slightly larger and more powerful than the M7. Both were manually operated, tubular-shaped devices contg in the cup (4) the chge of LA & Tetryl. They were initiated by a sharp pull on the firing wire (1) which withdraws the phosphorus -coated twisted end of a wire thru the friction sensitive compn (2), thus producing a flame. This ignited one end of the safety fuze (3) which provided 5 to 8 seconds delay betw firing and detonation, thus allowing a safe time interval for the operator to move from immediate vicinity of the destructor (Ref 2, p 372; Ref 3 and pp 6-7)



M6 or M7 Destructor

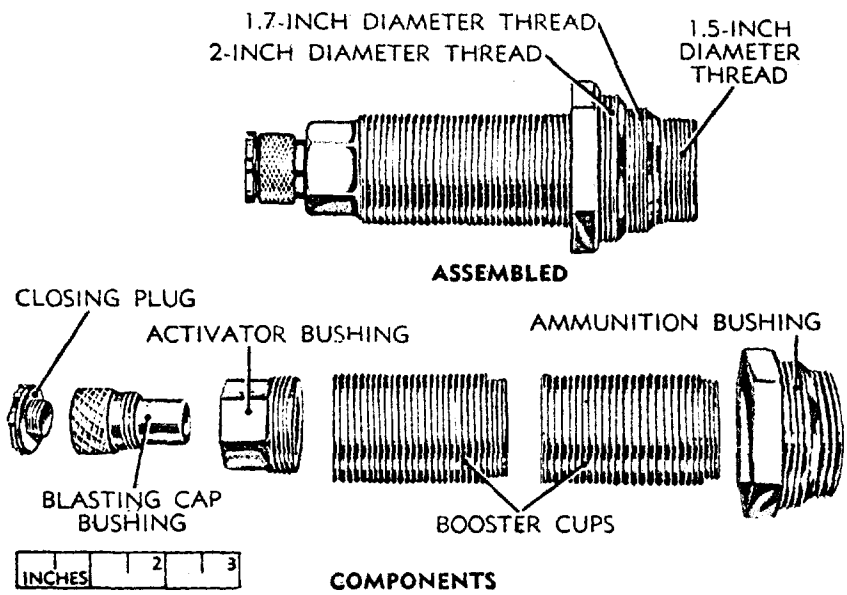
The destructor used on a target-seeking bomb, especially that used against power plants, is described in Ref 3, pp 8-9

The destructor to prevent enemy duplication of the instrument, which was used to make night signals from ship to aircraft and from ground to aircraft visible only with the aid of this secret instrument, is described in Ref 2, pp 9-11

The US *Universal M10 Destructor* is a HE chge initiated by a blasting cap or mine acutator and std firing devices. As shown in Fig, this destructor is essentially an adapter booster, with a threaded bushing that will fit std right -hand threaded fuze cavities. It is used in projectiles, bombs, improvised mines, booby traps & demolition chges (Ref 7). See Fig on p D96

See also Refs 4, 5 & 6 for info on foreign destructors. See Destructors under Demolition Materials in this Vol, p D62-R

Refs: 1)Anon, "Ammunition, General", US War Dept Tech Manual TM **9-1900** (1945), pp 204, 206, 210 2)Ohart (1946), 370-74 3)J.H.Robinson, "Destructors and Special Devices", Lecture delivered at Picatinny Arsenal (16 May 1947), pp 1-11 4)Anon, "German Explosive Ordnance (Bombs, Fuzes, Rockets, Land Mines, Grenades and Igniters)", TM **9-1985-2** (1953), pp 177-192 (Mechanical & electrical anti-withdrawal fuzes) 5)Anon, "Japanese Explosive Ordnance (Bombs, Bomb Fuzes, Land Mines, Grenades, Firing Devices, and Sabatoge Devices)", TM **9-1985-4** (1953), 151-54 (Anti-withdrawal fuzes) 6)Anon, "Comprehensive Classified List of Government Explosives", Admiralty Rept **BR 819 (1B/54)** (1955), pp 41, 42 & 49 (British destructors) (Conf) 7)Anon, Ammunition General, TM **9-1900** (1956), p 267 (M10 Destructor)



M10 HE Universal Destructor

DETA. Abbr for Diethylene-triamine. See Bis(aminoethyl)-amine or 1, 4, 7-Triazaheptane in Vol 2, p B128-L

"Detaclad" Explosion Bonded Clad Metals. These products derived from DuPont research consist of two or more flat metals joined metallurgically to yield clad plates fully meeting ASTM & ASME codes. The expln-bonding process permits manuf of a wide variety of "Detaclad" products, ranging from a combination of more conventional metals and alloys, such as stainless steel and nickel on carbon steel, to noble metals on steel, stainless steel or other metals

One interesting and widespread application of "Detaclad" is the prepn of expln-bonded strips for the minting of new coins by the US Treasury Dept

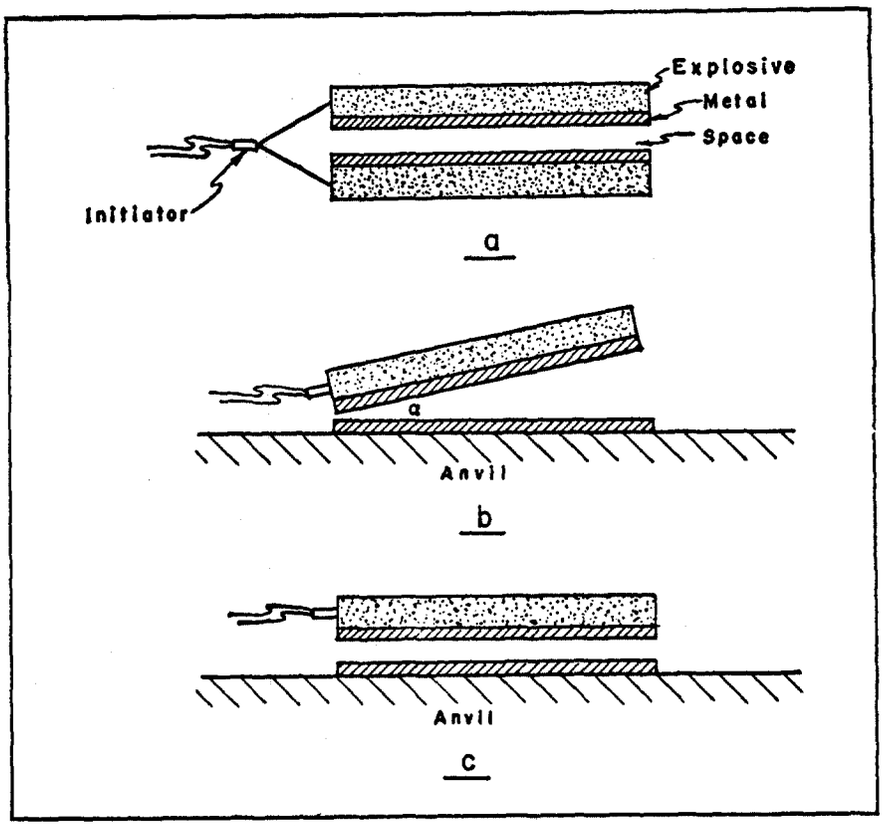
Ref: Blasters' Hdb (1966), 15

Detaclad Process or Explosive Cladding. (Explosive Bonding). This process developed in US ca 1960 consists of bonding dissimilar metals into "clad" plates by using precisely controlled detonation of large quantities of explosives. *Clads* are essentially structures in which one metal is metallurgically bonded to another as a "skin"

