DEPARTMENT OF THE ARMY TECHNICAL MANUAL **TM 9-1300-214**

MILITARY EXPLOSIVES

This copy is a reprint which includes current pages from Changes 1 and 2.

HEADQUARTERS, DEPARTMENT OF THE ARMY

NOVEMBER 1967

CHANGE No. 2

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WASHINGTON, DC, *22 June 1979*

MILITARY EXPLOSIVES

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1. Remove old pages and insert new pages as indicated below. New or changed material is indicated by a vertical bar in the margin of the page. When an entire chapter, section, or table is added or revised, the vertical bar is placed adjacent to the title only.

Remove pages	*Insert pages*
10-1 through 10-10	10-1 through 10-8
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MILITARY EXPLOSIVES

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Remove pages	*Insert pages*
i	i
1-1	1-1
2-1 and 2-2	2-1 and 2-2
2-7 and 2-8	2-7 and 2-8
3-1 through 3-7	3-1/3-8
5-11 and 5-12	5-11 and 5-12
7-3 and 7-4	7-3 and 7-4
7-63 and 7-64	7-63 and 7-64
18-1 and 18-2	18-1 and 18-2
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2. Using pen and ink, delete all references to TO 11A-1-34 on the cover and throughout the entire manual. (This is to comply with 7 July 1976 recision of the Technical Order.)

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TECHNICAL MANUAL
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HEADQUARTERS
DEPARTMENT OF THE ARMY
WASHINGTON, DC, *28 November 1967*

MILITARY EXPLOSIVES

REPORTING OF ERRORS

You can improve this manual by recommending improvements using DA Form 2028 (Recommended Changes to Publications and Blank Forms) or DA Form 2028-2 (Test) located in the back of the manual and mail the form direct to Commander, Picatinny Arsenal, ATTN: SARPA-AD-M-F, Dover, NJ 07801.

(Current as of 16 December 1976)

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* This manual supersedes TM 9-1910/TO 11A-1-34, 14 April 1955; including C1, 8 February 1956; C3, 6 November 1957; and C4, 25 July 1966.

CHAPTER 1
INTRODUCTION

1-1. Purpose

This manual is intended for use as a text for instruction or reference and as a basic source of general and technical information concerning military explosives.

1-2. Scope

a. This manual contains information and data pertaining to the chemistry, physics, manufacture, properties, identification, handling, use, inspection, preservation, storage, transportation, demilitarization, and disposal of military explosives and related substances.

b. This manual, as outlined below—

(1) Adds information on—

Explosives.
Test methods and procedures.
Methods of manufacture.
Pyrotechnic compositions.
Smoke compositions.
Tracer compositions.
Primer compositions.
Incendiary compositions.
Destruction of bulk explosives.
Packaging and marking, storage, and shipment.
Bibliography.

(2) Revises data for military explosives.

CHAPTER 2
GENERAL

2-1. Definitions

a. Explosive.

(1) An explosion, a violent bursting or expansion as the result of great pressure, may be caused by an explosive or the sudden release of pressure, as in the disruption of a steam boiler. An explosive produces an explosion by virtue of the very rapid, self-propagating transformation of the material into more stable substances; always with the liberation of heat and almost always with the formation of gas.

(2) An explosive may be a chemical compound such as TNT or nitroglycerin, a mixture of compounds such as TNT and ammonium nitrate comprising amatol, or a mixture of one or more compounds and one or more elements such as potassium nitrate, sulfur, and carbon comprising black powder.

(3) An explosive may be solid, liquid, or gaseous. TNT and nitroglycerin are example of solid and liquid explosives respectively. A mixture of 2 volumes of gaseous hydrogen and 1 volume of gaseous oxygen, when confined, is an example of a gaseous explosive. Military explosives are chiefly solids or mixtures so formulated as to be solid at normal temperature of use.

(4) The characteristic effect of explosives generally is the result of the great pressure produced when a solid or liquid is suddenly converted into a much larger volume of gas and the effective volume of this gas is greatly increased by the expansive effect of the heat liberated simultaneously. A few explosives, such as cuprous acetylide, do not form gases

$Cu_2C_2 \rightarrow 2Cu + 2C$

because the heat liberated is not sufficient to gasify the products. In such cases, the explosive effect is due to the rapidity of liberation of heat and its expansive effect on the adjacent air. A mixture of 2 volumes of hydrogen and 1 volume of oxygen, on reaction, yields only 1 volume of gaseous water, but the heat liberated makes the effective volume of the gaseous water much greater and pressure is produced suddenly.

(5) While most military explosives produce gases only, and this is necessary for maximum explosive effect, a few yield solid as well as gaseous products of explosion. On explosion, black powder yields solid potassium carbonate and sulfate as well as gases.

(6) The rates of transformation of explosives have been found to vary greatly. One group, which includes smokeless and black powders, undergoes autocombustion at rates that vary from a few centimeters per minute to approximately 400 meters per second. These are known as low explosives. A second group, which includes TNT and nitroglycerin, has been found to undergo detonation at rates from 1,000 to 8,500 meters per second. Such materials are known as high explosives. Low explosives undergo oxidation reactions or decomposition, elements or compounds being converted directly into other elements and compounds. Examples are the decomposition of typical nitrocellulose used in propellants—

$C_{24}H_{30}N_{10}O_{40} \rightarrow 5N_2 + 10H_2 + 5H_2O + 11CO_2 + 13CO$ and the oxidation reaction of black powder—in which the constitutents KNO_3, C, and S undergo a complex reaction, yielding CO_2 CO, N_2, K_2CO_3, K_2SO_4, and K_2S as the principal products.

High explosives undergo much more rapid decomposition as in nitroglycerin—

$2C_3H_5N_3O_9 \rightarrow 6C + 10H + 6N + 18O \rightarrow 3N_2 + 5H_2O + 6CO_2 + \frac{1}{2}O_2$

Some high explosives, such as nitrocellulose, can be caused by physical conditioning to be capable of functioning as a low explosive when ignited. Although the mechanisms and rates of explosion of the two groups differ greatly, human sensory perception cannot always distinguish between their actions.

(7) From the foregoing, it will be recognized that an explosive is characterized by a self-propagating reaction or decomposition with the liberation of heat and the development of a local pressure effect. An explosive, therefore, may be defined as a material that can undergo very rapid self-propagating decomposition with the formation of more stable materials, the liberation of heat, and the development of a sudden pressure effect through the action of the heat on produced or adjacent gases. It will be noted that this definition is applicable to the material in an atomic bomb, which undergoes nuclear fission.

b. Propellant. A propellant is a combustible (sometimes treated as an explosive material), that is suitable for effecting the controlled propulsion of a solid body, such as a projectile or a rocket. As disruption of the weapon must not take place and as flight of the projectile or rocket must be controlled closely with respect to range as well as direction, the explosive process must be subject to correspondingly close control. Because of these requirements, only low explosives have been found suitable for use as propellants. Because of the relatively low rates of transformation of these explosives, they do not undergo the too rapid acceleration that would cause disruptive instead of propulsive effect. Many propellants, because of certain ingredients, can be caused to undergo detonation as well as autocombustion, but acceleration of burning to the point where detonation takes place must be prevented. This is accomplished by control of the size and the form of each grain of propellant.

2-2. Development of Explosives and Propellants

a. Having its probable origin in the addition of potassium nitrate to combustible matter to form Greek fire, black powder has been known in the western world for an uncertain number of centuries. However, it was not until the middle of the 13th century that Roger Bacon described the material; which until that time had been used for pyrotechnic, incendiary, and demolition effects only. It was not until early in the following century that the monk Berthold Schwarz is credited with having invented a gun and using black powder to propel stones from it. This discovery of the usefulness of black powder for accomplishing mechanical work may be considered the real beginning of the history of explosives. For the next 500 years, black powder was the universal explosive. When ignited by a torch or spark from a flint, a loose charge of black powder above the borehole or in the priming pan of a gun served as a priming composition. The train of black powder in the borehole served as a fuse composition to advance the ignition to the propellent charge of black powder in the tube of the gun. When the projectile was of the shrapnel type, black powder in the delay fuse was ignited by the hot gases from the propellent charge, and the fuse in turn ignited the bursting charge of black powder. During this period, no essential change was made in the composition of black powder. It was not until 1425 that the granulation of black powder was accomplished and 1525 that the control of grain size by screening was reported by the French. In spite of other developments, black powder was used as a major military propellant throughout most of the 19th century.

b. The modern era of the history of explosives began in 1838 with the preparation of nitrocellulose by Pelouze by nitrating paper; but it was not until 1846 that Schönbein and Böttger independently discovered its explosive properties. Like black powder, it was at first used for both propellent and blasting purposes. In 1846 or 1847 Sobrero prepared nitroglycerin and described its explosive properties. Disastrous explosions in the course of manufacture, storage, and use retarded the development of nitrocellulose for a number of years, and the possibility of using nitroglycerin as an explosive attracted very little attention for some time after its discovery.

c. In 1863 Nobel began the commercial production of nitroglycerin, and in 1864 Schultze made the first successful smokeless powder. In 1865 Abel demonstrated the purification of nitrocellulose, so as to obtain a stable material. Because of the excessive hazard of liquid nitroglycerin, Nobel invented dynamite in 1867 and, in the same year, he invented the mercury fulminate blasting cap which assured detonation of nitroglycerin or dynamite. Simultaneously, Ohlsson and Norrbin discovered the usefulness of ammonium nitrate in what was essentially a dynamite. In 1868 E. A. Brown discovered that dry, compressed guncotton (nitrocellulose) could be detonated by means of a blasting cap and, shortly afterwards, he found that wet guncotton could be detonated by the explosion of a small quantity of dry guncotton. This latter established the principle of the booster explosive and made possible the use of blocks of wet guncotton in naval mines. The invention of dynamite with an active base by Nobel in 1869 led logically to his invention in 1875 of the nitroglycerin-nitrocellulose mixtures known as blasting gelatin and gelatin dynamite. In 1886 Turpin patented the use of picric acid as a bursting charge for shell, and it was used by the French under the name of melinite. The British began using it in 1888 under the name of lyddite.

d. Up to this time, the development of explosives had been chiefly in the direction of blasting explosives, and black powder remained the major military propellant. The partially gelatinized Schultze and E. C. powders were used in shotgun ammunition but were unsatisfactory for use in rifled guns because of too great a rate of burning. In 1884 Vieille invented Poudre B, the first smokeless powder suitable for use in rifled guns, and this was adopted immediately for use by the French Army and Navy. It was produced by gelatinizing nitrocellulose with an ether-ethanol mixture, kneading to form a jelly, rolling into thin sheets, cutting into squares, and drying. Vieille also introduced the use of amyl alcohol in this powder as a stabilizer. In 1888 Nobel invented the double-base propellent powder ballistite, which was manufactured from soluble nitrocellulose and nitroglycerin with the aid of a volatile solvent such as benzene. In the following year, Kellner and Abel developed the propellant used by the British under the name of cordite. This was manufactured from insoluble nitrocellulose, nitroglycerin, and petroleum jelly with acetone as a solvent. The colloid so formed was extruded through dies as a cord and the acetone was removed by evaporation. The petroleum jelly was found to act as a stabilizer. In the Spanish-American War, the United States forces still used black powder as a propellant for artillery, although the US Navy Powder Factory at Indian Head had started at the period to manufacture single-base powder (developed by Lt. Bernadou from the Russian pyrocollodion powder), the Army, however, was slower to accept it and did not start the manufacture of it until about 1900. They produced pyrocellulose powder by the gelatinization of nitrocellulose by means of an ether-ethanol mixture, extrision of the colloid, and removal of the solvent by evaporation. As early as 1909, diphenylamine was introduced as a stabilizer. During the same period, the use of centralite as a stabilizer for propellants was introduced in Germany and ammonium picrate was standardized in the United States as a bursting charge for armor-piercing shell.

e. The use of picric acid as a bursting charge for shell was considered unduly hazardous and difficult, and beginning about 1902, it began to be replaced by TNT, first by Germany and then by others. TNT was officially adopted in 1912 as the standard bursting charge for HE shell for the mobile artillery of the U.S. Army. World War I saw the introduction of lead azide as an initiator by the Germans, the use of amatols as substitutes for TNT by all the warring nations, and the introduction of tetryl as a booster explosive for shell charges.

f. During the next two decades RDX, PETN, lead styphnate, DEGN, and lead azide were developed as military explosives. The development in the United States of processes for producing toluene from petroleum removed limitations on the availability of TNT and permitted the development of the powerful and castable explosives composition B and pentolite. Flashless propellants were developed in the United States and low-erosion DEGN propellants were developed in Germany and Italy. Diazodinitrophenol was developed as an initiator in the United States, and tetracene was developed in Germany as a new explosive ingredient of priming compositions.

g. World War II saw the development of rocket propellants based on nitrocellulose-nitroglycerin or nitrocellulose-DEGN mixtures, and the use of nitroguanidine in artillery propellants. Haleite, a new high explosive, was developed in the United States as were tetrytol and picratol, special-purpose binary explosives used in demolition work, and in semi-armor-piercing bombs, respectively. A number of plastic explosives for demolition work were developed in Great Britain and the United States, the most important being the C-3 composition based on RDX. The discovery and great value of the blast effect of explosives led to the development of tritonal, torpex, and minol, which contain powdered aluminum and have powerful blast effects. Finally, the application of the shaped charge principle resulted in the use of special explosives of the pentolite type.

h. The expanding techniques of modern warfare lead to more and more specialized requirements for explosives and propellants. Future developments may be expected to take the direction chiefly of mixtures of currently known explosives and other materials. But in some cases, the requirements can be satisfied only by new and more powerful explosives, which presently are being sought.

2-3. Characteristics of Military Explosives and Propellants

a. High Explosives.

(1) *General.* During the past 100 years, many explosives have been studied for possible suitability for military use, yet less than a score have been found acceptable for such use and some of these have certain characteristics that are considered to be serious disadvantages. Required characteristics are such that but few explosives can meet most of them and be acceptable for standardization.

(2) *Availability and cost.* In view of the enormous quantity demands of modern warfare, explosives must be produced from cheap raw materials that are nonstrategic and available in great quantity. In addition, manufacturing operations must be reasonably simple, cheap, and safe.

(3) *Sensitivity.* All explosives are sensitive to some degree, but can be too sensitive for handling and use or too insensitive for use. It may be considered that the present standard explosives represent a range of sensitivity within which a new explosive must fall.

(4) *Brisance and power.* A military explosive must have shattering effect (brisance) and potential energy that make it comparable with or superior to other high explosives used as bursting charges; or it must have the ability to initiate the detonation of other explosives and be sensitive enough itself to be initiated by practicable means such as percussion, friction, flame, or electric current.

(5) *Stability.* In view of the long periods

of storage to which they are subjected during peace and because of the adverse conditions of storage to which they may be exposed, military explosives must be as stable as possible. Global warfare has increased the variety of adverse conditions to which ammunition is exposed and this has resulted in an increase in the requirements designed to prevent the harmful chemical and physical effects of such adverse conditions.

(6) *Density.* Loading density is an important characteristic of a military explosive, a maximum density being desirable because of the fixed volume of the space available for explosives in a round of ammunition. The greater the loading density at which a fixed weight of a given explosive is pressed or cast, the greater is its effect when detonated. However, the standard explosives having the greatest density values, mercury fulminate and lead azide, are not the most powerful standard explosives; and the selection of an explosive for a specific use cannot be based primarily upon its density.

(7) *Hygroscopicity.* Hygroscopicity, the property of absorbing moisture, can have an adverse effect on the sensitivity, stability, or reactivity of some explosives and must be negligible, if the explosive is to be considered satisfactory for military use. An exception is the very hygroscopic ammonium nitrate, which can be used in the manufacture of amatols, if kept under conditions that preclude the absorption of moisture.