Briefly, the method consists of placing one metal plate on a flat steel block (anvil) and installing the plate to be bonded above the first one, leaving a small air space between the two. The surfaces of the plates facing each other must be thoroughly cleaned. Then a thick layer of an explosive, provided with an electric initiator, is spread over the entire surface of upper plate. The detonation of expl charge produces bonding of dissimilar metals, which cannot be joined by welding or soldering. See Fig as in **b** & **c**. For example, a stainless steel plate can be bonded by this method with metals like titanium, or tantallum, thus producing laminates which combine strength with corrosion resistance or other special properties. Aluminum can also be bonded with various metals by using this method

Instead of using an anvil, the two plates can be bonded by arranging them in parallel, with an air space between, and placing expl charges on the outer surfaces of both plates (See Fig as in **a** on p D 97)

Ref: A.H.Holtzman & G.R.Cowan, Ordn **50**, 536-39 (March-April 1966) (Explosive Cladding)



Method of Producing Explosive Clads

Detacord. It is a DuPont detonating fuse which has less expls in the core and has a lighter weight covering. However, it is handled on the job and hooked up in the same manner as Plain "Primacord". Their average velocities of detonation are the same (21000 ft/sec), but diam of Detacord is smaller (0.175 inch vs 0.198 for Plain "Primacord".) The principal use of Detacord is for detonating large numbers of relatively small charges simultaneously, as for example, in short, small diam holes on a pipeline work and for secondary blasting in quarries and open pit mines. It is also applicable in underground mines for long hole work and for chute blasting. Detacord is not a satisfactory initiator for "Nitramon" primers, nor should it be used for down lines in primary blasts except in certain instances for small diam holes (Ref 1). It seems that Detacord is replaced now with "**E-Cord**", which is listed here under Detonating Cord (Ref 2)

Refs: 1)Blasters' Hdb (1958), 114 2)Blasters' Hdb (1966), 104

Detaflex Flexible Cord Explosive. Detaflex is the cord version of DuPont *Detasheet* (qv) and exhibits the same properties of flexibility, insensitivity to shock, elasticity and resistance to moisture as the sheet material. It meets the requirements of US Military Specification (Ref 1). The cord is available in diameters from 0.084" to 0.665 both in uncountered and nylon countered forms. It is particularly useful in underwater demolition work (Refs 2 & 3)

The Spec requirements and tests include (Ref 1):

Properties	Flexible Explosives Grade A	Grade B
Form, specified as	Cord or sheet	
Using service	Navy	Army
Color	Olive drab	Olive drab
Change in color, at 160°F	None	None
Density, g/cc (min)	1.4	1.4
Rate of deton		
Max, m/sec	7500	7500
Min, m/sec	6600	6600
Drop test sensitivity		
0.06" mild steel cylinder 5" diam × 12" long, chge press loaded	No blast or expln	-
ERL Bruceton impact Chge 35mg 2.5 kg weight	No more sensitive than Comp B	-
Friction sensitivity		
Sign of decompn	None	None
Electrostatic sensitivity		
Expln or fire	None	None
Bullet impact 30 caliber M2 ball at 40 ft		
Explosion	None	None
Bar drop impact 10 lb bar of 1/2" diam from 15 ft height		
Explosion	None	None
Flame sensitivity		
open fire, expln	None	None
Exudation at 60°		
Maximum, %	0.10	0.10
Vacuum stability 5 g sample at 100°		
Maximum, mm	5	5
High temp storage at 160°F		
cracks, max depth, inches	1/16	1/16
Low temp storage at -40°F		
cracks, max depth, inches	1/16	1/16
Detonation continuity		
Sheet 0.08" × 3" × 10" Under 10000psi hydrostatic load	Uniform	-

Refs: 1)US Specification **MIL-E-46676A** (MU) (17 April 1964) (Flexible Explosive) 2)"DuPont Explosives Specialties Selector", Pamphlet No **A-42939**, supplied on request by the DuPont Co 3)DuPont Co's advertisement in Ordn **50**, 583 (1966)

Detasheet Flexible Sheet Explosives. Detasheet is a high explosive developed and manufd by the DuPont Co. Composed of integral mixture of PETN and elastomeric binder, Detasheet is flexible over a wide range of temps, easy to cut and handle, yet, retains most of the expl props of PETN alone. In addition, it is safe to use and is completely water-proof, thus not requiring protective covering

There are two major types of this expl:

a) *Detasheet A* - commercial variety; it contains 85% PETN

b) *Detasheet C* - military variety; it contains 63% PETN & 8% NC. The A variety, colored red for identification, is available in sheets of various thicknesses, whereas the variety C, colored olive is manufd in sheets, ribbons, cords and many other complex forms. The formulation and props of variety C meet the requirements of US Military Specifications (Ref 1)

Properties of varieties A & C are as follows (Ref 5, p 6)

Properties	A	C
Detonation Rate, m/sec	7000	7200
Density, g/cc	1.48	1.48
Flexibility Range, °F	0 to 130	-65 to 160
Storage Life at Ambiant Temperature,	1 year	Indefinite (1)
Thermal stability, °F		
24 hours	250	250
1 hour	275	275
Hot Bar Ignition Temp, °F		
Instantaneous	565	565
5 seconds	456	456
15 seconds	380	380
30 seconds	353	353
Impact sensitivity, (5 kg drop test)	22"	56+"
Bullet, fired from cal 30 M1 rifle at 40 ft	No detonation	
Static Sensitivity, joules	>0.9	>0.9 (2)
Min Tensile Strength, psi (ASTM D-1566-60T)	-	30 (3)
Range of Percent Elongation (ASTM- D-412-61T)	-	15 to 150
Minimum Propagation Thickness, inch		
Sheet (steel back -up plate)	0.042	-
Sheet (unconfined)	0.080	0.025
Cord	-	0.060 (4)