(8) *Volatility.* Volatility of military explosives is an undesirable characteristic, and they must not be more than very slightly volatile at the temperature at which they are loaded or at their highest storage temperature. Loss by evaporation, the development of pressure in rounds of ammunition, and separation of constituents of mixtures are sometimes the result of undue volatility.

(9) *Reactivity and compatibility.* Minimum reactively and consequent maximum compatibility with other explosives and nonexplosive materials are necessary properties of a military explosive. As the explosive must be loaded in contact with metal or coated metal and may be mixed with another explosive or mixed with the other ingredients of a propellant, the explosive must be nonreactive therewith. Reaction, particularly in the presence of moisture, may produce sensitive metallic salts, cause deterioration and loss of power or sensitivity, or may result in the liberation of gaseous products of reaction. Compatibility is particularly important, if the explosive is to be mixed with liquid TNT to make an explosive mixture suitable for loading by casting.

(10) *Toxicity.* Many explosives, because of their chemical structures, are somewhat toxic. To be acceptable, a military explosive must be of minimum toxicity. Careful attention must be paid to this feature, because the effects of toxicity may vary from a mild dermatitis or a headache to serious damage to internal organs.

b. Propellants.

(1) *Availability and cost.* Like high explosives, military propellants must be manufactured from relatively cheap, nonstrategic materials that are available in large quantity. While nitrocellulose is not considered entirely satisfactory because of some inherent instability, it has retained its position as a general ingredient of propellants, because of the nonavailability in quantity of a more stable material having the same advantageous characteristics.

(2) *Sensitivity.* Military propellants, as used, are not unduly sensitive to

shock or friction. They must be sufficiently sensitive to ignition by flame that initiation may be positive and burning uniform.

(3) *Stability.* Stability is even more important in the case of propellants than high explosives, since practically all propellants contain nitrocellulose, which is less stable, particularly in the presence of moisture, than any of the standard high explosives, except mercury fulminate. In order to insure the maximum stability of a given propellant composition, a stabilizer is included.

(4) *Potential.* The ballistic effect of a propellant, sometimes termed its ballistic potential, is an important characteristic for which it is designed. The ballistic effect is a function of the absolute value of the quantity of heat produced and the absolute value of the quantity of gas produced.

(5) *Density.* Absolute density is seldom critical in small arms and artillery propellants, since they are seldom used at maximum density. However, this characteristic sometimes is of importance in propellants used for jet propulsion.

(6) *Hygroscopicity.* As nitrocellulose is distinctly hydroscopic, propellant compositions containing this material tend to be hygroscopic. This is an undesirable characteristic, since changes in moisture content cause changes in ballistic effect. Furthermore, the rate of deterioration of a propellant is proportional to the amount of moisture it contains.

(7) *Volatility.* Unduly volatile ingredients are not used in the manufacture of propellant compositions, but when propellants are manufactured by a volatile solvent process, the solvent cannot be removed completely by drying. This residue of volatile matter is an undesirable feature of the propellant and must be kept constant, if ballistic effects are to remain unchanged. Compositions containing nitroglycerin and made without a volatile solvent are considered to be undesirably volatile under certain conditions. In such cases, the effects on the rate of burning and its acceleration are very marked.

2-4. Classification of Military Explosives and Propellants

a. From the viewpoint of chemical composition, military explosives can be divided into three classes as outlined in (1) through (3) below.

(1) Inorganic compounds, such as lead azide and ammonium nitrate.

(2) Organic compounds that include the chemicals outlined in (*a*) through (*e*) below.

  (*a*) Nitric esters, such as nitroglycerin and nitrocellulose.

  (*b*) Nitro compounds, such as TNT and picric acid.

  (*c*) Nitramines, such as haleite.

  (*d*) Nitroso compounds, such as tetracene.

  (*e*) Metallic derivatives, such as mercury fulminate and lead styphnate.

(3) Mixtures of oxidizable materials (fuels) and oxidizing agents that are not explosives separately. Black powder and pyrotechnic compositions are examples of this class.

b. It is to be noted that nitrogen is present in practically all explosives of any of the three classes. While there are numerous chemical compounds and mixtures of compounds that contain no nitrogen and have explosive properties, from a practical viewpoint, the chemistry of explosives is that of nitrogen compounds.

c. With respect to functioning characteristics as used, explosives and propellants can be classified as described in (1) and (2) below.

(1) Burning explosives, which undergo autocombustion, such as black powder, pyrotechnic compositions, and colloided nitrocellulose.

(2) High explosives under undergo detonation.

(*a*) Initiating agents, which are caused to detonate by spark, friction, or impact and can initiate the detonation of relatively insensitive explosives. Examples are lead azide and mercury fulminate.

(*b*) Noninitiating explosives that must be detonated by an initiating agent. These are comprised as described in *1* through *3* below.

1. Booster explosives, such as tetryl and PETN, that are easily initiated and detonate at high rates, but are not suitable for loading in large masses.

2. Bursting charge explosives, such as TNT and explosive D, that must be initiated, usually by means of a booster explosive, and which can be loaded en masse.

3. Substances that can be used only as ingredients of mixtures. These include explosives that are too sensitive to be used alone, such as nitroglycerin, and substances which are too insensitive to explode when used alone.

d. It is by utilizing the special characteristics or explosives in each of these classes that it is practicable to establish the explosive train in ammunition. An example of this is the initiation by a percussion firing pin of a priming composition which, in turn, detonates a charge of lead azide and other lead charges. This initiates the detonation of a booster charge of tetryl and this, in turn, causes the detonation of a surrounding bursting charge of TNT.

2-5. Care and Precautions in Handling

a. Although explosives are hazardous materials, and there is a long history of accidents involving their manufacture and use, they can be handled and transported with safety. The excellent record of the explosives industry during the past half century, which is much better than those of some supposedly less dangerous occupations, is attributable, in part, to the use of carefully designed buildings and equipment and, in part, to the training of personnel in accordance with stringent safety regulations. These designs and regulations have the primary objective of preventing human injury or fatality and the secondary objective of minimizing property damage in case of accident.

b. In addition to explosive hazard, explosives also represent varying degrees of toxicity hazard when inhaled, ingested, or absorbed through the skin. Because of this and the fact that dust-air mixtures present additional explosion hazard, explosives should be handled under conditions of good ventilation, so that dust-air mixtures cannot be formed. Prevention of the spark discharge of static charges of electricity also should be insured by proper grounding devices.

c. The inhalation of vapors of nitroglycerin or the nitrated glycols can cause severe headache, and some individuals are sensitive to very small amounts of such materials. The inhalation of the dusts or vapors of nitro compounds such as TNT and picric acid has been known to have fatal effects. If practicable, explosives should always be handled in well-ventilated places.

d. Effects of contact of the skin with explosives vary from simple discoloration to dermatitis and from headache to poisoning, because of absorption through the skin. The hands should be dry when handling explosives, as moisture increases the absorption through the skin. After handling small quantities of explosives, the hands should be washed thoroughly, preferably with a dilute solution of sodium sulfite and then with water. If exposed to contact with explosives throughout the day, the worker should bathe and change clothes.

e. Since they represent explosion hazards because of their characteristic of sensitivity, explosives must be handled with care proportional to their relative sensitivity. For this reason, the degree of sensitivity of a new or unknown explosive should be determined before anything else is done with it. The outcome of sensitivity tests then will determine in what quantity and under what conditions the material should be handled. The more sensitive the explosive, the smaller the quantity that should be handled at one time and the greater the precautions to be taken to prevent injury and damage in case of accidental explosion. It should be borne in mind that sensitivity is a characteristic involving initiation by any form of applied energy, regardless of whether by friction, compression, shock, mechanical, thermal, chemical, or electrical sources.

f. Because of the very real danger from fragments, the more sensitive explosives should not be handled in glass vessels when dry and only behind a barricade when this is necessary. Metal vessels are dangerous, if the explosive can react with the metal to form a sensitive compound. Metal spatulas, scoops, etc., should not be used for handling explosives, rubber articles being preferable.

g. The nervous reaction of the individual working with explosives is of great importance. The extremely nervous individual is not compatible with sensitive explosives, nor is the hurried worker. A slightly phlegmatic disposition and a consciously deliberate attitude are excellent complements to close observance of standard safety regulations.

CHAPTER 3
CHEMISTRY OF EXPLOSIVES AND PROPELLANTS
(DELETED)

All data on pages 3-1 through 3-8 deleted.

CHAPTER 4

PHYSICS OF EXPLOSIVES AND PROPELLANTS

4-1. General

a. The universe consists of matter and energy distributed throughout space. Once thought to be entirely separate and nonconvertible entities, it is now known that matter and energy are the same thing in different forms. Matter can be transformed into energy in accordance with the equation—

$$E = mc^2$$

where E is energy in ergs, m is matter in grams and c is the velocity of light in centimeters per second (3×10^{10}). Such a transformation involves changes within the atom and is the basis of the functioning of the atomic bomb. It has no significance in connection with the transformation of conventional explosives.

b. Matter may be defined as that which occupies space and possesses mass. The mass of any material object is proportional to its weight. Because of the wide range of properties of various forms of matter, it is not practicable to formulate a more particularized definition.

c. Energy is the capacity to do work. Unlike matter, it does not occupy space or possess mass. Heat, light, electricity, and the motion of matter are forms of energy. The energy of a moving body is termed *kinetic energy,* while the chemical energy possessed by substances and that due to the elevated position of bodies is known as *potential energy.* Energy in the form of heat or light is classed as *radiant energy* and consists of units called photons or quanta, which correspond to the atomic units of matter. Electricity is a form of energy representing the stripping away of loosely held electrons from atoms.

4-2. Phases of Matter

a. Matter exists in three states: *solid, liquid,* and *gaseous.* These states are not separated by sharp lines of demarcation, but overlap, the particular state that the matter assumes being dependent upon the temperature and pressure to which it is subjected.

b. A solid is a body of matter that resists any force tending to change either its shape or volume and, therefore, possesses elasticity of both shape and bulk. It is characterized by small mobility of its molecules and the possession of surfaces of distinct outline on all sides. Solids are generally classified as *crystalline* or *amorphous.* A crystalline solid is one in which the atoms or molecules are arranged in some definite order, constantly repeated. As a result, when such a body is formed from its liquid condition, the solid particles tend to occur in definite geometrical forms called crystals. Amorphous solids, such as glass, are not characterized by regularity of molecular arrangement and do not form particles with geometric forms. When a pure crystalline solid is heated to a definite temperature, called the *melting point,* there is a sharp change to the liquid state. When an amorphous solid is heated, it gradually softens, becomes mobile, and eventually acquires the properties characteristic of a liquid. An amorphous solid may be considered as a liquid having great viscosity and rigidity.

c. A liquid is a body of matter that has no elasticity of shape and, therefore, takes the shape of the containing vessel. Like a solid, it resists any force tending to change its volume, but offers little resistance to a shearing force. A liquid is characterized by considerable mobility of its molecules and the possession of a distinct upper surface, usually of meniscus shape. Liquids are distinguished by the existence in their free upper surface of a characteristic known as *surface tension.* A liquid is not characterized by regularity of molecular arrangement and is unable to retain physical equilibrium when subjected to a shearing force, and tends to *flow*. When cooled, a liquid becomes a solid at a definite temperature, which is termed the *freezing point* and is essentially the same as the melting point of the solid, if the solid is crystalline. The transition is gradual and indefinite, if the solid is amorphous. If a liquid is heated under constant pressure and is a pure material, it is converted at a definite temperature termed the *boiling point,* into a gas without further change in temperature in spite of continued heating.

d. A gas is a body of matter, the atoms or molecules of which are held together only by the action of external force. It has no elasticity of shape, takes the shape of its containing vessel, and does not have the nearly flat upper surface characteristic of liquids. A gas offers resistance to a force tending to decrease its volume, but none to the increase of its volume. Unlike solids and liquids, gases have great elasticity of volume and are compressible. A gas is characterized by almost perfect mobility of its molecules and offers very little resistance to a shearing force. The distinguishing characteristic of a gas is its tendency to fill all available space (expand). Any gas mixes with any other gas with great ease and, in some cases, can pass through solids impermeable to liquids.

4–3. Mixture, Emulsion, Suspension, Colloid, and Solution

a. Solids, liquids, and gases are capable of forming a variety of mixtures within their own classes and between classes. Certain types of these mixtures are of considerable practical or theoretical importance. The mixtures are all *physical mixtures*, those involving no chemical change or loss of identity by molecules.

b. A mechanical mixture consists of two or more solids, the particles of which are distributed among one another, so that portions of the same weight taken from different locations in the mixture contain approximately the same number of particles of each ingredient. The classic example of a mechanical mixture is that of iron filings and sulfur, which can be separated into its ingredients by means of a magnet or caused to undergo chemical reaction by heating.

c. An emulsion is a physical mixture of immiscible liquids, comparable with a mechanical mixture of solids. When two such liquids are shaken or beaten together vigorously, one of these is broken up into smaller and smaller drops, which are dispersed more or less uniformly throughout the other liquid. Such a mixture is known as an emulsion. In many cases, cessation of shaking or beating results in coalescence of the droplets, because the surface tension of these makes a large drop more physically stable than a small one. The addition of an emulsifying agent results in the formation of a membrane or film around each droplet so that the emulsion is stable when shaking is stopped. In the preparation of mayonnaise dressing, oil is dispersed in vinegar by beating. An egg is added and beaten into the mixture. This acts as an emulsifying agent, by coating the oil droplets.

d. A suspension is a nearly uniform physical mixture of a solid and a relatively large amount of a liquid, the distribution of the solid throughout the liquid being maintained by agitation or by existence of the solid in very small particles that do not settle out or do so very slowly. If very finely powdered sand is added to sufficient water to form two layers and the mixture is stirred, suspension of the sand in the water is obtained. The essential difference between a suspensoid and an emulsoid is that in the latter, the particles are penetrated by the dispersion medium, becoming swollen and evel fluid in the presence of a sufficiently high ratio of dispersing medium to solid.

e. A colloid is a special case of a suspension, the particles of one form of matter being unable to settle out of another, even though of density different from that of the suspending medium. Colloidal particles are molecular aggregates, having diameters of only 0.000001 to 0.0001 mm. When dispersed in another form of matter, they remain in this dispersed condition. While foams, fogs, emulsions, and smokes represent colloidal suspensions of gas in liquid, liquid in gas, liquid in liquid, and solid in gas, respectively, the colloidal suspension of solids in liquids is of greater interest in the explosives field. In such colloids, the solid is kept in suspension by the presence of the particles of electrical charges that tend to repel the particles from each other, the preence on the particle of films of adsorbed solvent, and the continuous stirring effect on the liquid resulting from the Brownian movement of the colloidal particles.

f. A solution is the ultimate in physical mixtures, being a uniform molecular mixture of substances. It is distinguished from a colloid in that the largest individual particle present in a solution is a molecule. However, some macromolecules are too large to yield true solutions, and form a colloidal dispersion, even in dilute solutions. Although most familiar with solutions of solids in liquids, liquids and gases also may form solutions in liquids. Solids dissolve in solids, examples being certain alloys said to represent *solid solutions*. In some cases, gases dissolve in solids, an example being the solution of more than 800 volumes of hydrogen in 1 volume of palladium under certain conditions. The ability of all gases to dissolve each other has been mentioned previously.

4–4. Gas Volume

a. Avogadro's hypothesis, a basic theorem in chemistry, states: At the same temperature and pressure, equal volumes of all gases contain the same number of molecules. *Avogadro's number*, 6.02×10^{23}, is the number of molecules in a gram-molecule of matter (the number of grams equal to the molecular weight). As a logical consequence of the hypothesis, it has been found that the volume of a gram-molecular weight of gas, measured at standard temperature and pressure, is 22.414 liters. This is known as the *gram-molecular volume*. The standard conditions referred to are a temperature of 273.16° K (0° C.) (32° F.) and a pressure of 760 mm of mercury (one atmosphere).

b. The gram-molecular volume of 22.414 liters holds only for an ideal gas. Real gases have values that deviate somewhat from this; but the deviations have been determined for the common gases and appropriate corrections can be made in calculations.

4–5. Gas Pressure

a. The peculiar nature of gas pressure is best shown by the fact that this pressure is exerted uniformly in all directions. If there is considered a sealed cubical container filled with gas at standard temperature and pressure, the gas can be visualized as consisting of molecules relatively far apart and moving with great speed. This motion is in a straight line until the molecule collides with another molecule of the gas or with the wall of the container, when the molecule rebounds with such perfect elasticity that there is no loss of motion. The motions of the molecules are random, but the number of molecules is so enormous that the number moving in any one direction will be, on the average, the same as that moving in any other direction. Consequently, the number of molecular impacts on a unit area of any of the inner surfaces of the container will be the same and the pressure effect of these impacts will be uniform in all directions. In order to assist in this visualization, it is to be noted that at 0° C., molecules of hydrogen move with a velocity of ¼-mile per second and, at 20° C., a single hydrogen molecule collides with other molecules 10 million times per second. The illustration presented above is that based on the kinetic—molecular theory.

b. If a given sample of gas is maintained at constant volume and temperature, the gas maintains its pressure indefinitely. This indicates the perfect elasticity of the gas molecules. A perfectly elastic body is one that develops no friction on contact with another body and loses none of its kinetic energy by transformation into heat.

4–6. Temperature

a. For each degree centigrade change in temperature, the volume of an ideal gas changes very closely to 1/273 of its volume at 0° C. At absolute zero (0° K or −273.16° C), gas molecules theoretically are at rest and have no pressure effect. If energy in the form of heat is absorbed by the molecule, motion begins with a velocity proportional to the energy absorbed. The absorption of additional increments of energy results in corresponding proportional increases in the rate of motion of the molecule. Temperature ordinarily is thought of in terms of hot or cold, as indicated by human sense perception, but it is to be remembered that everything on earth is relatively hot on the absolute temperature scale. We may, therefore, say that the absolute temperature is a measure of the kinetic energy of the molecules of a gas.

b. The *critical temperature* is that temperature above which the gaseous form of a substance cannot be liquefied, no matter how much pressure is applied. The minimum pressure required to cause liquefaction at the critical temperature is termed the *critical pressure.* The volume occupied by a gram molecular weight of a gas or liquid at the critical temperature and pressure is termed the *critical volume.*

4-7. Gas Laws

a. Boyle's Law covers the effect of change in pressure on the volume of a gas and states that, at constant temperature, the volume of a gas is inversely proportional to the pressure. This may be stated mathematically as:

$$v \propto \frac{1}{p}$$

where v is the volume and p the pressure.

b. Charles' Law covers the effect of change in temperature on the volume of a gas and states that, at constant pressure, the volume of a gas is directly proportional to the absolute temperature. Absolute temperature is expressed in °Kelvin (° C. −273.16). This relationship is expressed as:

$$v \propto T$$

c. From these two laws it is apparent that if the volume of a gas is kept constant and the temperature is increased, the pressure will be increased. Then at constant volume, pressure is directly proportional to absolute temperature, or

$$p \propto T$$

The first two equations may be combined to give—

$$v \propto \frac{T}{p}$$

and introducing a proportionality factor—

$$v = a\,\frac{T}{p}$$

or $pv = a\,T$

If this equation is applied to one gram-molecular weight of gas, the *molar gas* constant, R, may be used instead of the constant a and V is used instead of v. The equation is made more general by introducing the factor n, which indicates the number of gram-molecular weights involved in any given case. Then the expression for the ideal gas law is—

$$pv = nRT$$

and when n=1, $pV = RT$

This expression is in agreement with Avogadro's hypothesis, according to which, equal volumes of gases at the same temperature and pressure contain the same number of molecules. The numerical value of the constant R depends upon the units used for expressing volume and pressure, and the work done in effecting change in volume can be calculated to the basis of the energy required to do such work. The numerical values for R are found to be as follows per degree per mole (gram-molecular weight):

0.08205 liter-atmospheres
8.314 joules
1.9864 calories

In calculations involving the gas laws, it is usually convenient to express R in liter-atmospheres; in electrochemical calculations, R is best expressed in joules; and in thermochemical calculations, R usually is given in calories.

d. Only ideal or perfect gases have pressure-volume relations in exact agreement with the equation pv=nRT. Deviations from this are ascribable to the electrical attractive forces between molecules and the incompressibility of the molecules as compared with the gas as a whole. The molecular attractive force increases any pressure effect and so tends to decrease volume; while the effect of the incompressibility of molecules, as such, is in the opposite direction. As a result, real gases are more compressible than a perfect gas at low pressures and less compressible at high pressures. At some intermediate pressure, the two effects counter balance and the gas follows the relation pv =RT over a small range of pressures. Examples of deviations from the simple gas laws are shown by table 4–1, in which pressure is expressed in atmospheres and volume in liters.

Table 4–1. Pressure-Volume Relations of Gases at 0° C.

Pressure, ATM.	Hydrogen		Oxygen		Carbon dioxide	
	V	V	pV	pV	V	pV
1	22.428	22.43	22.393	22.39	22.262	22.26
100	0.2386	23.86	0.2075	20.75	0.044497	4.497
400	0.07163	28.65	0.05887	23.55	0.04051	16.21
800	0.04392	35.13	0.04207	33.66	0.03779	30.23
1000	0.03837	38.37	0.03886	38.86	0.03687	36.87

e. The deviations of real gases from the simple gas laws are expressed by van der Walls' equation—

$$(P+a/v^2)\ (V-b)=RT$$

where b represents the volume occupied by the molecules of the gas and a represents the attractive force between the molecules. At low pressures the value b is the more important, but at high pressures and small volumes the value a becomes important. Table 4–2 gives a and b numerical values for gases representing products of explosion, the units involved being liters, atmospheres, moles and degrees absolute.

Table 4–2. Van Der Waals' Equation Constants

Gas	*a*	*b*
Ammonia	0.05136	4.390
Methane	0.04278	2.253
Carbon monoxide	0.03985	1.485
Carbon dioxide	0.04267	3.592
Hydrogen	0.02661	0.2444
Water	0.03049	5.464
Nitrogen	0.03913	1.390
Nitric oxide	0.02789	1.340
Nitrogen dioxide	0.04424	5.284
Oxygen	0.03183	1.360
Sulfur dioxide	0.05636	6.714

f. An equation that describes the relationships between the temperature, volume, and pressure of a gas is known as an equation of state. Because the equation of van der Waals is inconvenient to use, since it involves the solving of a cubic equation, other equations of state have been developed. That of Berthelot—

$$pv=n\,RT\left[1+\frac{9}{128}\,\frac{p}{p_c}\,\frac{T_c}{T}\left(1-6\frac{T_{c2}^{2}}{T}\right)\right]$$

involves critical pressure, p_c, and critical temuerature, T_c, and is useful at moderate pressures. The equation

$$pv=n\,RT+a\ (v)\ p$$

developed by M. A. Cook, where a(v) is the average covolume of the products of explosion, is applicable at the high temperatures and pressures involved in explosions. The average covolume is the proportion of the gas volume occupied by the molecules themselves.

4–8. Thermochemistry

a. General. Thermochemistry is concerned with the heat effects and internal energy changes accompanying chemical reactions. As an explosion is always an exothermic (heat-liberating) reaction and gas is produced by all the standard as well as almost all other explosives, the thermochemistry of explosives affords a basis for calculating the pressure and work effects of explosives. The thermal units are the calorie (cal) and kilogram-calorie (kg cal). These are the quantities of heat required to increase the temperature of 1 gram and 1 kilogram of water, respectively, from 15° to 16° C. For purposes of comparison and conversion, the following relationships between calories and other energy units are given:

1 calorie = 4.1855 international joules
= 4.1855×10^7 ergs
= 0.0413 liter-atmosphere

b. Heat of Formation. When a compound is formed by the union of its elements, the reaction may require the input of energy or may result in the liberation of heat energy. In the former case, the compound is said to have a negative heat of formation and, in the latter case, a positive heat of formation. As it is assumed that each of the elements has zero heat of formation, such reactions can be represented as follows when the heats of formation and reaction are expressed in kilogram-calories per mole.

$$N_2+2\ H_2=N_2H_4-22.70\ \text{kg cal}$$
$$-22.70$$
$$Ca+2\ C=CaC_2+14.6\ \text{kg cal}$$
$$+14.6$$

c. Heat of Combustion. As it is very difficult in many cases to form compounds directly from their elements and determine their heats of formation, it is necessary to derive such values indirectly. As many compounds are susceptible to combustion (oxidation), it is practicable to determine the heat of combustion and calculate the heat of formation from this. An example follows:

$$C+O_2=CO_2+94.385\ \text{kg cal}$$
$$+94.385$$
$$CO+\tfrac{1}{2}\ O_2=CO_2+67.957\ \text{kg cal}$$
$$+94.385$$

From this the heat of formation of carbon monoxide is calculated to be 94.385 − 67.957 or 26.428 kg cal per mole. In the case of water, the heat of combustion of hydrogen is found to be—

$$H_2+\tfrac{1}{2}\ O_2=H_2O\ \text{(liquid)}+63.387\ \text{kg cal}$$

and the heat of formation of liquid water therefore, is 68.387 kg cal. However, the heat of liquefaction and cooling of gaseous water has been determined to be 10.561 kg cal per mole, so the heat of formation of gaseous water would be 68.387 − 10.561 or 57.826 kg cal and the equation for its formation would be—

$$H_2+\tfrac{1}{2}\ O_2=H_2O\ \text{(gas)}+57.826\ \text{kg cal}$$
$$+57.826$$

In making thermochemical calculations, therefore, it is necessary to take into account the physical conditions of the reactants and products of reaction. Heats of combustion are determined in a calorimetric bomb under conditions of constant volume. If the oxidation reaction were to take place under conditions of constant pressure, some of the energy liberated would be utilized for displacement of the air surrounding the reactants. Determination at constant pressure would allow for the differences between compounds, with respect to changes in volume, and make heat of combustion and derived heat of formation values more directly comparable. It therefore is customary to determine heats of combustion at constant volume, calculate this to constant pressure basis, and calculate from this the heat of formation at constant pressure. Since the volumes of solids and liquids are negligible as compared with those of their gaseous products of oxidation, and gaseous products are always formed from solid or liquid explosives, their heats of combustion at constant pressure are always less than those at constant volume. To convert the heat of combustion measured at constant volume to that at a constant pressure, the gram-molecule increase in gas volume is multiplied by 0.582 kilogram calorie and then subtracted from the heat of combustion measured at constant volume. The result is the heat of combustion at constant pressure. An example is the combustion of dinitrobenzene—

$$C_6H_4N_2O_4\ \text{(solid)}+5O_2\ \text{(gas)}=$$
$$6CO_2\ \text{(gas)}+N_2\ \text{(gas)}+2H_2O$$
$$\text{(liquid)}+704.8\ \text{kg cal}$$

From this it is seen that the seven molecular volumes of gas formed represent an increase of two over the five of oxygen required for complete oxidation. The heat of combustion at constant pressure therefore is—

$$704.8-(2)\ (0.582)\ \text{or}\ 703.634\ \text{kg cal}$$

This method gives only an approximation, due to deviations from the gas laws and the fact that solids and liquids do not actually have zero volumes.

d. Heat of Reaction. Since, from the equations in *a* through *c* above, the heat of reaction is equal to the difference between the heat of formation of the product and the sum of the heats of formation of the reactants, it is practicable to calculate the heat of reaction, if the heats of formation of all the reactants and products of reaction are known. An example of this is the following:

$$H_2O \text{ (liquid)} + SO_3 \text{ (gas)} = H_2SO_4 \text{ (liquid)} + 29.843 \text{ kg cal}$$

A statement of heat of combustion or reaction has little significance unless the temperature is stated or understood. It is necessary that all heats are referred to the same temperature, which is usually taken as 25° C. Temperature is important, since a reaction may be endothermic at room temperature, but exothermic at explosion temperature.

e. Heat of Explosion. All military high explosives, except nitroglycerin and ammonium nitrate, and all solid propellants are oxygen deficient, that is, they do not contain sufficient oxygen to convert all of the carbon and hydrogen present to carbon dioxide and water, respectively. Since they cannot utilize atmospheric oxygen during detonation or auto combustion, the products of explosion consist chiefly of mixtures of carbon monoxide, carbon dioxide, hydrogen, water, and nitrogen; and the heat liberated by explosion is less than that produced by complete combustion to carbon dioxide, water, and nitrogen. An example follows: 80–20 amatol is an oxygen-balanced explosive that detonates according to the thermochemical reaction.

$$\begin{array}{llll} 21NH_4NO_3 + & 2C_7H_5N_3O_6 = & 14CO_2 + & 47H_2O \\ 1846.53 & 32.72 & 1321.39 & 2717.82 \\ & \text{(gas)} + 24N_2 + 2159.96 & & \end{array}$$

kg cal

This is equivalent to a heat of explosion of 1011.1 kg cal/kg and a gas volume of 892.24 liters/kg measured under standard conditions of 0° C. and 1 atmosphere pressure. 60–40 amatol, corresponds to the mollecular proportions

$$17NH_4NO_3 + 4C_7H_5N_3O_6$$

and contains 75 percent of the oxygen required for complete oxidation of the carbon and hydrogen present. In order to determine the distribution of the available oxygen in the formation of products of explosion, advantage is taken of the water-gas reaction equation

$$\frac{CO}{CO_2} \cdot \frac{H_2O}{H_2} = K$$

It has been found that the value of k varies with temperature above 1,200° C., being approximately 7.0 at the temperature of explosion of the composition. If this also is used in the setting up of simultaneous equations, it is found that the equation for the explosion of 60–40 amatol is—

$$\begin{array}{lll} 17NH_4NO_3 + & 4C_7H_5N_3O_6 = & \\ 1494.81 & 65.44 & \\ 17CO + & 11CO_2 + & 36H_2O \\ & \text{(gas)} + 8H_2 + 23N_2 + 2009.24 \text{ kg cal} & \\ 449.53 & 1038.23 & 2081.74 \end{array}$$

This is equivalent to a heat of explosion of 884.4 kg cal/kg and a gas volume of 858.33 liters/kg. By comparing these values with those for 80–20 amatol, it is seen that lack of oxygen balance results in decrease in both heat of explosion and gas volume on a weight basis. The same method can be applied to propellants containing a number of compounds, by calculating the empirical formula of 1 kilogram and using this as a basis for deriving an equilibrium equation similar to that shown above for 60–40 amatol. In calculating the volume of gas produced, the water being gaseous, advantage can be taken of the fact that this is the same as if the explosive or propellant had been oxidized completely to carbon dioxide, gaseous water, and nitrogen, but only if appreciable amounts of methane, ammonia, or solid carbon are not formed.

f. Temperature. The temperature developed by the products of an explosion depends upon the quantity of heat liberated, the volume of gaseous products of explosion, and the specific heat (at constant volume) of the products. No method for determining the maximum temperature of an explosion has been developed, and this value must be calculated. The best method for this involves the solving of five simultaneous equations conforming with the hydrodynamic theory of detonation (see para 4–10). Application of this method has given the representative values shown in table 4–3.