(1) No change in flexibility or performance after more than 4 years

(2) Failed to detonate at 30Kv discharged thru a capacitance of 2000pf

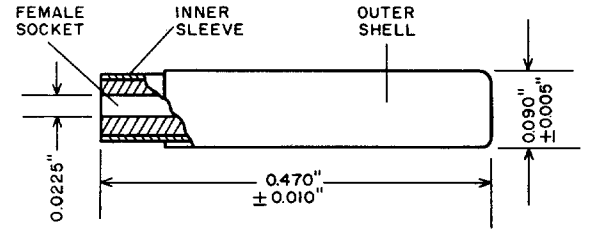
(3) Will creep to rupture under continuous stress as low as 1.5psi. The reported tensile value was developed at 20 in/min crosshead travel

(4) The cord diam corresponds to ca 15 grains per foot explosive

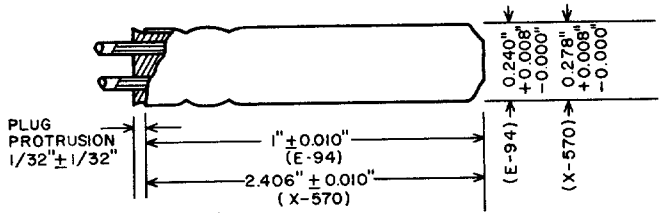
Detasheet can easily be cut to shape using a fixed blade knife or type metal die on a non-metallic anvil. Cutting with ordinary scissors is not recommended because any situation subjecting small particles of expl to a grinding or shearing action should be avoided. Detasheet has been designated by the ICC as Class A Explosive and the rules and regulations for transportation of explosives (49CFR 71-90) apply

Detasheet flexible explosives are covered by the following composition of matter patents assigned to E.I. duPont de Nemours & Co: USP 2999743 (1960) by C.J.Breza & C.O.Davis [CA **56**, 1663 (1962)]; and USP 2992087 (1961) by H.H. Fassnacht, R.W.Felch & C.D.Forrest [CA **55**, 26447 (1961)]

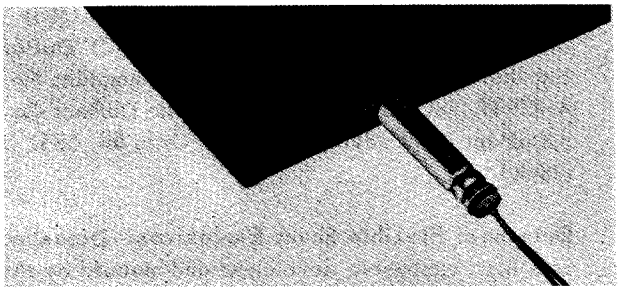
For consistent initiation of Detasheet A, an M-6 US Engineers' Cap (cong 13.5 grains PETN) is recommended. Detasheet C is reliably initiated by a detonator of No 8 strength (6.9 grains PETN) or greater. Three specific DuPont blasting caps, designed to provide additional safety to personnel, are recommended for initiation of Detasheet C. The caps are: 1) DuPont E-94 (contg only 2 grains PETN); 2) DuPont X-570-similar to E-94 but offering additional protection; and 3) DuPont *Minidet*, X-549C, a miniature blasting cap contg only 20mg of expl (Ref 3, p 9)

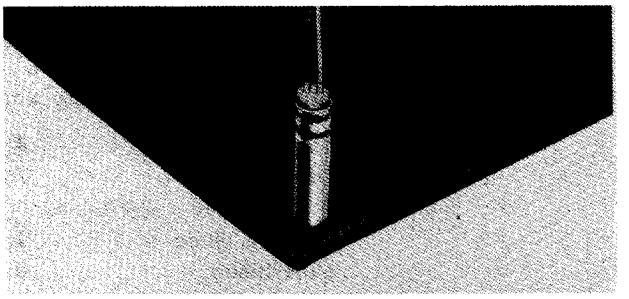


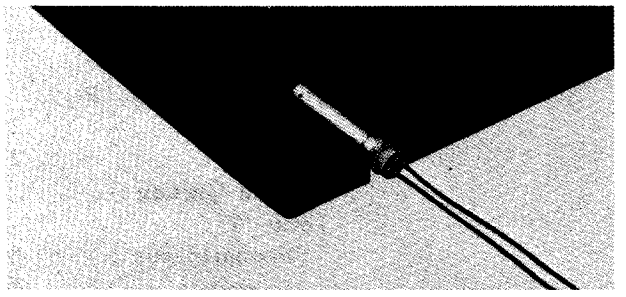
Special DuPont Blasting Cap X-549C 0.470" × 0.090" Plug-in Type



Special DuPont Blasting Caps E94 1" × 0.240" and X-570 2.406 × 0.278"







Initiation of Detasheet C

The illustrations demonstrate three proper methods to initiate "Detasheet" with the recommended detonators. Masking tape or some similar adhesive material should be used to secure an intimate bond between the detonator and the explosive

Note that in all three cases shown the entire butt end of the detonator is in contact with the explosive. Initiation techniques in which this is not true, such as placing the cap lengthwise on top of the explosive, are not recommended

Where very thin sections of "Detasheet" are used, a small additional thickness of "Detasheet" should be bonded to the larger section at the point of detonator contact to insure receipt of the full force from the detonator

Ref 2 describes a Detasheet flexible expl, designated as *EL-506*, which can be cut to fit any configuration. This expl is reported to meet the requirements of Ref 1 listed further. It detonates under water w/o the use of a protective covering; it is extremely insensitive to shock, such as by bullet impact, yet a std blasting cap detonates it. In tests it was fired at pressures of more than 11000psi, equivalent to depths of more than 2500 inches. Detasheet EL-506 was stored at temps from -40°F to 165°F and retained its flexibility under these conditions

Principal applications of Detasheet expls include: field demolition, underwater explosions, seismic prospecting and metal cutting and hardening. For example, manganese (Hadfield) steel can be hardened by inducing a shock wave in the metal thru detonation of Detasheet placed directly on the metal's surface (Ref 5, p 5)

Excess Detasheet explosive may be destroyed by detonating scraps as a unit or using established practices to burn HE's (Ref 5, p 10)

US Military Specification requirements and tests include (Ref 1) those listed under Detaflex Flexible Cord Explosives

Refs: 1)US Military Specification **MIL-E-46676A** (MU) (31 Oct 1962) (Flexible Explosives) 2)DuPont Co's Advertisement in Ordn **48**, 393 (Jan-Feb 1964) 3)DuPont Co's Advertisement in Ordn **50**, 583 (May-June, 1966) 4)"DuPont Explosives Specialties Selector", Pamphlet No **A-42939**, supplied on request by the DuPont Co 5)"DuPont Detasheet Flexible Explosive", Pamphlet No **A-45322**, supplied on request by the DuPont Co

Detectors. See under Chronographs in Vol 3, p C306

Detent and Detent Lock. *Detent* is a stop, or checking device, as a pin, lever, pawl, etc used on racks or ratchet wheels to sustain loads, or in time pieces in connection with a spring. *Detent Lock* is a lock with a notched bolt into which slides a detent operated by lug, or a knob, so as to retain the bolt in extended, or retracted position

Ref: Anon, "Stress Analysis of Detents", **TA 5-0003**, Watertown Arsenal, Nov 1951, p 6

Detergency and Detergents. Accdg to definition given in Kirk & Othmer (Refs 1 & 6) the term *detergency* in technological usage means "cleansing the surface of a solid object by means of a liquid bath. Furthermore, it implies that the cleaning process involves a physicochemical action other than simple solution". Detergency is generally considered to mean an unusually enhanced cleaning effect of liquid bath caused primarily by the presence in the bath of a special agent *detergent*. The term detergent formerly signified a substance or mixt that had cleansing action due to a combination of properties including lowering of surface tension, wetting action, emulsifying & dispersing action and foam formation. Ordinary soap is a good detergent but in hard water it percipitates Ca and Mg soaps; in acid solns soap is decompd and ppts as a free acid

In modern terminology, the word detergent refers to a synthetic variety and is not derived from natural fats and oils as are soaps

Synthetic detergents (syndets) belong to the group of *surface active agents* [which are substances which affect (usually reduce) surface tension when dissolved in water or in water solns] and have structurally unsymmetrical molecules contg both hydrophilic, or water-soluble, groups and hydrophobic, or oil-soluble hydrocarbon chains

Accdg to Ref 5, p 344, there are three types of synthetic detergents:

a) Anionic detergents. These include: alkyl-aryl sulfonates (such as sulfonates of dodecyl benzene). sulfates of straight chain primary alcohols (such as Na lauryl sulfate) and lignin sulfonate derivs. Soap is also an anionic detergent but it is not synthetic

b) Cationic detergents or invert soaps, These include quarternary ammonium halides, such as benzethonium chloride and cetalkonium chloride

c) Nonionic detergents. These do not ionize in water but acquire hydrophilic character from an oxygenated side chain, usually polyoxyethylene. The oil-sol part of the molecule may come from fatty acids, alcohols, amides or amines. Eg "Ninol" and "Surfynol"

Accdg to Riegel (Ref 2), the commercial synthetic detergents were devised originally as improved *wetting agents* (substances dissolved or suspended in water that cause water to penetrate

more easily or spread over the surface of a material to be cleaned or dyed) for textiles about to be dyed. They were intended to provide chemicals which would have all the virtues of soaps and none of its defects

Refs: 1)Kirk & Othmer **4** (1949), 938-60; A.M. Schwartz, "Detergency" (65 refs) 2)Riegel, Ind Chem (1949), 701-04 3)D.Price, "Detergents", Chem Pubg Co, Brooklyn, NY (1952) 4)W.W. Niven, Jr, "Industrial Detergency", Reinhold, NY (1955), 340pp 5)Cond Chem Dict (1961), 344 (Detergents); 1098 (Surface active agents); 1226 (Wetting agents) 6)Kirk & Othmer **6** (1965), pp 853-95; A. M. Schwartz (Detergency)

Determination of Purity of Inorganic Compounds of Ordnance Interest by Titration in Inorganic Medium. Title of PATR **1947** (1953) by Seymour Kaye

Deterioration of Cellulose. See Degredation and Deterioration of Cellulose

Deterioration of Explosives. See Degradation and Deterioration of Explosives in Storage

Deterioration of Propellants. See Degradation and Deterioration of Propellants in Storage

DeTerré patented in 1871 in England mining expls contg K or Na nitrate, sulfur and sawdust (which replaced charcoal of ordinary BkPdr)

Ref: Daniel (1902), 193

Deterrents or Moderants, called by the British **Modifiers** (Also known as **Deadeners**). The following information may be added to the brief description given in Vol 3, p C381 under Coating of Explosive, Propellant, and Pyrotechnic Compositions

The practice of using deterrents in proplnts permits not only the attainment of higher muzzle velocities & more uniform pressures along the length of the barrel, but also it reduces the erosion, and this results in longer life of the gun

Hale & Cameron of Pic Arsenal patented (Ref 1) a NC proplnt consisting of NC 75-88, TNT 10 -15 & triacetin 2-10%. The triacetin served as a deterrent and as a flash-reducing agent

Spurlin & Pfeiffer of Hercules Powder Co patented (Ref 2) a progressive-burning proplnt by surface-coating the grains with a deterrent, such as butyl ricinoleate, glycerol phthalate resin or hydrogenenated methyl abietate

Olsen et al of Western Cartridge Co (now Olin-Mathieson Chemical Corp) (cited in Ref 3, pp 328-30 and also in Refs listed in Vol 2, p B15-R) patented a unique deterrent coating process, which is used now in combination with so-called "quick" stabilization of NC. The so -called Ball Powder or Ball Grain Powder is manufd by this process (See Vol 2, p B11-R)

Aaronson et al of Pic Arsn (Ref 4) investigated and developed techniques for deterrent coatings of single-base proplnt grains by the following phthalates: dicarbitol, dicyclohexyl, dibutyl, diphenyl, dimethyl, diisobutyl, dimethoxyethyl, butylbenzil, butylcyclohexyl, di (2-ethylhexyl) and di (2-ethylbutyl). The amt of each coating required to yield acceptable ballistic characteristics was also established

The term deterrent can also be applied to substances, such as comphor oils, paraffin & vaselin, which are incorporated in formulations of expls, proplnts and pyrotechnic compns in order to reduce or to control their rate of burning. Compare with Desensitizers, p D 88-R

Coating of proplnt grains with graphite, as often practiced, serves not only as an eliminator of static electricity, but also as a deterrent

See also:

Burning and Burning Characteristics of Explosives in Vol 2, p B343-R

Burning and Burning Characteristics of Propellants for Artillery Weapons and Small Arms in Vol 2, p B346-L

Burning and Burning Characteristics of Propellants for Rockets in Vol 2, p B350-R; and

Coating of Explosive, Propellant, and Pyrotechnic Compositions in Vol 3, p C381

Refs: 1)G.C.Hale & D.R.Cameron, USP 2026531 (1936) & CA **30**, 1233 (1936); USP 2035471 (1936) & CA **30**, 3650 (1936) 2)H.M.Spurlin & G.H. Pfeiffer, USP 2187866 (1940) & CA **34**, 3918 (1940); USP 2198746 (1940) & CA **34**, 6078 (1940) 3)Davis (1943), 291, 319 & 328-30 4)H.A.Aaronson et al "Deterrent Coatings for Single-Base Propellants", PATR **2031** (1954)