4–9. Ignition Temperature

a. A combustible substance, such as wood or coal, must be heated to a certain temperature before its reaction with the oxygen of the air proceeds so rapidly that the heat of combustion is sufficient to maintain a temperature at which the reaction is self-sustaining and emits light. The minimum temperature required to bring about such self-propagating combustion is termed the kindling temperature of the substance, and if the burning substance is cooled below its kindling temperature, combustion ceases. The kindling temperature of a material may also be considered the minimum temperature at which the heat of reaction is liberated faster than it is conducted and radiated away. The kindling temperature of a material will vary somewhat with conditions, particularly the state of subdivision of the material.

b. While many military explosives and propellants contain sufficient oxygen to permit autocombustion, each must be heated to a minimum temperature for a period of time, before combustion can be self-propagating. This is termed the ignition temperature, and is generally taken as the minimum temperature which will cause ignition within a 5-second interval. Experimental work has indicated that the ignition temperature of a high explosive corresponds rather closely with its probable boiling point, and that the vapor phase is what actually undergoes ignition. As TNT, PETN, and picric acid have been found neither to detonate nor burn in a vacuum, it is probable that their boiling points in a vacuum are lower than their ignition temperatures.

c. The ignition temperatures of high explosives are of relatively little practical importance, but those of propellants are important. Since propellants are used under conditions where they undergo burning, the lower the ignition temperature, the more rapidly will ignition take place and the more uniformly will burning progress.

4-10. Theory of Deflagration and Detonation

a. Deflagration. If a particle of an explosive is subjected to heat so as to cause a rapid increase in its temperature, a temperature is reached at which the rate of exothermic decomposition becomes significant. At temperatures within the approximate range of 500° to 1,700° C., this decomposition involves volatilization from the surface prior to decomposition. The heat liberated by decomposition increases the rate of reaction, and the resulting rate of increase in temperature is exponential. At a certain temperature, characteristic of the explosive, the output of heat is sufficient to enable the reaction to proceed and be accelerated without input of heat from another source. At this certain temperature, called the ignition temperature, deflagration begins. Deflagration is a surface phenomenon, with the reaction products flowing away from the unreacted material below the surface. Deflagration of all the particles in a mass of finely divided explosive can occur nearly simultaneously. In such case, the confinement of the particles within the mass, because of the viscosity of the gaseous products, has the effect of increasing pressure. Increase in pressure, in turn, has the effect of increasing rate of reaction and temperature. The final effect of deflagration under confinement is explosion, which may be violent deflagration or even detonation. In the case of low explosives such as loose black powder and pyrotechnic compositions, only violent deflagration can take place. Nitrocellulose propellants can undergo burning or, if confinement is sufficient, deflagration can become so rapid that detonation ensues. High explosives undergo a variety of reactions to intense heat, such as that of flame. Lead azide undergoes no initial deflagration, even at atmospheric pressure, while diazodinitrophenol undergoes only deflagration, unless there is sufficient confinement to insure the development of some pressure during deflagration. Nitroglycerin in small quantity can undergo deflagration at atmospheric pressure, but if the quantity is larger, deflagration produces so much heat that detonation follows quickly. TNT can deflagrate even in large quantities when there is only slight confinement, but deflagration is followed by detonation if confinement is such that pressure of some magnitude can be developed. Even ammonium nitrate, a very insensitive material, can be caused to detonate if confinement is such that a pressure of 2,500 to 3,000 psi can be developed during deflagration.

b. Detonation.

(1) The hydrodynamic theory of detonation, based on physical theories of shock waves and the chemical theory of absolute reaction rates, utilizes the established laws of conservation of mass, energy, and momentum. As derived, the hydrodynamic theory applies to explosives under complete confinement or in the form of cylindrical cartridges or spheres of sufficient diameter that the material near the center of the mass may be regarded as conpletely confined.

(2) By application of the three laws, there can be established three equations relating the five variables, pressure, density (volume $^{-1}$), temperature, detonation rate, and translational velocity of the gaseous molecules of the reaction products. An equation of state

$$pv = n\,RT + a\ (v)\ p$$

is used; and a fifth equation is obtained, by applying the physical principle that a shock wave passes through a gas with a velocity equal to the sum of the translational velocity of the gas plus the velocity of sound in the gas at its final temperature and density. By solution of the five simultaneous equations, the characteristics of a given explosive can be calculated.

(3) From the information obtained through the hydrodynamic theory, a

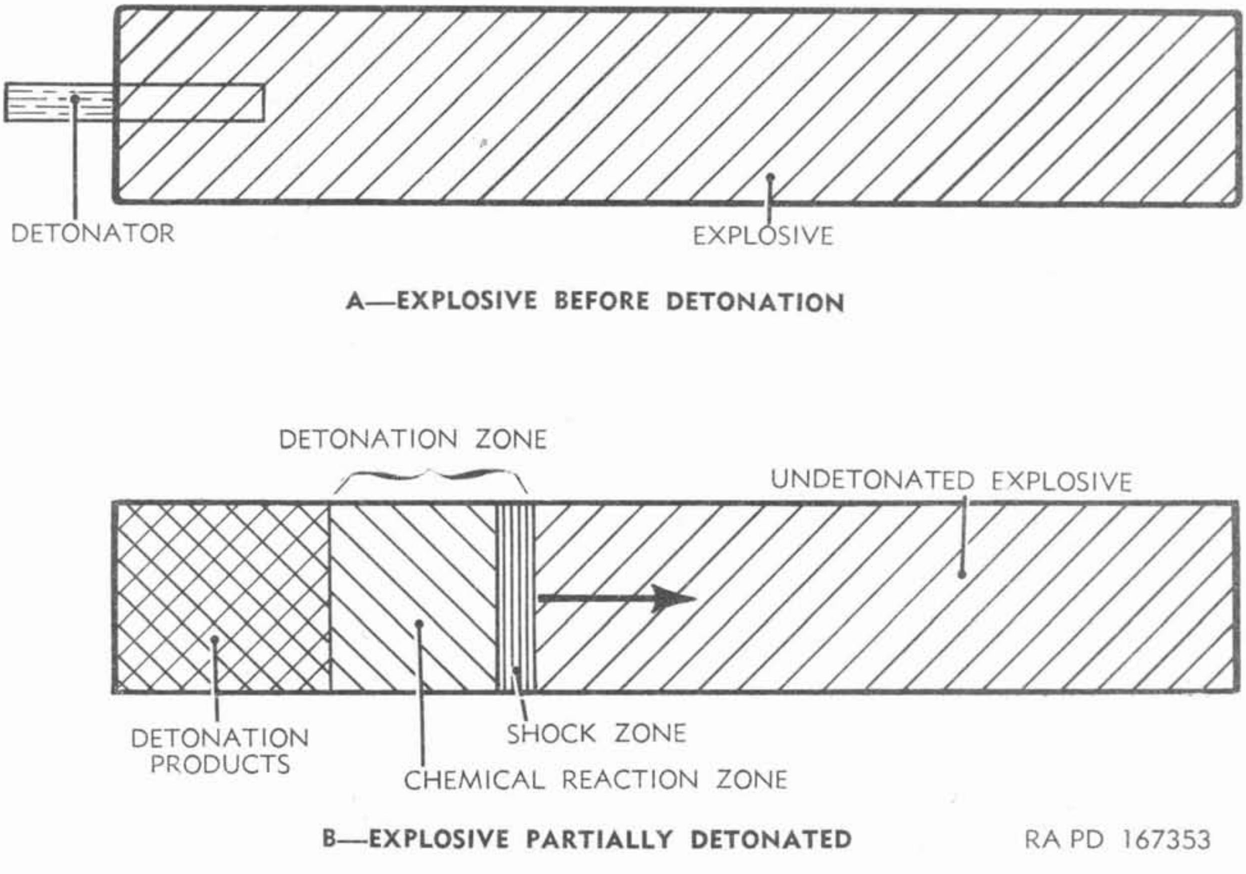


Figure 4–1. Progress of detonation through a column of explosive.

mechanism of detonation can be visualized (see fig. 4-1). After the detonator functions, a *detonation zone*, in which the chemical reaction is taking place, travels through the column of explosive. This detonation zone is generally considered to include a very narrow *shock zone* (10^{-5} cm) or *shock wave*. Little or no chemical reaction occurs in this shock zone, but the pressure reaches its peak. The detonation zone includes not only this shock zone, but also the *chemical reaction zone* (0.1–1.0 cm). Following this detonation zone are the *detonation products*. In front of the shock zone is the unreacted explosive in its original state of density, pressure, velocity, and temperature. At or near the beginning of the chemical reaction zone, the high temperature to which the material is raised by compression in the shock zone initiates chemical reaction. Maximum density and pressure occur at the beginning of the reaction zone, while the temperature and velocity reach their peak at the completion of the chemical reaction. The detonation products flow with great velocity, but of lesser degree than the velocity of the detonation zone, *toward* the undetonated explosive. This is characteristic of detonation in contradistinction to deflagration, in which case the reaction products flow *away* from the unreacted material. The velocity of advance of the detonation zone is termed the *detonation rate*.

(4) As each individual molecule of explosive undergoes ordinary thermal reaction starting with a low initial temperature, there is a lag effect or induction period that depends exponentially on the reciprocal of the initial absolute temperature. With an initial temperature of 725° C., the induction period is of the order of 10^{-5} second. With high initial temperatures, it appears that the last 75 percent of the reaction requires only about 10^{-11} second.

(5) On the basis of the hydrodynamic theory, the characteristics of detonation of explosives have been calculated and representative values are given in table 4-3.

Table 4-3. Detonation Characteristics of Explosives

	Loading density, (G/ML)	*Temperature, (° C.)*	*Pressure, (10 ATM)*	*Detonation rate, (M/SEC)*
Nitroglycerin	1.60	5,370	1.99	8,060
Tetryl	1.50	4,480	1.48	7,125
TNT	1.50	3,600	1.10	6,480
Ammonium nitrate	1.00	1,350	0.25	3,420

4-11. Rate of Burning

a. As ordinarily applied, the term burning refers to the high temperature oxidation of an organic material or other fuel, such as sulfur or hydrogen. Ordinarily the oxygen utilized in such burning is that present in the surrounding air. Some high explosives and all propellants can undergo a type of autocombustion, also referred to as burning, in which the oxygen present in the material is utilized for oxidizing the carbon and hydrogen. The exothermicity of this recombination is such that autocombustion is self-sustaining from layer to layer of the material. If the material contains sufficient oxygen to convert all the carbon and hydrogen present to carbon dioxide and water, these and nitrogen are the gaseous products of burning. If less than this proportion of oxygen is present in the high explosive, or propellant, carbon monoxide and hydrogen also are formed.

b. Some high explosives, such as mercury fulminate, can undergo burning only when they have a thickness of layer of about one crystal. If the layer is thicker, burning is transformed into detonation. If desensitized by pressing (dead pressed) at 25,000 psi or more and then ignited, mercury fulminate burns without detonation, the rate of burning decreasing with increase in density.

c. Priming compositions burn with great rapidity but do not undergo detonation. The detonation of those containing lead azide, mercury fulminate, or lead styphnate is prevented by the dampening effect of relatively inert ingredients, such as antimony sulfide, lead thiocyanate, ground glass, or sulfur.

d. While high explosives, such as TNT and tetryl, burn with the aid of atmospheric oxygen, they do not undergo autocombustion at atmospheric pressures. The destruction of such materials by the burning of thin layers, therefore, is practicable. Nitroglycerin, on the other hand, undergoes little if any autocombustion prior to detonation, even at atmospheric pressure.

e. Except as ingredients of propellant compositions, high explosives are not used under conditions where they undergo burning rather than detonation. The burning rates of high explosives, therefore, are of little practical significance. Propellants are utilized under conditions where they undergo autocombustion and detonation is very undersirable.

4–12. Rate of Detonation

a. The rate of detonation of a given explosive, provided that sufficient initiator or booster explosive is used, is determined by its degree of confinement and loading density. If confined only slightly, as by a cardboard or glass tube, a cylindrical column of high explosive detonates at a lower rate than if a heavy steel tube surrounds the explosive. This is because of the greater loss of energy in directions at right angles to the axis of the column. This effect is seen also if the diameter of the column of explosive is decreased. In such case, there is a minimum diameter, also dependent upon degree of confinement, below which detonation cannot propagate ifself throughout the length of the column. In practice, detonation rates are determined with columns 1 inch or more in diameter confined in Shelby steel tubing or as strong a material as the test method will permit. Decrease in loading density causes decrease in rate of detonation, the relationship being linear.

b. Each explosive has a characteristic maximum rate of detonation for a given density, although in the least sensitive explosives, particle size has some effect on this value. If an explosive is improperly initiated or has become desensitized, a detonation wave can, in some cases, progress through the explosive at much less than its normal maximum rate. Although nitroglycerin usually detonates at a maximum rate of about 8,000 meters per second, it can do so at rates as low as 1,500 to 2,000 meters per second. However, investigation has indicated that not all of the nitroglycerin is detonated in such cases. Gelatin dynamites, which normally detonate at rates of about 4,000 meters per second when manufactured, sometimes become desensitized during storage and detonate at about half the original rate.

c. As used in military ammunition, high explosives are loaded by casting in a molten form or by pressing to near their maximum densities. A comparison of the rates of detonation of some of the standard high explosives

Table 4–4. Rates of Detonation of High Explosives

	Rate of detonation, meters per second at density of—						
	1.50	1.55	1.57	1.60	1.63	1.70	1.71
RDX	7,650	-------	-------	-------	-------	-------	8,400
PETN	7,525	-------	-------	-------	-------	8,300	-------
Haleite	7,570	7,750	-------	-------	-------	-------	-------
Tetryl	7,125	-------	-------	-------	-------	-------	7,850
Nitroguanidine	7,450	7,650	-------	-------	-------	-------	-------
Picric acid	6,775	-------	-------	-------	-------	7,350	-------
Picric acid, cast	-------	-------	-------	-------	-------	-------	7,350
Explosive "D"	6,710	-------	-------	-------	7,150	-------	-------
TNT	6,620	-------	-------	6,970	-------	-------	-------
TNT, cast	-------	-------	6,675	-------	-------	-------	-------

at a given density and at their maximum loading density values is given by table 4-4.

d. In general, the rates of detonation of explosives are reflected by their relative brisance (shattering or fragmentation) values. The higher the rate of detonation of an explosive, the greater the brisance and the more effective it is as a booster charge to initiate another explosive. This is the reason for the use of RDX, PETN, and tetryl in compound detonators, and the use of tetryl in boosters for artillery shell.

CHAPTER 5

PROPERTIES AND TESTS OF HIGH EXPLOSIVES

5–1. Sensitivity

a. Sensitivity to Impact.

(1) It is well known that explosives present considerable hazard when subjected to shock, but it is only recently that the mechanism of explosion by impact has been elucidated. It was long thought that initiation of explosion by a blow was due to rapid shear causing rupture of explosive molecules or the energy of impact causing direct mechanical excitation of such molecules. It has been since found that explosion by impact probably is due to the development in the explosive of hot spots of finite size. Such hot spots are indicated to be formed by the adiabatic compression of small air or vapor bubbles trapped within the explosive, the friction of a particle of grit with a crystal of the explosive, or the viscous heating of rapidly flowing explosive under the pressure of impact. The first of the three causes is the most generally operative and can explain the initiation of explosives free of grit and those having very high melting points.

(2) The greatly compressed gas bubble becomes highly heated and is surrounded by explosive also under considerable pressure at the instant of impact. If the temperature of the gas and the pressure are sufficiently great, explosion of the molecules adjacent to the gas is initiated and the explosive wave propagates itself throughout the rest of the explosive. The temperature required for explosion and, therefore, the degree of compression and severity of impact will vary for different explosives.