Detonability of Propellants. Colloided proplnts, either single- or double-base, or composite proplnts, behave usually as deflagrating expls, but they can detonate when strongly compressed and confined. When a large quantity (such as in storage) is ignited, the deflagration may be quickly transformed into detonation. Some proplnt can detonate by impact
Refs: 1)W.H.Andersen & R.F.Chaiken, "Detonability of Solid Composite Propellants", Tech Memorandum of Aerojet-General Corp No **809** (Jan 1959), Contract NOrd 17012, Task II and NOrd 18487 [A fundamental investigation of the factors involved in the detonation and abnormal (accelerating) deflagration of solid composite proplnts is described] 2)T.G.O.Berg, "Study of Susceptibility of Solid Composite Propellants to Explosion or Detonation", Aerojet-General Corp, Final Report No **0253-05(01)FP** (April 1963). Contract NOrd 18487(FBM/Task 4) (A study of sensitivity to initiation of proplnts and expls by a modified Impact Machine)

Detonateur. Fr for Detonator

Detonateur secondaire. Fr for Booster

Detonating Cables. See under BANGALORE TORPEDOES in Vol 2 of Encycl, p B 16-R

Detonating Caps. Same as Blasting Caps described in Vol 2, p B 185-L. May also be applied to Detonators

Detonating Caps, Testing of. Haid et al (Ref) described the test in which a given amt of TNT contg 30 or 40% talc was exploded in a standard lead cap. The detonating effect of the cap was ascertained from the bulge produced by the detonation
Ref: A.Haid et al, Jahresber CTR **8**, 141-45 (1930) & CA **26**, 4474(1932)

Detonating Cord of Bofors, Examination of. This cord, known as "Bonocord" was briefly described in Vol 2 of Encycl, p B 218. Its examination consists of:
a) Appearance. Make a visual inspection of the surface and color of covering
b)Resistance to Cold. Wind spirally on a rod, 4cm in diam, appr 2 meters of the cord and store it at −25°C for 24 hrs. On unwinding at the temp, the plastic covering must not show any cracks
c) Sensitivity to Initiation. Cut a length of cord ca/m and seal off ends against water by metal caps. Wind the cord on a rod 2cm in diam and place the ensemble under a water pressure of 25 m for 24 hrs. The cord must be completely detonated by a No 8 blasting cap
d) Propagation of Detonation. Connect by a knot or clamp a length of ca 0.5m to another, either as a splice to lengthen the line or as a branch line. On initiating by a No 8 blasting cap, check the propagation of detonating wave between the two lengths
e) Velocity of Detonation. Det the velocity of deton by the Dautriche Method or by the Siemens spark Chronograph
Note: These methods for detn of vel of deton are described in Vol 3 of Encycl under Chronographs, p C 305-R and under Dautriche Method, Item H, p C 311-R
Ref: Anon, "Analytical Methods for Powder and Explosives", AB Bofors, Nobelkrut, Bofors, Sweden (1960), pp 207-08

Detonating Cords or Detonating Fuses. See Cord Detonating; Cordeau; Cordeau Fuse in Vol 3 of Encycl, p C 529-R and the following:
Addnl Refs: A)A. LeRoux, MP **32**, 99-104(1950) (Influence of manufg conditions on the characteristics of flexible Cordeaux Détonants contg PETN) B)M.Ortigues & G.Ziebelen, MP **32**, 107-10(1950) (Quality of PETN required for Cordeaux Détonants) C)McAdam & Westwater (1958). 59-60 (Detonating fuses) D)Cook (1958), 17 (Primacord)

In the DuPont's Blasters Handbook(1958), p 11-13 are described under Detonating Fuze the "Primacords" and in pp 113-14 the "Detacord" (qv). In the 1966 edition of that Handbook, pp 101-104, are described the following Detonating Fuses: *"Primacords" Bickford,* commonly known as *"Primacords"* are detonating fuses manufd by the Ensign Bickford Co of Simsbury, Connecticut, which manufs also Bickford Fuse or Miner's Safety Fuse (See Vol 2 of Encycl, p B 112-L). The core of "Primacords" consist of PETN (mp ca 280°F), which is protected against the damage and penetration of water or oil by means of a flexible sheath of good tensile strength, which may be a textile, plastic or other material. All "Primacords" have high velocity of detonation (21000 ft/sec, or nearly 4 miles/sec) and their energy

of expln is sufficient to detonate a HE lying beside it in a borehole. If a "Primacord" is attached to the first cartridge loaded into the hole, it acts as a primary agent thruout the entire length of the column of expl. In spite of high velocity, energy and brisance, "Primacords" are relatively safe to handle and use, because they are fairly insensitive to sparks, friction, stray electrical currents or shocks

"Primacords" are used principally for multiple shooting of drill holes ranging in diam from ca 3 to 4 in. For certain specialized types of blasting, however smaller holes, on the order of 1.5 in diam may be initiated in the same manner. The number of holes that can be fired simultaneously is practically unlimited

"Primacords" for commercial blasting are normally supplied in the following types
a)"Reinforced", which has an inner plastic jacket with an outer covering of textile and wax finish ave diam of cord 0.202 inch. It is a comparatively rugged general utility, all-purpose cord. It is recommended for downlines and trunklines in all but the most critical and severe blasting applications. It is also occasionally used for "coyote" (See Vol 3 of Encycl, p C 551-L) tunnel blasting
b)"Plastic Reinforced", which has an added covering of textiles, plus water-proofing material followed by an outer covering of tough plastic (ave diam of cord 0.215 inch). It is used, in certain cases, for downlines in deep holes, sleeper charges, river crossings, or wherever tensile strength and excellent resistance to side penetration by water are needed
c)"Plastic Wire Countered", which is similar to previous cord, but is further strengthened by a countering of steel wires under the outer plastic jacket(ave diam of cord 0.238 inch). It is used for downlines in rough drill holes or in any situation where extremely difficult loading conditions are encountered. It is recommended for coyote tunnel blasting and generally preferred for use with "Nitramon" and "Nitramex" expls
d)"E-Cord", which is essentially identical to Reinforced Primacord, except for a reduced expl core load. It is a lower cost, general utility cord (of ave diam 0.162 inch). It is commonly used in all types of secondary blasting, in downlines and trunklines for small to medium diam blast holes, in trench blasting for pipelines, and in long hole blasting in underground mines. Initiating strength of "E-Cord", while proportionally less than that of Reinforced Primacord, is adequate to reliably detonate cap sensitive Dynamite. "E-Cord", however, is not a satisfactory initiator for "Nitramon" primers. It seems that "E-Cord" is used for the same purposes as previously described "Detacord"
Note: The 1966 edition of Blasters' Hdb does not describe the so-called "Plain Primacord" described on p 112 of 1958 edition. This consisted of a PETN core provided only with a cotton and wax covering (ave diam of cord 0.175 inch). It was used for trunk lines and occasionally for branch lines in short drill holes; also for trench blasting for pipe lines and to some extent for seismic prospecting