(3) In practice, sensitivity to impact is expressed as the minimum height of fall of a given weight required to cause at least 1 explosion in 10 trials, or the minimum height of fall of a given weight to cause explosions in 50 percent of the trials. In such tests, the explosive is ground so as to pass through a No. 50 seive and be retained on a No. 100 sieve. Examples of the test equipment used are the Picatinny Arsenal standard apparatus shown in figures 5–1 and 5–2 and the U.S. Bureau of Mines apparatus shown in figure 5–3. In carrying out the test with the Picatinny apparatus, a steel die cup is filled with the explosive, covered with a brass cover, surmounted with a steel vented plug, placed in a positioned anvil as shown in figures 5–1 and 5–2, and subjected to the impact of a weight falling from a predetermined height. The minimum height, in inches, required for explosion is found after repeated trials. In making the test with the Bureau of Mines apparatus, 0.02 gram of the sample is spread uniformly on a hard steel block, over a circular area 1 centimeter in diameter (fig. 5–3). A hard steel tip of that diameter, imbedded in a steel plunger, is lowered so as to rest on the explosive and turned gently so as to insure uniform distribution and compression of the explosive. The plunger then is subjected to the impact of a weight falling from a predetermined height. When the minimum height required

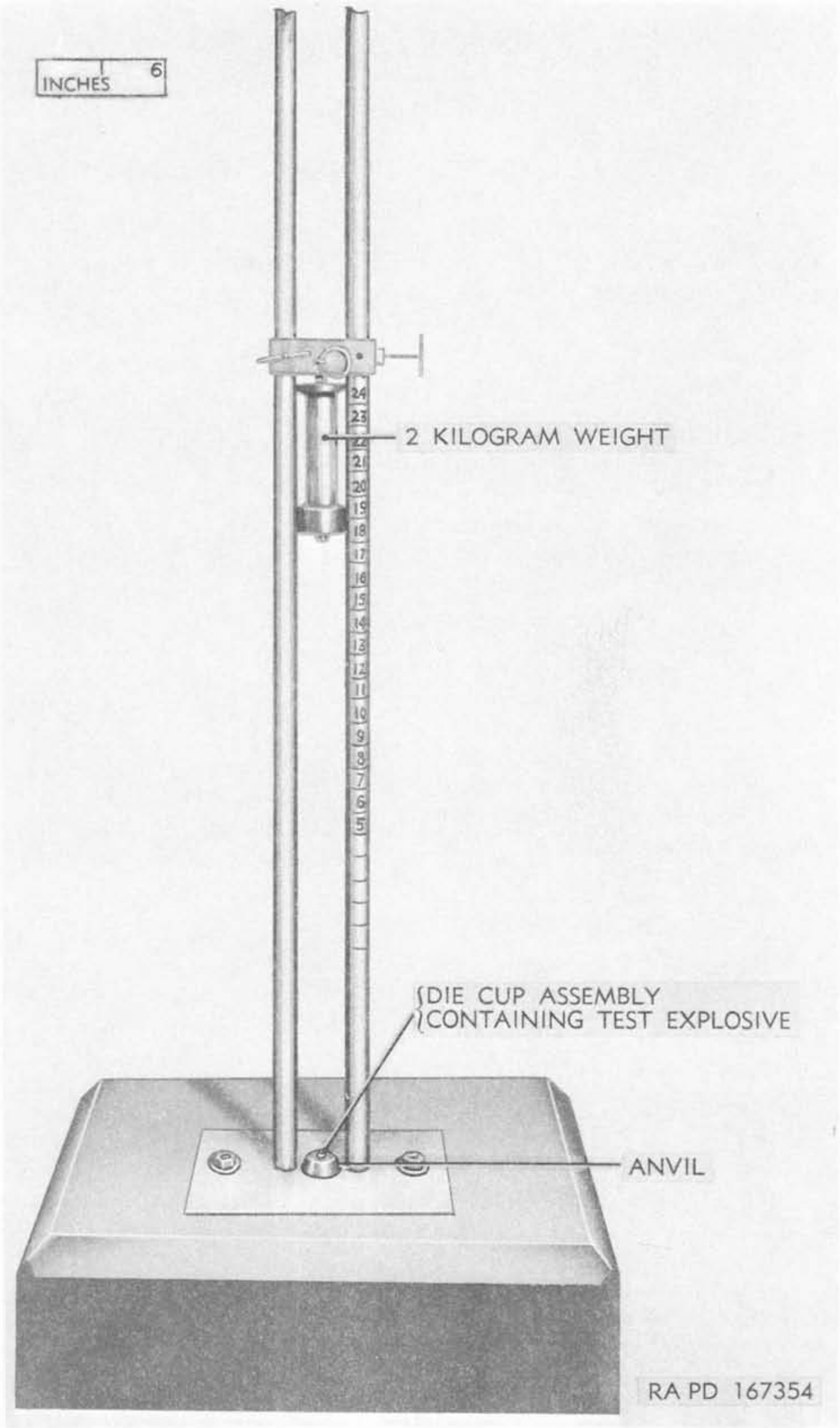


Figure 5-1. Picatinny Arsenal impact test apparatus.

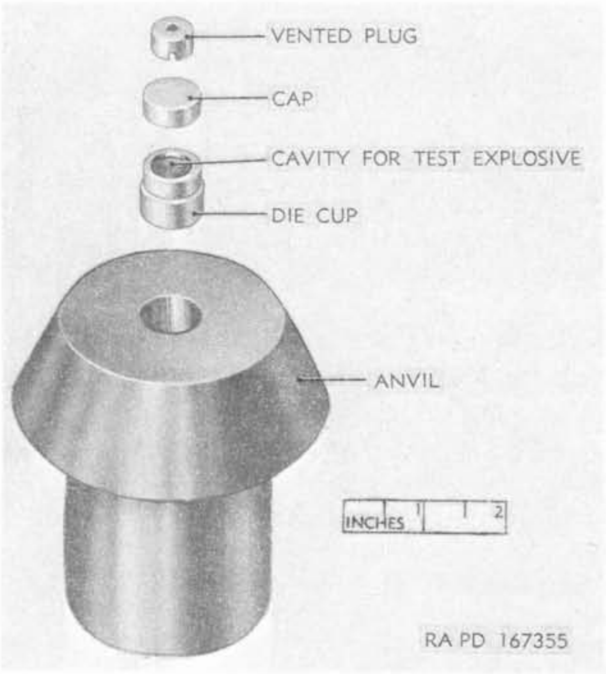


Figure 5–2. Parts of Picatinny Arsenal impact test apparatus.

for explosion is found after repeated trials, this is expressed in centimeters. The Picatinny apparatus can be used for testing explosives having a very wide range of sensitivity, but the Bureau of Mines apparatus cannot cause the explosion of the most insensitive explosives and can be used only for testing explosives no less sensitive than TNT. The Picatinny apparatus can be used for testing solid or liquid explosives. The test with the Bureau of Mines apparatus can be modified so as to be applicable to liquid explosives. This is accomplished by using 0.007–0.002 gram (1 drop) of the explosive absorbed in a disk of dry filter paper 9.5 mm in diameter.

(4) In general, the sensitivity of an explosive, as determined by the impact test, increases with temperature; and the molten material is much more sensitive than the hot, solid material. Test data for TNT, RDX, and ammonium nitrate in this connection are given in table 5–1.

Table 5–1. Effect of Temperature on Sensitivity to Impact

Temperature, ° C.	*Impact test, Picatinny apparatus minimum height in inches, of fall of 2 KG weight to explode*		
	RDX	*TNT*	*Ammonium nitrate*
25	8	14	31
75			28
80		7	
90	8	[a] 3	
100			27
105	5	[a] 2	
150			27
175			[a] 12

[a] Material in molten condition.

(5) Comparative values for the sensitivity of military explosives to impact are given in tabular form in appendix A.

b. Sensitivity to Friction.

(1) Like explosion by impact, the initiation of explosion by friction is ascribed to the formation of hot spots. In the case of frictional initiation, the first stage usually is a burning process. Lead azide is an exception to this, true detonation starting at the point of initiation. For a given frictional force, the incidence of hot spots is determined by the thermal conductivity of the sliding surfaces involved. The melting point of the friction-producing material also is of importance, as this determines the highest temperature that can be developed. If this is low in value, explosion cannot be caused by friction. The sensitizing action of grit appears to be of no effect if the melting point of the grit is below 500° and if the hardness is below 2–3 on the Moh's scale. The most effective seem to be particles with high hardness (4 and up) and high melting point.

(2) In practice, the sensitivity of explosives to friction is determined by the pendulum friction test (fig 5–4). This is made by means of a pendulum, to

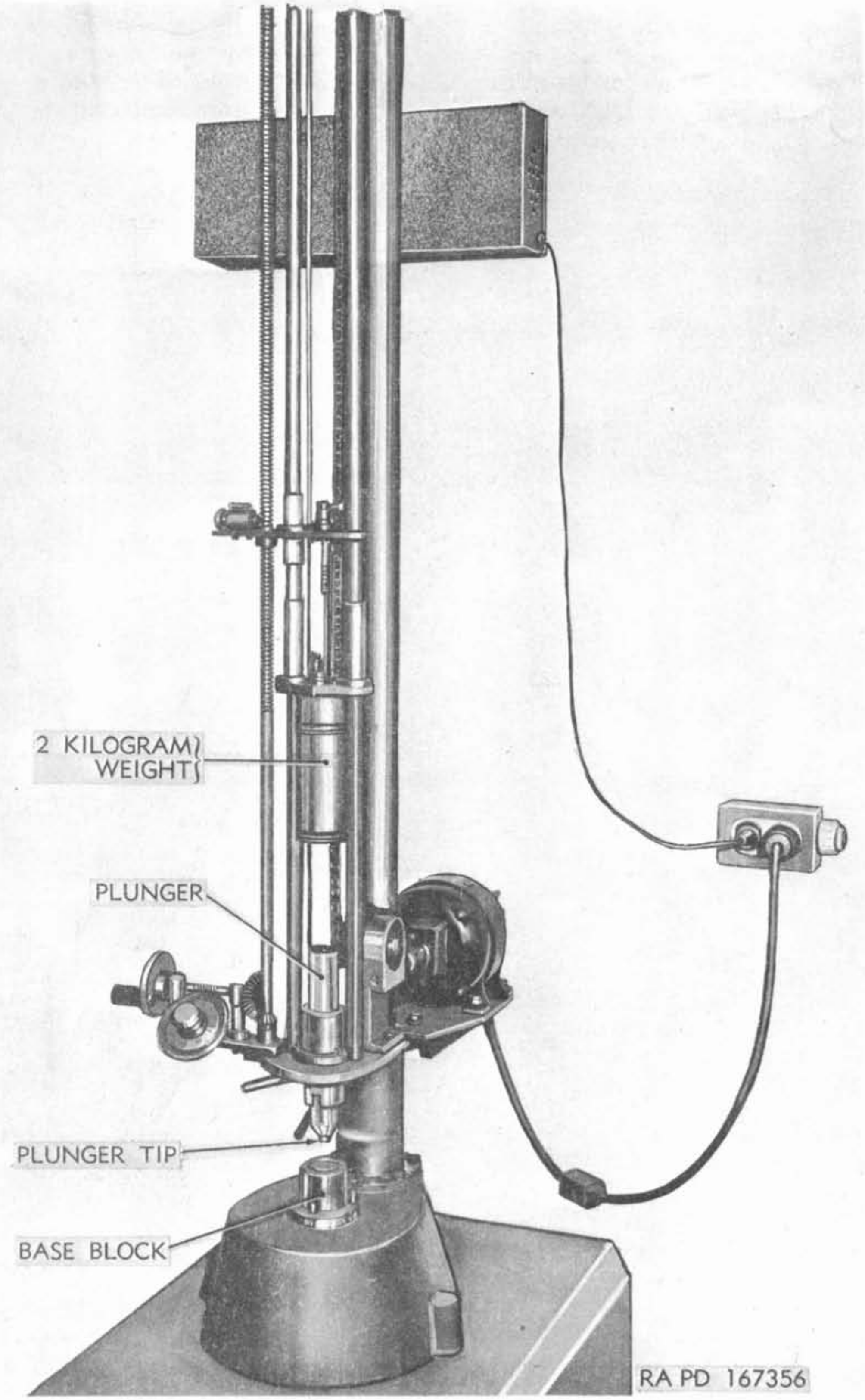


Figure 5-3. Bureau of Mines impact test apparatus.

the lower end of which is attached a 20-kilogram shoe with an interchangeable face of steel or fiber. The shoe is permitted to fall from a height of 1 meter and sweep back and forth across a grooved steel friction anvil. The pendulum is adjusted to pass across the friction anvil 18±1 times before coming to rest when no explosive is present. The adjustment

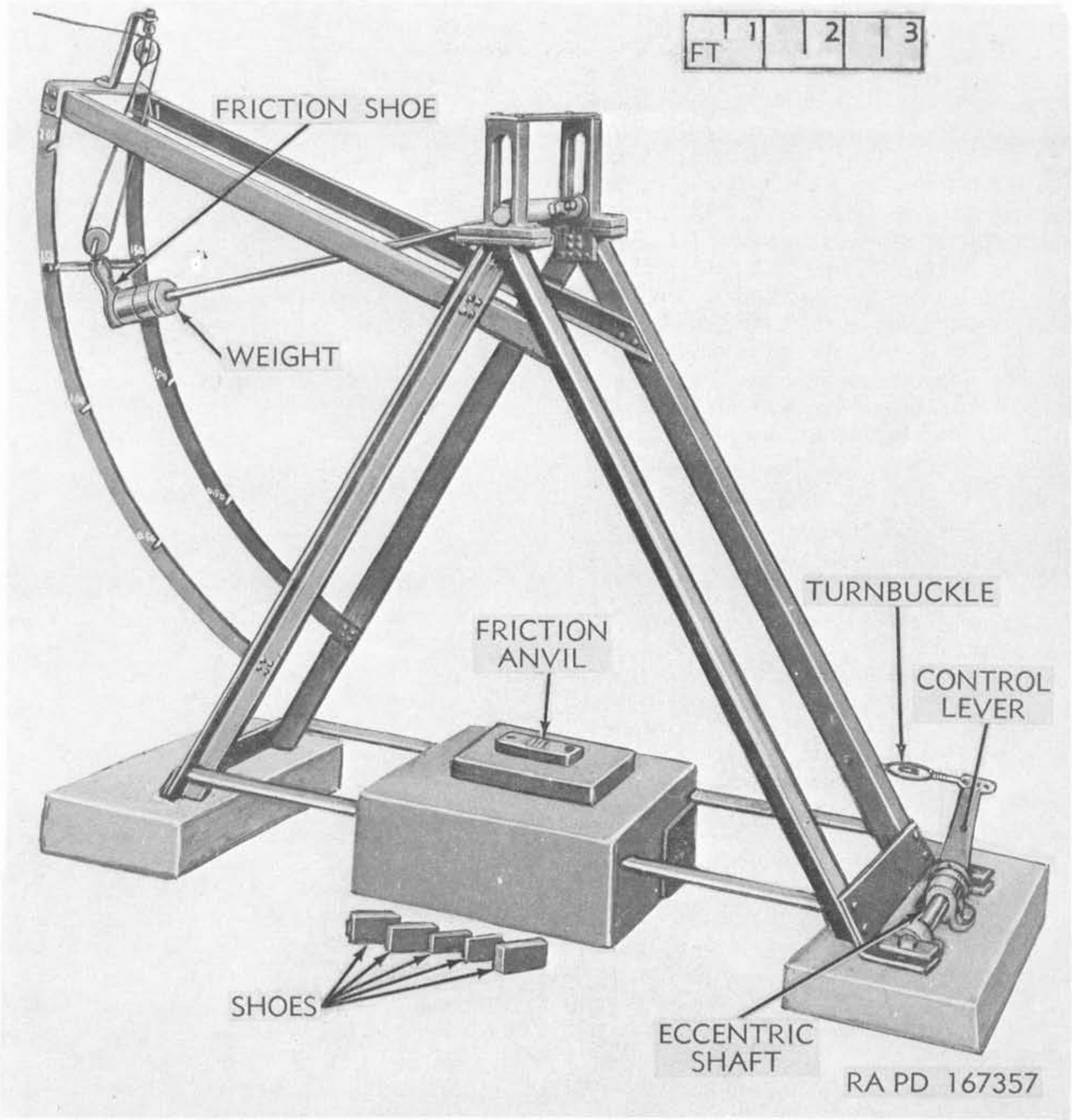


Figure 5–4. Pendulum friction test apparatus.

is made by raising or lowering the A-frame by means of an eccentric shaft connected to a control lever. A turnbuckle connected to the control lever and A-frame holds the control lever in the desired position. A 7-gram sample of the explosive is then spread evenly in and about the grooved portion of the friction anvil, and the shoe is allowed to sweep back and forth over the anvil until it comes to rest. Tests of 10 portions of the sample are made, and the number of snaps, cracklings, ignitions, and/or explosions is noted. As the steel shoe is the more effective in causing explosions, tests with the fiber shoe sometimes will show differences between explosives indicated by the steel shoe to be of the same degree of sensitivity.

(3) Comparative values for the sensitivity of military explosives to friction are given in tabular form in appendix A.

c. Sensitivity to Frictional Impact.

(1) Another test used for differentiating explosives, with respect to sensitivity to mechanical shock, is the rifle bullet impact test, in which the explosive is subjected to a combination of impact and friction (fig. 5-5). A bomb is prepared by screwing a closing cap to one end of a piece of cast-iron pipe 3 inches long, 2 inches in diameter, and threaded at both ends. The bomb is filled with the cast, pressed, or liquid explosive and is closed by screwing on a closing cap. With the loaded bomb in a vertical position, a caliber .30 bullet is fired through it from a distance of 30 yards, so that the bullet strikes between the two closing caps and at a right angle to the axis of the bomb. Five or more such tests are made and the percentage of explosions is noted.

(2) Comparative values obtained by rifle bullet impact tests of military explosives are given in tabular form in appendix A.

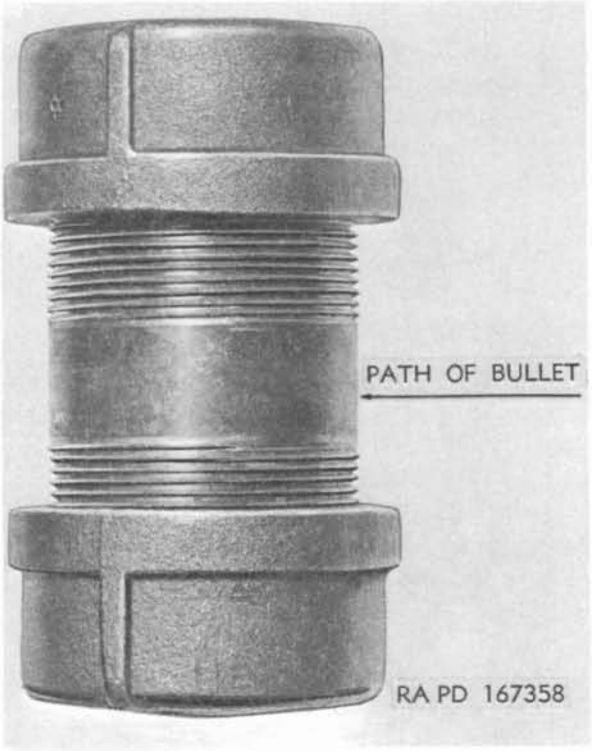


Figure 5-5. Rifle bullet impact test bomb.

d. Sensitivity to Heat and Spark.

(1) Heat causes the decomposition of an explosive at a rate that varies with the temperature. Almost all explosives are characterized by a critical temperature, below which the rate of decomposition is so small as to be negligible. Nitroglycerin has a critical temperature of approximately 50° C., and, above this, the increase in rate of decomposition with increase in temperature is disproportionate. Other explosives have higher critical temperatures, those of TNT and tetryl being about 460° and 235° C., respectively.

(2) If a small mass of an explosive is suddenly subjected to a high ambient temperature, there ensues an induction or incubation period during which the explosive absorbs heat. If the ambient temperature is sufficiently high, decomposition of part of

the explosive liberates heat and causes acceleration of the increase in temperature of the remaining explosive. When the temperature reaches a certain value characteristic of the explosive, the rate of the decomposition value becomes so great that explosion of the remaining material takes place. The temperature of the products of explosion is enormously greater than that developed during the prior slow decomposition; and if the explosion is so brought about at the end of a column of explosive, self-propagating detonation of the column can ensue. This occurs with ease when the explosive is of the initiating type, but it is not practicable to initiate the detonation of high explosives such as TNT and tetryl by externally applied heat unless the explosive is under considerable pressure. This is due to the impracticability of developing the extremely high ambient temperatures necessary to reduce the incubation period to the order of 10^{-5} second.

(3) In practice, the relative sensitivity of explosives to heat is determined by means of the explosion temperature test, which is made by means of the equipment shown by figure 5-6. Samples of 0.02 gram of the explosive, of such fineness as to pass through a No. 50 sieve, are transferred to empty gildingmetal cups of the type used for No. 8 blasting caps and compacted by tapping. Each blasting cap is then immersed to a fixed depth in a bath of molten Woods' metal. The molten metal bath is maintained at a predetermined temperature by means of an electric furnace equipped with a rheostat, the temperature being read by means of a thermoelectric pyrometer, having a temperature range from 0° to 1,400° C. and graduated at 10-degree intervals. By means of a stop watch the time of immersion required to cause flashing or explosion is noted. A number of such tests is made with the temperature of the bath varied so as to produce flashes or explosions over a range of approximately 2 to 10 seconds. The data so obtained are plotted as a time-temperature curve and from this is found the temperature required to cause flashing or explosion in 5 seconds.

(4) Another method, which has been used extensively, is to drop individual particles of the explosive on the surface of molten metal, the temperature of which is indicated by means of a suitable thermometer. The temperature of the metal is increased until explosion or flashing of the material takes place in 0.1 second or less.

(5) Test values for the sensitivity of military explosives to heat are given in tabular form in appendix A.

(6) The intensely heated volumes of air caused by discharges of static electricity can cause the ignition of solid or liquid explosives or the explosion of mixtures of explosive dust with air. No standard test has been established for evaluating sensitivity to spark, but the results of experimental work are summarized in table 5-2.

Table 5-2. Classification of Explosives With Respect to Hazard From Electric Sparks

Not dangerous	*Dangerous*
Bullseye powder	Black powder
Composition B	Diazodinitrophenol
Composition C-3	E. C. powder
Graphited smokeless powder	Explosive "D"
PETN	Lead azide
RDX	Lead styphnate
TNT	Mercury fulminate
	Tetracene
	Tetryl
	Tetrytol
	Ungraphited smokeless powder

(7) Nonelectric sparks, such as those from steel, burning wood, etc., are much more effective in causing ignition and, therefore, represent greater hazard. As an example, black powder must be classed as a very dangerous

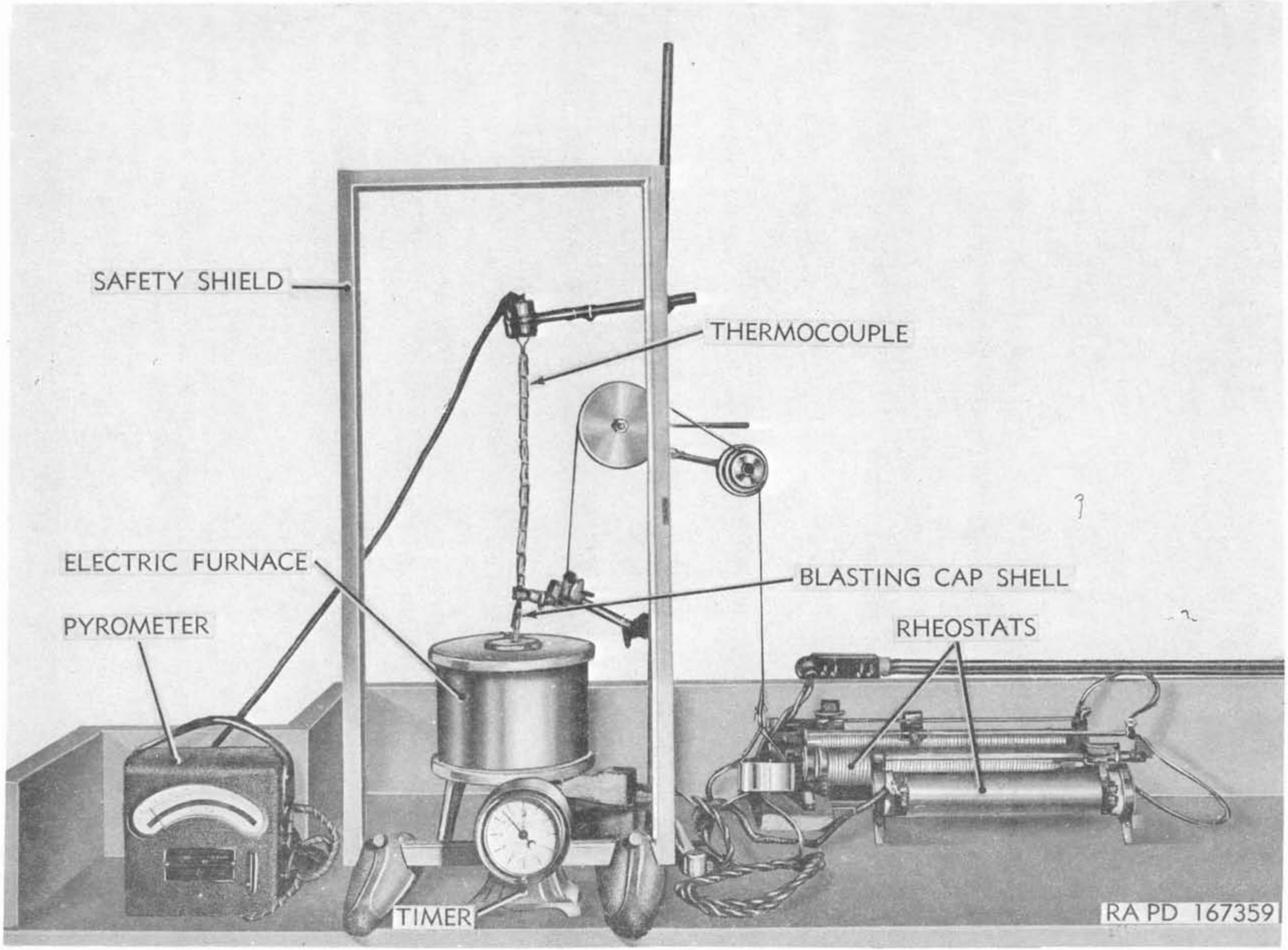


Figure 5-6. Explosion temperature test apparatus.

explosive with respect to solid sparks, and all explosives must be classed as dangerous in this respect. It is for this reason that only nonsparking tools are used in mechanical operations in connection with both high and low explosives.

e. Sensitivity to Initiation.

(1) Just as explosives vary greatly with respect to sensitivity to impact, friction, and heat, so do they vary in sensitivity to initiation by another explosive. Particle size affects the sensitivity to some extent, as do temperature, density, and physical state. Cast TNT is distinctly less sensitive to initiation than pressed TNT. Solid nitroglycerin is less sensitive than the liquid material. Decrease in initial temperature causes decrease in sensitivity to initiation, but this is not of great magnitude. Increase in density causes decrease in sensitivity, this effect being most pronounced with the least sensitive explosives. The effects of change in particle size vary considerably. Decrease in the average particle size of TNT causes increase in sensitivity to initiation, but reduction of the average particle size of ammonium nitrate past a certain value causes desensitization.

(2) In general, sensitivity to initiation is parallel to sensitivity to impact; but this is not true with respect to sensitivity to initiation and heat.

(3) Relative sensitivity to initiation is determined by means of the sand test, conducted with diminishing weights of an initiator, such as lead azide, until there is obtained the minimum amount which will cause complete detonation of 0.40 gram of the powdered explosive when pressed in a blasting cap shell under a pressure of 3,000 psi (see fig. 5-7). A few explosives, such as explosive D, cannot be detonated directly with lead azide or mercury fulminate. In such cases, there is pressed on top of the 0.400 gram of explosive under test a weighed charge of powdered tetryl and then a charge of 0.200 gram of lead azide. By repeated tests with diminishing weights of tetryl, there is determined the minimum weight of tetryl required to cause complete detonation of the 0.400 gram of explosive.

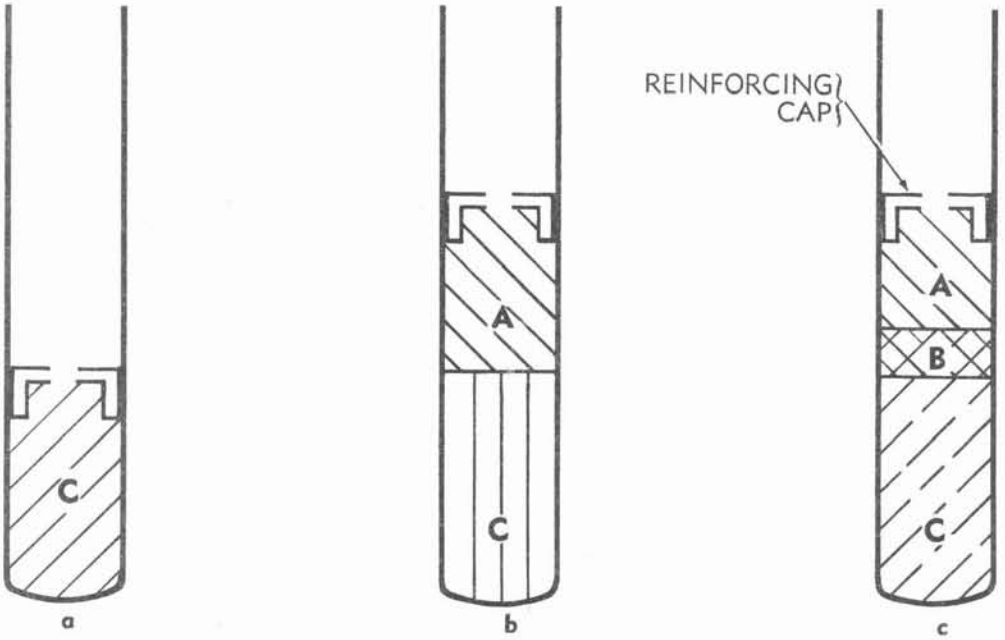
(4) Minimum detonating charge values for military explosives are given in tabular form in appendix A.

5-2. Stability

a. The very fact that certain chemical compounds can undergo explosion when heated indicates that there is something unstable in their structures. While no precise explanation has been developed for this, it is generally recognized that the nitrate, nitro, nitroso, diazo, and azide groups are intrinsically in a condition of internal strain. Increased strain through heating causes a sudden disruption of the molecule and consequent explosion.

b. In some cases, this condition of molecular instability is so great that decomposition takes place at ordinary temperatures at rates characteristic of the compounds. TNT has been found to undergo negligible decomposition in 20 years, inositol hexanitrate [$C_6H_6(NO_3)_6$] decomposes rapidly in a few days, and mercuric azide sometimes explodes as rapidly as it crystallizes from solution. The presence of moisture, in some cases, accelerates such decomposition by adding a hydrolytic reaction that produces free nitric and nitrous acids.

c. Because of the necessity for withstanding long time storage and adverse tropical temperatures, military explosives are selected so as to have maximum chemical stability as well as other satisfactory characteristics. Furthermore, efforts are being made continuously to replace compounds that are considered to be weak with respect to stability. An example is the virtual replacement of mercury fulminate, which was used for many years in spite of poor stability because of the nonavailability of a superior substitute. More stable new materials are replacing nitroglycerin in some propellants. The replacement of nitrocellulose at this time is impracticable for logistics and



a—TEST EXPLOSIVE INITIABLE BY FLAME: INITIATING EXPLOSIVE NOT REQUIRED
b—TEST EXPLOSIVE NOT INITIABLE BY FLAME: INITIATING EXPLOSIVE REQUIRED
c—TEST EXPLOSIVE NOT INITIABLE BY FLAME: INITIATING AND BOOSTER EXPLOSIVES REQUIRED
A—INITIATING EXPLOSIVE: 0.300 GRAM LEAD AZIDE IN **b**; 0.200 GRAM IN **c**
B—BOOSTER EXPLOSIVE: 0.100 GRAM TETRYL
C—HIGH EXPLOSIVE UNDER TEST: 0.400 GRAM

RA PD 167360

Figure 5-7. Loading of blasting caps for sand test of high explosives.