Detonating Cords; US Military Requirements and Testing. The cords used for US military blasting operations (such as for demolitions, cutting charges in bombs, initiating devices, some warhead applications and for other purposes), are covered by the US Military Specification MIL-C-0017124B of 25 July 1964, which superseded the MIL-C-17124A of 11 June, 1959. These detonating cords are listed in the Table, which follows

Material Requirements:
A. PETN (applicable to Type I cords) shall comply with Spec MIL-P-387, Class 1 or 2
B. RDX (applicable to Type II cords) shall comply with Spec MIL-R-398 Type I or II, Class C
C. PVC (Polyvinyl chloride) filler used in the production of inert loaded cord (Type III) shall be of standard commercial grade and quality
D. Thermoplastic Resin, used for coating the cords shall comply with Spec L-P-390. The plastic outer covering shall be of smooth texture and, unless otherwise specified,unpigmented and colorless
Other Requirements and Tests:
a)Breaking Strength. The minimum strength is indicated in Table and any piece which breaks at a tension less than that shown, shall be classed defective. Cut from each of the spools (which can be 50, 100,500 or 1000 ft long) an 18 inch length of cord and test at 70 ± 5°F in a machine (split-barrel grips or equal), having grip 1 inch diam, minimum. There shall be ca 7 inches of cord betwn the grips and the test speed shall be 12 ± 0.5 inches per min

b) Detonation of Cords, Type I. Cut from each spool a 6 ft length of cord and attached, by means

US Military Detonating Cords

Type	Class	Minimum Wt of Explosive Core per 1000 feet, lbs[2]	Inner Construction	Finish	Diameter Inches Maximum[6]	Maximum Wt of Finished Core per 1000 feet, lbs	Breaking Strength Minimum, lbs
I	a	5 PETN	Textile	Plastic coating	0.175 ±.010	14	60
I	b	6 PETN	Textile	Plastic coating	0.216	19	175
I	c	6.4 PETN	Textile	Plastic coating	0.200 ±.005	18	175
I	d	7 PETN	Textile	Plastic coating	0.200	19	110
I	e	7 PETN	Textile	Plastic coating	0.235	22	190
I	f	12.5 PETN	Textile	Plastic coating	0.245	26	75
I	g	12.5 PETN	Textile	Plastic coating	0.270	33	190
I	h	14.5 PETN	Textile	Plastic coating	0.235	29.5	110
I	j	6.4 PETN	Textile w/plastic sheath[3]	Textile w/wax[4]	0.200 +.010 −.005	18	150
II	a	7 RDX[1]	Textile	Plastic coating	0.216	19	175
II	b	8.5 RDX[1]	Textile	Plastic coating	0.235	22	190
III	All type III detonating cord shall be inert loaded with PVC filler. Weight, construction, finish and breaking strength requirements shall be the same as indicated above for the Type and Class specified to be Inert[5]						

[1]For identification purposes, the RDX core shall be dyed pink with one percent maximum of water soluble dye suitable for combination with the ingredients of the core

[2]In order to change pounds per 1000 feet to grains per foot multiply the number of pounds by 7

[3]Textile shall be rayon

[4]Wax shall be olive drab in color

[5]Outer finish shall be blue (No. 35109 Fed Std 595) in color

[6]If not otherwise indicated

of adhesive tape, a No 6 commercial blasting cap, parallel and in close contact with dry cord. Complete deton of cord shall be produced on initiating the cap

c)Detonation of Cords, Type II. Cut a 6ft of cord and securely attach a No 6 blasting cap to a square-cut dry end of the cord using a butt joint. The end of the cap shall be in line with the cord and in immediate contact with the expl core. Complete deton of cord shall be produced on initiating the cap

d)Detonation of Cords, Type I, Class b,c & j. Tie a 6 ft length of cord snugly around a TNT demolition block (conforming to Spec MIL-T-234), with 3 turns plus a half hitch on the end leading to a No 6 blasting cap. The other end of cord shall be secured beneath all the turns. Complete deton of block shall be produced

e)Detonation Velocity of Cords (Applicable to Types I & II). Determine velocity of each 6ft length either by the Dautriche Method (See Vol 3 of Encycl, p C 311-R) or by the Counter Chronograph Method (See Vol 3, p C 309-R). The velocity shall be not less than 5900 m/sec and total length of cord shall be completely detonated. The vel of the detonating cord used as a standard for comparison shall be tested electronically

f)Detonation, Propagation of, (Applicable to Type I). Cut several 3ft lengths of dry cord and attach them by a girth hitch as branches to a long piece of the same type of cord (main or trunk line). When the main line is detonated by any satisfactory means, all branch lines shall be initiated

g)Flame Sensitivity. Cut a 6inch length of cord from each spool and spread 4oz of Black Powder (complying with Spec MIL-P-223) around and over (including the open ends) each piece of cord. Ignition of BkPdr shall produce complete deton of the cord

h)Impact Sensitivity. Place each 6 inch piece of cord on a steel anvil and drop a 25 pound steel bar with a diam of 2 inches at the striking face from a height of 16ft. Complete deton of each piece shall be produced

i)Waterproofness (Applicable to Types I & II). Immerse in water not more than 1 ft in depth, for not less than 72 hrs, 75ft of a continuous piece of 80ft long cord (if the lot is comprised of 50ft spools, two spools shall be used). The remaining 5ft of cord shall be kept dry. After completion of immersion, cut a 50ft length (including 5ft of dry end) for use as a trunk line, and ten 3ft sections for branch lines. Beginning 15ft from the dry end of the trunk line, attach, by means of girth hitch knots, the branch lines at 3ft intervals. Initiate the trunk line from the dry end and see if all lines are completely detonated

j)Flexibility (Applicable to all cords except Type I, Class C). Cut ca 12ft length of plastic coated cord with a 0.25 inch OD or less and wrap 5 times around a ¼ inch mandrel. (If the cord is with an OD greater than 0.25 inch, use the ¾ inch mandrel). Condition all samples at −65°F for 72 hrs and initiate each cord within one minute after removing from the conditioning chamber. Complete deton shall be produced

k)Flexibility (Applicable to Type I, Class C). Using the ¼ inch mandrel, tie a 12ft length of cord (0.200 inch OD) in hitches and connections normally encountered in blasting and demolition operations. Condition as above at −65°F and detonate with No 6 cap