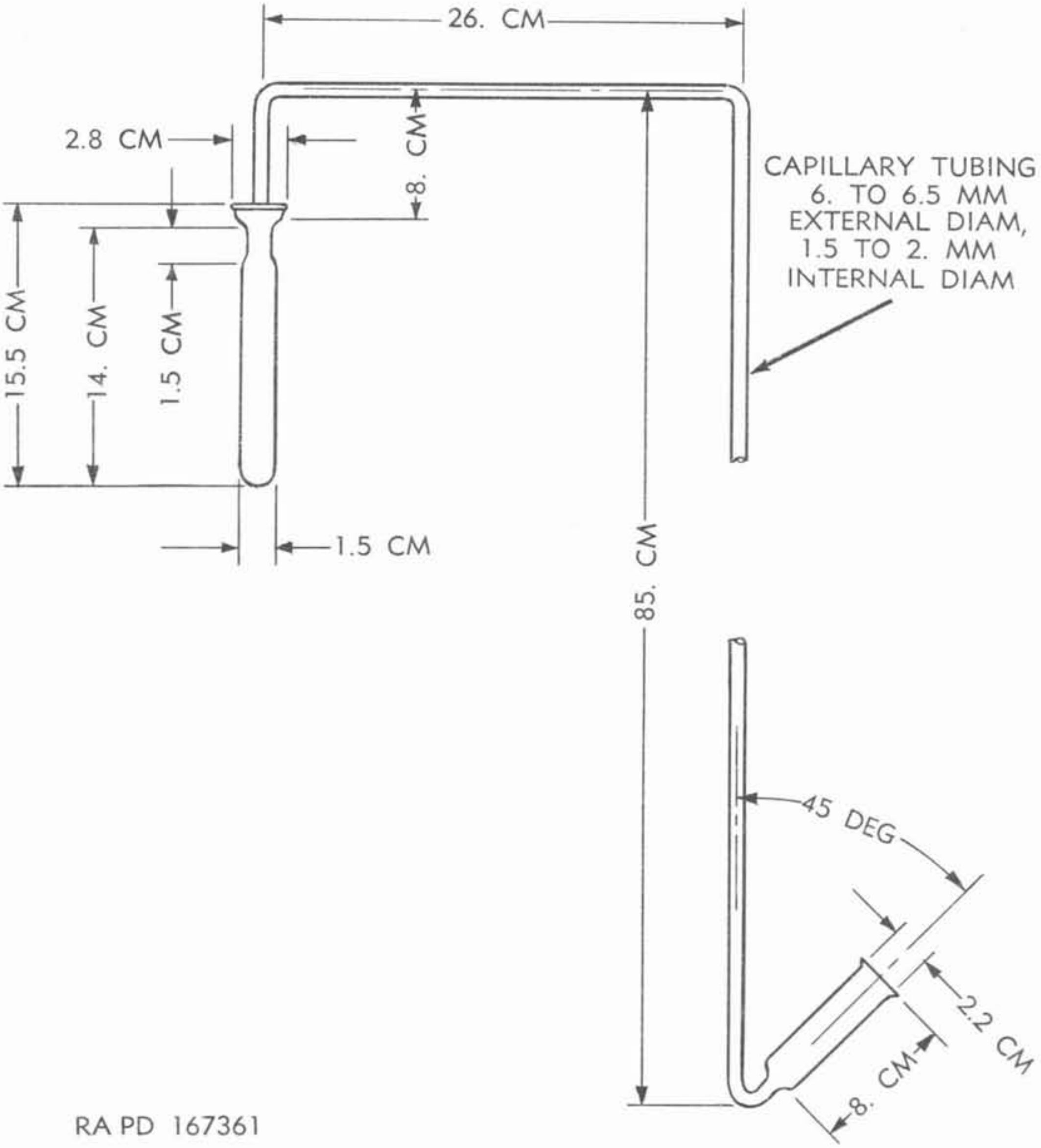
economic reasons; but acceleration of decomposition of this inherently unstable material is retarded for many years by the admixture of a stabilizer that neutralizes the acid products of decomposition as rapidly as they are formed.

d. All of the standard military explosives may be considered to be of a high order of stability at temperatures of 15° to 25° C. (59° to 77° F.), but each has a higher temperature at which the rate of decomposition becomes rapidly accelerated and instability is marked. Mercury fulminate undergoes unduly rapid deterioration at temperatures of only 30° to 35° C. (86° to 95° F.). Nitroglycerin withstands many years of storage at ordinary temperatures, but undergoes accelerated decomposition at 50° C. (122° F.) or more. PETN and nitrocellulose begin to undergo rapid decomposition at less than 120° C. On the other hand, TNT and ammonium picrate withstand heating at 150° C. for 40 hours without significant decomposition and RDX is only slightly less stable at that temperature.

e. For determining the relative stability of high explosives and propellants, there have been developed a number of empirical tests that are based on heating a sample at an elevated temperature for a certain time and measuring the effect of such treatment. The most generally used tests are described in (1) through (4) below.

(1) *75°C. international test:* A 10-gram sample of the explosive is placed in a tared weighing bottle 35 mm in diameter and 50 mm deep, on top of which is placed a watch-glass. The bottle and contents are heated at 75° C. for 48 hours, cooled, weighed, and examined for fumes or decomposition as indicated by appearance or odor. If the loss in weight exceeds any moisture present in the sample, as determined by drying in a desiccator, volatility or decomposition is indicated. This test is applicable to blasting as well as military explosives and is significant only when the material is volatile or unduly unstable. It is of interest chiefly as a preliminary test for new explosives.

(2) *100° C. heat test.* The moisture content of the



RA PD 167361

Figure 5-8. Tubes for vacuum stability test.

explosive to be tested is determined by drying a weighed sample to constant weight in a vacuum desiccator and calculating the loss in weight to a percentage basis. A weighed 0.60-gram portion of the sample is placed in each of two test tubes, 75-mm in length and 10 mm in diameter, one of which has been tared. The tubes and contents are placed in an oven maintained at 100±1° C. After 48 hours the tared tube is removed, cooled, weighed, and replaced in the oven. After an additional 48 hours of heating, the tube and contents are again cooled and weighed. The percentage of loss in weight during each heating period is calculated, correcting the first for the moisture present as previously determined. The untared tube and contents are heated at 100° C. for 100 hours, and any ignition or explosion is noted. This test, because of the high temperature and greater time of test, is more rigorous than the 75° C. international test and it yields data that are more significant with respect to stability and volatility. Nevertheless, like the test at 75° C., it is of interest chiefly in approximating the order of stability of new explosives and does not differentiate sufficiently well between explosives of the same order of stability.

(3) *Vacuum stability test.* This test can be made at 100°, 120°, or 150° C. or any other desired temperature, but a temperature of 100° or 120° C. generally is employed. A weighed 1- or 5- gram sample of the dried explosive is placed in a glass heating tube, so designed that the ground neck can be sealed with mercury after a calibrated capillary tube with a ground stopper end has been connected to the heating tube (fig. 5-8). The lower end of the capillary tube is attached to a cup in which about 7 milliliters of mercury is placed after the connection of the two tubes has been made. The system is evacuated until the pressure is reduced to about 5 mm of mercury. The level of the mercury in the capillary tube rises to near the top, and its exact position is marked and recorded. The junction of the two tubes is scaled with mercury. The heating tube is inserted in a constant temperature bath maintained at the desired temperature ±0.5° C. (fig. 5-9). If an excessive amount of gas (11 + milliliters) is not evolved in less time, heating is continued for 40 hours. The tube is removed from the bath and cooled to room temperature, and the level to which the mercury in the capillary tube has fallen is noted. The volume of gas liberated is calculated from the difference between the initial and final levels, the volume of the capillary per unit of length, the volume of the heating tube, and the atmospheric pressure and temperature conditions at the beginning and end of the test. Vacuum stability tests yield reproducible valves; and when an explosive is subjected to this test at two or more temperatures, a rather complete picture of its chemical stability is obtainable. In some cases tests at two or more temperatures are required to bring out significant differences with respect to stability between explosives, but a test at 100° C. is sufficient to establish the order of stability of an explosive. Vacuum stability tests have been found suitable for determing the reactivity of explosives with each other or nonexplosive materials. This is accomplished by making a vacuum stability test of the mixture and determing if the gas liberated is significantly greater than the sum of the volumes liberated by the two materials when tested separately. When used for this purpose, the test generally is made at 100° C.

(4) *Potassium iodide—starch (KI) test.*

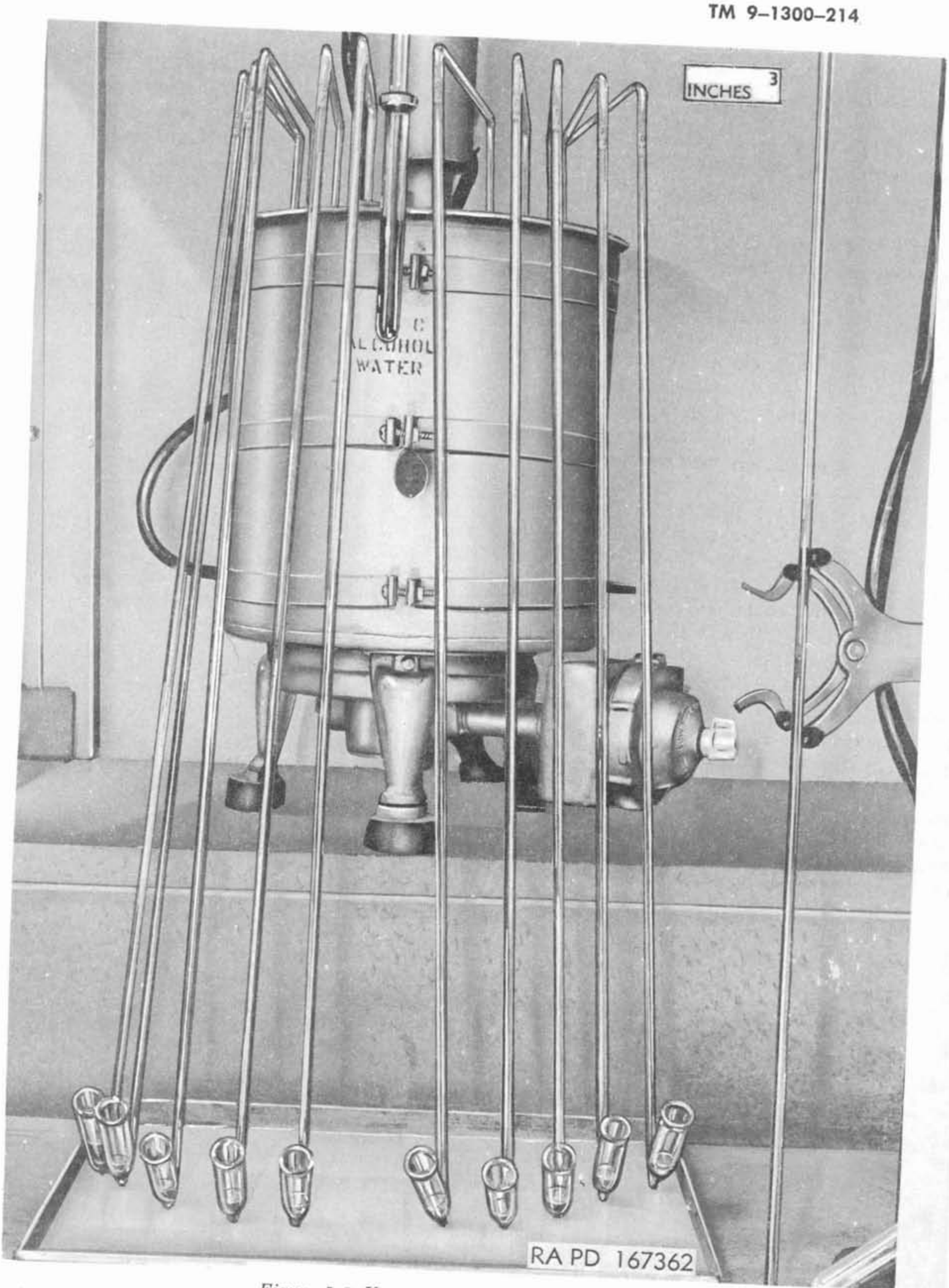


Figure 5-9. Vacuum stability test apparatus.

This test is made at 65.5° C. when applied to nitrocoellulose and at 82.2° C. in the case of nitroglycerin. It is a test for traces of acidity or other impurities that might cause deterioration, rather than a test of the inherent stability of the material. Nitrocellulose is conditioned for the test by drying at 40° to 45° C. on a paper tray until the moisture content has been reduced to such a point that, after rubbing, the material

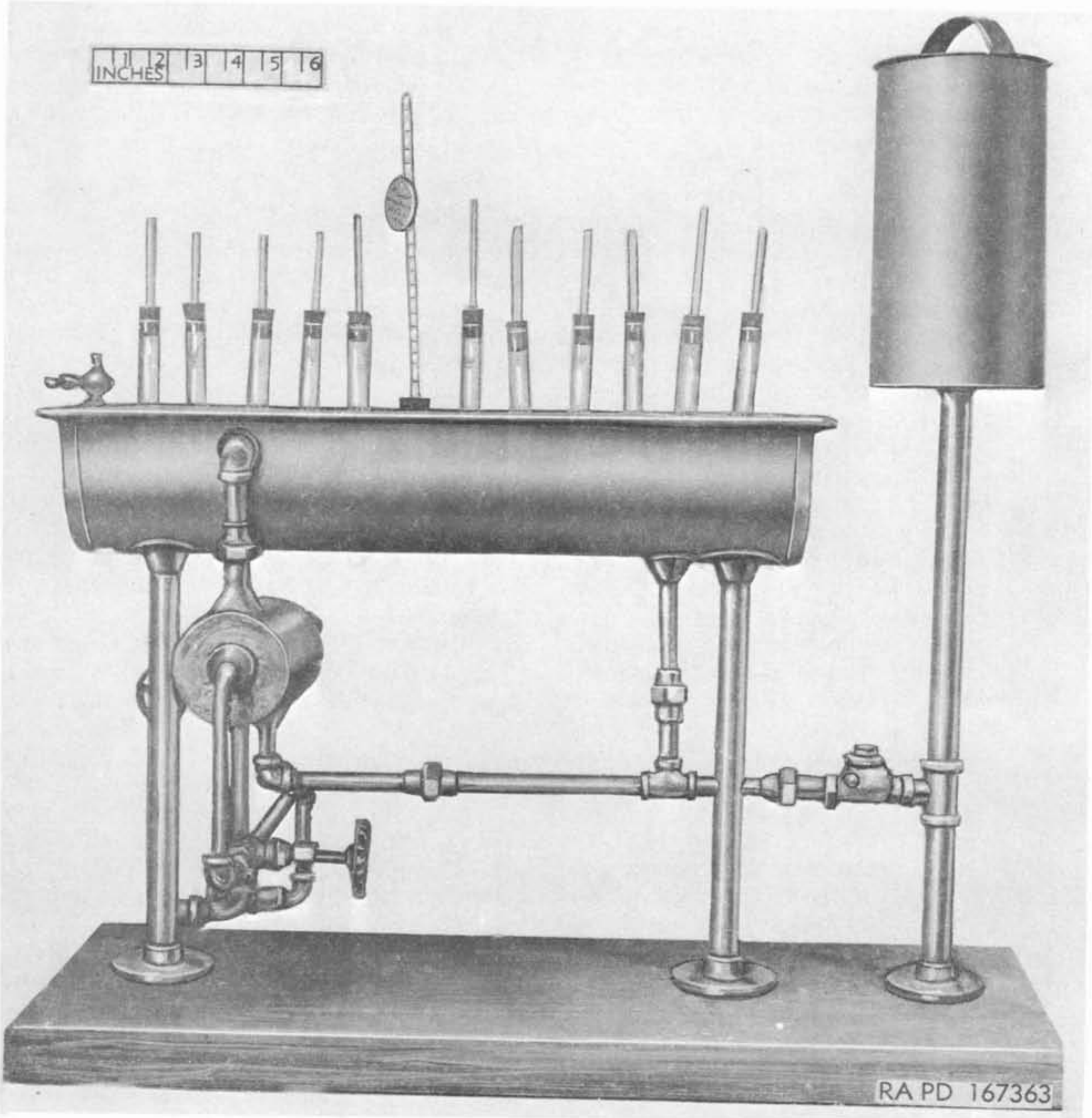


Figure 5-10. KI test apparatus.