l)Temperature Conditions (Modification described in Ordnance Corps Engineering Order 37806-S, Sheet 5, July 23, 1965). Cut a 10ft length of cord, coil it in a single circle ca 3ft in diam, and spread in a horizontal plane on a perforated stainless steel tray, placed in a special chamber. Expose the cord for 4hrs to +125°F ambient air with 360 BTU/sq ft/hr solar radiation condition. The intensity of solar radiation shall be monitored once per hr, during the 4-hr exposure period by means of the Pyroheliometer, Eppley Laboratory, Inc, Model 10, S/N 27370, Horizontal Surface Type and also using Portable Potentiometer, Leeds & Northrup, FA No Q3440, S/N, 1026920. After completion of the solar test, lower the temp of the chamber to RT (ca 72°F) and inspect the sample visually for any possible physical damage. If no damage is observed, place the cord horizontally, in the form of a loose elliptical coil ca 1ft × 2ft, into a perforated stainless steel basket which is maintained at temp of −65°F for 72 hrs in the special chamber. Then raise the temp of chamber to RT and inspect the cord visually for any physical changes, such as of color, form, dimensions, cracks in the cover, etc. The cord so exposed shall detonate completely when initiated with a No 6 blasting cap

Note: After completion of description of these tests, it was learned that the Specification MIL-0017124C is in the course of preparation. As this new Spec was not available at the Arsenal, the

Spec MIL-C-0017124B with its Engr Order 31081-S modification was used

Detonating Corks (Knallkorken in Ger). Accdg to a brief item appearing in SS **6**, 398(1911) & CA **6**, 547(1912), these devices were manufd before WWI in Sweden and used in Germany in the so-called "bicycle pistols" (Encycl, Vol 2, p B 113-L). A portion of a carton contg 1000 detonating corks, shipped from Sweden, exploded in 1911 in the Berlin Post Office, causing considerable damage. Our efforts to find the description of "bicycle pistols" and of "detonating corks" were unsuccessful

Detonating Explosives. Under this title are known expls which cannot be initiated by a flame, spark, friction or trigger impact but require the intermediate items such as *blasting caps* (See Vol 2, p B 185-L) or *detonators* (See Vol 4). Some detonating expls require, in addition, *boosters* (See Vol 2, p B 243-R). To this group belong most of the so-called **HE**'s (High Explosives). When such an expl is initiated, a detonation wave is created which passes thru the entire mass of the expl almost instantaneously (or more exactly in a matter of microseconds, 10^{-6} secs) changing it from a solid (as in case of TNT) or a liquid (as in case of NG) to a gaseous state. The sudden generation of gases and their extremely rapid expansion produce a strong shattering effect (brisance) which can overcome great resistance in their path

LE's (Low Explosives), such as BkPdr, blasting expls, loose NC's and colloided proplnts, known also as "deflagrating" expls, are distinguished from "detonating" expls by their low power & velocity of propagation, but by high "heaving" action. They can be initiated by a flame or spark but not by a stab action or by a commercial blasting cap. Some of them, however, can be initiated by a commercial blasting cap easier than some HE's, such as cast TNT, Comp C3 or Comp C4. The LE's change from a solid to a gaseous state relatively slow (in a matter of milliseconds, 10^{-3} secs). They deflagrate progressively over a relatively sustained period of time, and this action can be utilized to push, heave or shove, rather than to rend or tear, the object against which they are placed. The principal military uses of deflagrating expls are as proplnt chges for projectiles and as the powder train in a time fuse. The principal commercial uses of such expls are in blasting (See Vol 2 of Encycl, pp B 184-L & B 202-L), which includes coal mining (See Vol 3 of Encycl, pp 434-Rff)

The detonating or HE's may be subdivided into "primary (priming) expls" and "secondary HE's". The primary expls, such as MF, LA, Pb Styphnate, Tetracene, Cyanuric Triazide, etc) are used as primary charges in detonators in conjunction with secondary (or base) charges, which are usually pressed Tetryl, RDX, PETN or PA. The primary chge is initiated by flame, spark or stab action and the resulting detonation initiates, in turn, the base charge of a detonator, which is located either inside of a "main" or "bursting" charge of ammunition (such as grenade, bomb, shell, mine, torpedo, rocket head or demolition chge). The expls used for such chges are TNT, Composition B, Cyclonite, Explosive D, PA, Pentolite & Tetrytol. Pressed Guncotton was used in some countries (such as Russia) in demolition chges, Whitehead torpedoes, mines and AP shells. Secondary "detonating explosives" may be called "brisant explosives"

Some of the above HE's (especially cast TNT) cannot be initiated by a detonator alone unless its primary and base chges are very large. For such expls, an auxiliary device, called booster (See Vol 2 of Encycl, p B 243-R) is placed betw the detonator and the main chge. The expls comprising boosters are easier to detonate than cast TNT and they include pressed Tetryl, PETN or RDX. PA & TNT, in powder form, can also be used

It must be noted that detonating expls (such as NG or TNT) may behave as deflagrating ones, when unconfined and not in bulk and that some deflagrating expls (such as NC & proplnts) may behave as detonating ones when compressed and under confinement (See also Detonability of Propellants)

Refs: 1)Ohart(1946), 16-19 2)S. Paterson, "Detonating Explosive", 5th Symposium on Combustion, Pittsburgh **1954**, 672-84 & CA **49**, 16437(1955) 3)Dunkle's Syllabus', Dec 13, 1955, pp 3-4 (PicArsn Lecture Series)

Note: Analysis of Detonating and Priming Mixtures was described by C.A. Taylor & W.H. Rinkenbach in US Bur Mines Tech Paper **282**, Washington, DC(1922)

Detonating Fuses. See under Cord Detonating; Cordeau Fuse or Detonating Fuse in Vol 3, p C 529-R. See also "Primacords" Bickford and "Detacord" in Blasters' Hdb (1958), pp 110-116 and "Primacord" Bickford and DuPont "Primacord" in Blasters' Hdb (1966), 101-106

Detonating Ignition. This term, which was introduced by Ramsay & Weston (Ref), refers to initiation by a strong local expln instead of by a spark or flame. For example, a mixt of charcoal and saltpeter (without sulfur) is not explosive but will burn on contact with flame or spark but not on impact. If, however, a small amt of NG will be added and the mixt subjected to an impact, a powerful expln will result. This is because NG will detonate and the resulting hot flame will deflagrate the mixt of charcoal and saltpeter (See also under Detonating Explosives)
Ref: A.F.J. Ramsay & H.C. Weston, "Manual on Explosives", Routledge, London (1916), 80

Detonating Point. See Detonation (and Explosion) Temperature in the section on DETONATION AND EXPLOSION

Detonating Pressure. See Detonation (and Explosion) Pressure in the section on DETONATION AND EXPLOSION

Detonating Red Substance (Fr Corps rouge détonant). It is a reddish-brown, very sensitive explosive substance first found during WWI in the plants manufg TNT, on the walls of various lead vessels, such as nitrators, tanks, pumps, pipes, etc. Similar material was found on the walls of some iron vessels. The fires and explosions in several French TNT plants were presumed to be initiated by that "red substance"

Kovache & Thibon (Ref) examined after WWII some of the deposits on lead and iron, which were caused by contact of these metals with TNT and/or DNT in the presence of acids. These deposits were found to deflagrate on heating, to explode on impact, and to ignite when brought in contact with concd sulfuric, nitric or mixed nitric-sulfuric acids. These substances seemed to be similar to those found during WWI
Ref: A. Kovache & H. Thibon, MP **34**, 369-78 (1952)
Note: The deposits of red substance were observed on the walls of several storage tanks at Keystone Ordnance TNT Plant at Meadville, Pennsylvania, after the tanks were drained. Some of deposits deflagrated when they were allowed to dry in the sunny weather. The deposit was not sensitive when wet and could be easily scraped off. Some of this material was collected and investigated at the laboratory. The dried material could be ignited by flame and deflagrated by heat. When treated with water and then with alcohol, part of the substance dissolved, coloring the soln red. The residue was greyish in color and proved to be extremely sensitive to heat and impact when dried. Its behavior was typical of a primary explosive. It was tentatively named "Keystonite" and started to be analyzed. Unfortunately investigation could not be completed because the plant was closed in the early fall of 1945

Detonating Relays. These are devices for obtaining short-delay blasting in conjunction with detonating fuse. They consist of two open-end delay detonators coupled together with flexible neoprene tubing. In practice, a relay is inserted in the main line of detonating fuse which is cut at any desired point and the two ends are crimped into either end of the relay. The delay provided is 15 ± 3 millisecs
Ref: McAdam & Westwater (1958), 60

D

A cumulative alphabetical listing of items discussed in Vols 1, 2 & 3, which may not necessarily appear alphabetically in the text. NOTE: All page numbers preceded by alphabet letter **A** refer to Vol 1; pages preceded by letters **B** & **C** to **C213** refer to Vol 2; pages **C236ff** & **D1 to D108** are found in Vol 3.

A

Abbreviations, Code Names & Symbols Vol 1, Abbr 1-65; Vol 2, IX

Abbreviations for Books, Periodicals, etc. Vol 1, Abbr 66-76; Vol 2, XI

Abel's Method of Manufacturing NC. See under CORDITE C536-L

Ability to Transmit Detonation at a Distance. See CSE C390-L

Acetazidine or Azidine A24-R, A627-R

Acetoacetyldiphenylamine, Same as N,N-Diphenylacetamide

Acrylic Aldehyde or Acrolein A96-L

Action of Solvents on Cellulose Nitrate. See Cellulose Nitrate, Action of Solvents C115-R

Active Oxygen A101-L & A515-L

Actual Nitric as Nitric Acid A89-L & A90-L

Actual Sulfuric as Nitric Acid A90-R

Actual Sulfuric as Sulfuric Acid A90-L

Actuated Devices. See under Cartridges C70-R

Acyl and Aryl Derivatives of Azidodithiocarbonic Acid A632-R

Adipic Ketone. See Cyclopentanone C603-R

Aerojet Propellants A108-L

Aerojet Propellants. See under Ammonium Nitrate Blasting Explosives, High Explosives and Propellants A350(table)

Agglomeration or Caking and Its Prevention C3-R

Agglutinant or Binder B120-R

Airships and Balloons; Application in War B10-L

Aitch-Tu-Ess. See under Asbestos A494-L

Akardit. See Acardite A7-R

Aldehyde. See Acetaldehyde A14-L

Aldolcondensation Product of 5-Aminotetrazole A260-R

Alizarine, Nitro Derivatives. See under Dihydroxyanthraquinone

Alkali and Alkaline Earth Salts of Azidodithiocarbonic Acid A633-R

Alkali Amides. See under Amides A168-R

Alkali Hydroxide Reagent C405-L

Allophanamide. Same as Biuret B164-L

Allophanic Acidamide. Same as Biuret B164-L

Allylazidodithiocarbonate A632-L

Aluminized Composition A C475-L

Aluminum Acetylide A70-L

Aluminum Azide A521-L

Aluminum Carbide. See under Acetylides and Carbides A70-L

Aluminum Chlorate C184-L

Aluminum Chloride C236-L

Aluminum Chloride-Nitrobenzene Complex C236-R

Aluminum Triazide A521-L

American Ammonium Nitrate Dynamites A355 (table)

American Ammonium Nitrate Gelatin Dynamites A368 (table)

American Closed Vessels (or Bombs) C334-R

4-Amidino-1-nitrosamidino-tetrazene. See Guanylnitrosaminoguanyl-tetrazene or Tetracene under Guanylaminoguanyl-tetrazene and Derivatives

Amidocarbonic Acid. See Carbamic Acid C40-L

Aminobutane. Same as Butylamine B377-L

Aminoformic Acid. See Carbamic Acid C40-L

Ammonalmatrit. See under Almatrites A140-L

Ammon-Gelatine Dynamite and Ammon-Gelignite (British Not-Permitted (Ammonium Nitrate Dynamites) A368 (table)

Ammonium Acetate A27-R

Ammonium-Aluminum Alum A156-L

Ammonium Azide A521-L

Ammonium Azide Ammonates A521-R

Ammonium Azidodithiocarbonate A634-L

Ammonium Chlorate C184-R

Ammonium Chloride C236-R

Ammonium Chlorite C245-L

Ammonium Chromate C275-R

Ammonium Chrome Alum A156-L
Ammonium Dichromate C283-R
Ammonium Hydroxide Reagent C405-R
Ammonium-Iron Alum A156-R
Ammonium Nitrate A311 to A340
Introduction A311
Historical A312
Laboratory Preparation and Manufacture A313 & A340
Explosive and Other Properties A318
Uses A334
Analytical Procedures, General A369
Analytical Procedures, Spencer Chemical Co A379
US Specification Requirements and Tests A370
Ammonium Nitrate Blasting Explosives, High Explosives and Propellants A341 to A354
Ammonium Nitrate Dynamite (AND) (American and European Types) A355 to A356
Ammonium Nitrate Explosions, Fires and Hazards A357 to A363
Ammonium Nitrate Explosives of Spencer Chemical Co A354 (table)
Ammonium Nitrate Explosives, Tests of Spencer Chemical Co [a)Cook-off temperature b)Detonation velocity c)Wax-gap and d)Impact-friction pendulum] A354 (notes)
Ammonium Nitrate Fertilizer Grade (FGAN) A364 to A367
Ammonium Nitrate Gelatin (ANG) A367 to A368
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