clings to a spatula or tissue paper that has been wrapped around the fingers. Nitroglycerin is conditioned by filtering the sample through two thicknesses of filter paper. In testing nitrocellulose, a 1.3-gram portion of the sample is transferred to each of five special test tubes, and, in the case of nitroglycerin, a 2-ml portion is transferred to each of three such tubes. These are 5.5 inches long, 0.5-inch ID, and 0.62-inch OD. Each tube is closed by means of a tightly fitting cork stopper, through which passes a tightly fitted glass rod equipped with a platinum holder for a strip of test paper. The nitrocellulose in each tube is pressed or shaken down until it occupies a space 1.62 inches long. On the platinum holder is suspended a strip of standard potassium iodide—starch test paper approximately 1 inch in length and 0.37 inch wide and the upper half of this strip of test paper is moistened with a 50 percent solution of pure glycerin in water. Each tube is then immersed to a depth of 2.25 inches in a bath maintained at 65.5°±1.0° C. or 82.2°±1.0° C. and placed so that the tubes are viewed against a white background illuminated by bright, diffused daylight (fig. 5–10). The time at which heating is begun is noted. As heating continues, the position of each test paper is adjusted so that the line of demarcation between the wet and dry portions is kept on a level with the lower edge of the slight film of moisture, which condenses on the inside of the tube as the test progresses. The first appearance of discoloration of the damp portion of the test paper in each tube is considered as the end of the test, and the time required to produce this is noted. The minimum time required for completion of a test in a series is considered as the test value of the sample. A blank test is run simultaneously, using an empty tube, and the discoloration taken as indicating a test must be greater than any noted at the same time in the blank test. In making this test, extreme care must be taken to prevent contamination of the sample by the hands, traces of acid in the air, etc. The usefulness of the KI test is chiefly in the direction of assuring completeness of purification during manufacture and freedom from contamination subsequent to this.

f. Stability test values for military high explosives are given in tabular form in appendix A.

5–3. Brisance

a. General. The shattering power of an explosive, as distinguished from its total work capacity, is termed its *brisance.* This characteristic is of practical importance, because it determines the effectiveness of an explosive in fragmenting shell, bomb casings, grenades, etc. As the shattering effect of an explosive is dependent upon the suddenness with which the gaseous products of explosion are liberated, the rate of detonation is at least a major factor in determining brisance. It has been found that there is a general linear relationship between rate of detonation and brisance, as determined by different methods.

b. Sand Test. This test consists essentially in determining the amount of standard Ottawa sand crushed by 0.400 gram of the explosive. In order to accomplish this, the initiator is detonated alone and correction is made for any sand crushed by it. A 0.400-gram charge of the explosive, of such granulation as to pass through a No. 100 sieve, is transferred to each of five empty No. 6 commercial blasting cap shells of copper, gilding metal, or aluminum (see fig. 5–7). The blasting cap shell is held in a loading block (fig. 5–1) during the transfer. If the explosive can be initiated by flame, a reinforcing cap, having a small hole in the center, is placed in the blasting cap over the explosive, the loading block is assembled to a base fixture, a plunger is inserted in the blasting cap, and the charge is subjected to a pressure of 3,000 psi. for 3 minutes by means of a barricaded press. If the explosive charge cannot be initiated by flame, it is pressed without the insertion of a reinforcing cap. A 0.300-gram charge of lead azide is then placed on top of the first charge, a reinforcing cap is placed over the lead azide and this is subjected to a pressure of 3,000 psi for 3 minutes. If the explosive charge is too insensitive to be initiated with maximum effect by lead azide, a 0.100-gram charge of tetryl is placed on top of it and this is pressed at 3,000 psi for 3 minutes. A 0.200-gram charge of lead azide then is added, a reinforcing cap is inserted and the lead azide is subjected to a pressure of 3,000 psi for 3 minutes The powder train in one end of a piece of black powder safety fuse 8 or 9 inches long, is pricked with a pin and this end is inserted in the loaded blasting cap, so that it is against the reinforcing cap. The blasting cap is crimped tightly to the fuse. Eighty grams of standard Ottawa sand, of such granulation that it passes through a No. 20 sieve but is retained on a No. 30 sieve, is poured into the cavity of a bomb (figs. 5–14 and 5–12) and leveled by striking the bomb with a hammer. The loaded blasting cap is lowered into the bomb so that the cap is centered at the axis of the bomb and just touches the sand. An additional 120 grams of sand are poured into the bomb without disturbing the position of the cap. The upper end of the fuse is passed through a hole in the bomb cover, a section of rubber tubing being used to make a tight fit (fig. 5–12). The cover is lowered into position and fastened securely, as shown in figure 5–12. The upper layer of sand is leveled by striking the bomb with a hammer. The fuse is lighted and, after the explosion, the bomb cover is removed. The contents of the bomb are transferred to a piece of glazed paper, cleaning the bomb and cover thoroughly. After removing pieces of the blasting cap and burnt fuse, the sand is transferred to a No. 30 sieve fitted with a bottom pan and cover. The assembly is shaken sufficiently to insure passage through the sieve of all sand crushed sufficiently to do so, and the crushed sand caught in the bottom pan is weighed with an accuracy of ±0.1 gram. If lead azide or tetryl and lead azide was used as an initiator, the amount of sand crushed by the initiating charge is determined separately and subtracted from the weight of sand crushed by the combined charge. The corrected values obtained by the five tests are averaged and the average value is considered representative of the bri-

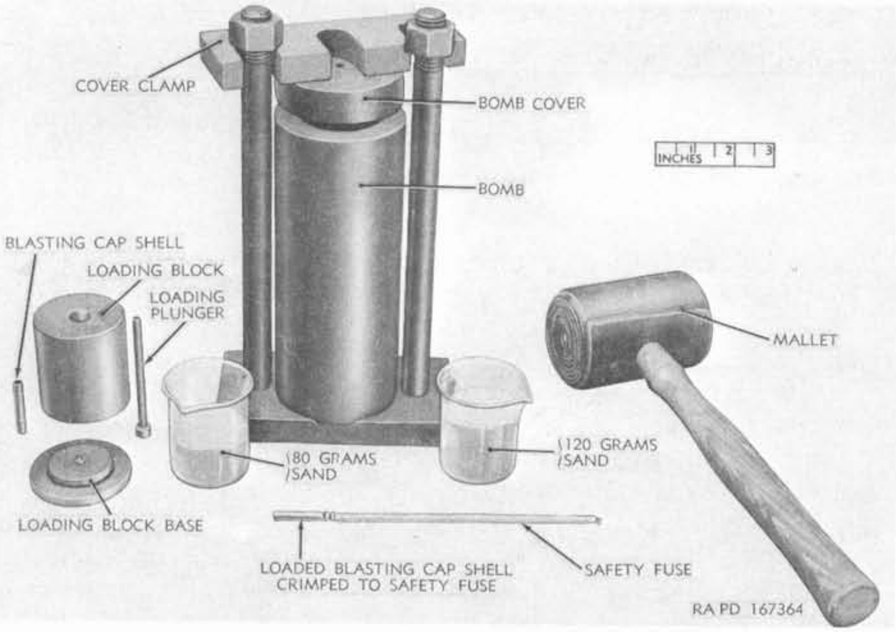


Figure 5–11. Sand test equipment.

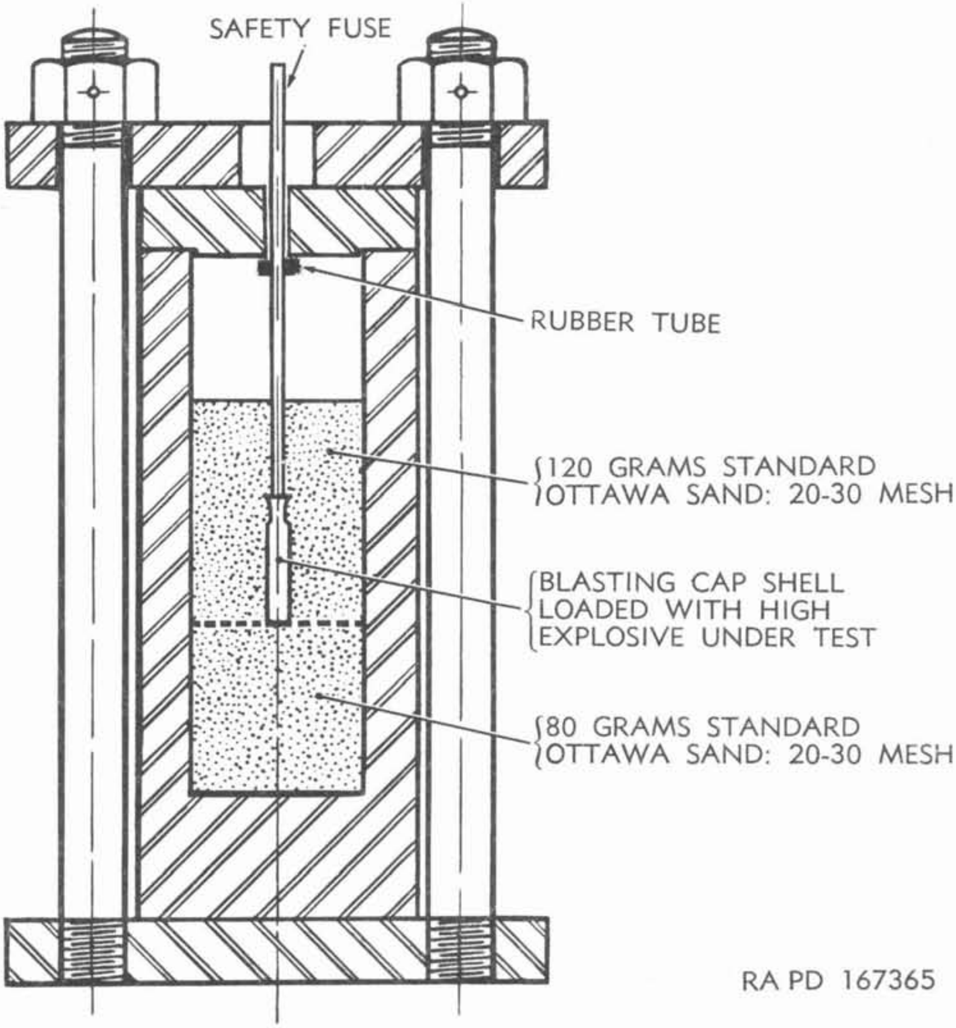


Figure 5–12. Sand test.

sance of 0.400 gram of the explosive under test. Sand test values for military explosives are given in tabular form in appendix A.

c. Plate Dent Test. Although no actual shattering by an explosive is effected and measured in this test, there is produced a deformation of a solid, which investigation has shown to be related to brisance. A 20-gram sample of the explosive is pressed or cast into a piece of copper tubing having an internal diameter of 0.75 inch and a 1/16-inch wall thickness. This tube is placed so as to be normal to the center of a horizontal plate of cold-rolled steel 4 inches square and ⅝-inch thick. The steel plate is placed so as to rest on the upper end of a short piece of steel tubing having internal and external diameters of 1.5 and 3 inches, respectively. This is in a vertical position and rests on a firm base. As finally assembled, the axes of the explosive charge, steel plate, and steel tube coincide. The explosive charge is detonated by means of a No. 8 blasting cap and a 5-gram pellet of tetryl. This produces a depression in the steel plate, having a smooth surface and a depth that can be measured within 0.001 or 0.002 inch. The reproducibility of the test result is within 0.003 inch. Table 5–3 gives the results of tests of a number of explosives.

d. Fragmentation Test. This direct measurement of the brisance of explosives is made by loading a shell or grenade with explosive, detonating the charge, and recovering the fragments. The shell is loaded by casting or pressing the explosive into the cavity. After assembling a booster and a fuze that can be caused to function by means of a current of electricity, the shell is placed in a wooden box. After this has been buried in a sand-pit within a building, the charge is detonated. The fragments are recovered by passing the sand through a 4-mesh sieve and are classified into weight groups of 0 to 75, 75 to 150, 150 to 750, 750 to 2,500 and over 2,500 grains (fig. 5–13). A typical 75-mm shell fragmentation yields about 1,000 fragments. Recovery of the shell, generally, is 90 to 95 percent by weight. Comparisons of explosives loaded in the same size of shell are on an equal volume basis. The total number of fragments produced is determined and compared with the number of fragments produced by a standard explosive. Determination of the number of fragments of certain sizes permits evaluation of the charge, from the viewpoint of effectiveness in causing casualties. A comparison of the relative fragmentation efficiency values of a number of military explosives is given in tabular form in appendix A.

Table 5–3. Plate Dent Tests of Explosives

	Pressed explosive		*Cast explosive*
	Density	*Depth, inch*	*Depth, inch*
TNT	1.50	0.184	0.206
	1.60	0.204	
RDX	1.50	0.252	
	1.60	0.268	
	1.65	0.276	
PETN	1.50	0.240	
	1.60	0.258	
Haleite	1.50	0.230	
	1.60	0.247	
Tetryl	1.50	0.217	
	1.60	0.237	
Picric acid	1.50	0.200	
	1.60	0.217	
Nitroguanidine	1.50	0.177	
Explosive "D"	1.50	0.170	
Composition B			0.241
50–50 Pentolite			0.229
50–50 Ednatol			0.217

5–4. Initiating Value

a. When Nobel first developed nitroglycerin for blasting operations, black powder squibs were utilized for bringing about detonation; but the nonuniformity of initiation led to his developing the mercury fulminate blasting cap. This invention of initiation of detonation by a heat- or shock-sensitive detonating compound has afforded a basis for utilizing more modern and less sensitive high explosives, such as TNT, tetryl, explosive D, RDX, and PETN, since these cannot be caused to detonate by the sudden application of heat furnished by a flame. Even when confined in a blasting cap and subjected to the intense heat of thermite compositions, they do not undergo detonation.

b. When the initiating compounds are considered as a class, it is found that considerable differences exist between them with respect to initiating value. Diazodinitrophenol can detonate ammonium picrate and cast TNT, while lead azide, mercury fulminate, and lead styphnate cannot cause the detonation of either of these materials. Lead azide and mercury fulminate are about equally effective for the initiation of the less sensitive pressed explosives TNT and picric acid, but lead azide is more efficient than mercury fulminate for initiating the more sensitive explosives tetryl, haleite, RDX, and PETN. Lead styphnate cannot cause the detonation of any of these explosives except unpressed PETN, but can initiate certain dynamites. In general, it may be said that the initiating value of a compound of this type depends upon its rate of detonation as used. Mercury fulminate, lead azide, and diazodinitrophenol have rates of detonation of approximately 4,000, 4,300, and 5,500 meters per second, respectively, at the densities used for determining the minimum detonating charges of these required for various noninitiating high explosives (see app. A).

c. Loading operations involving large quantities of the shock and friction-sensitive initiating explosives would be unduly hazardous. This, along with the recognized fact that high rate of detonation results in initiating efficiency, has led to utilization of the *booster charge* in loading blasting caps and military

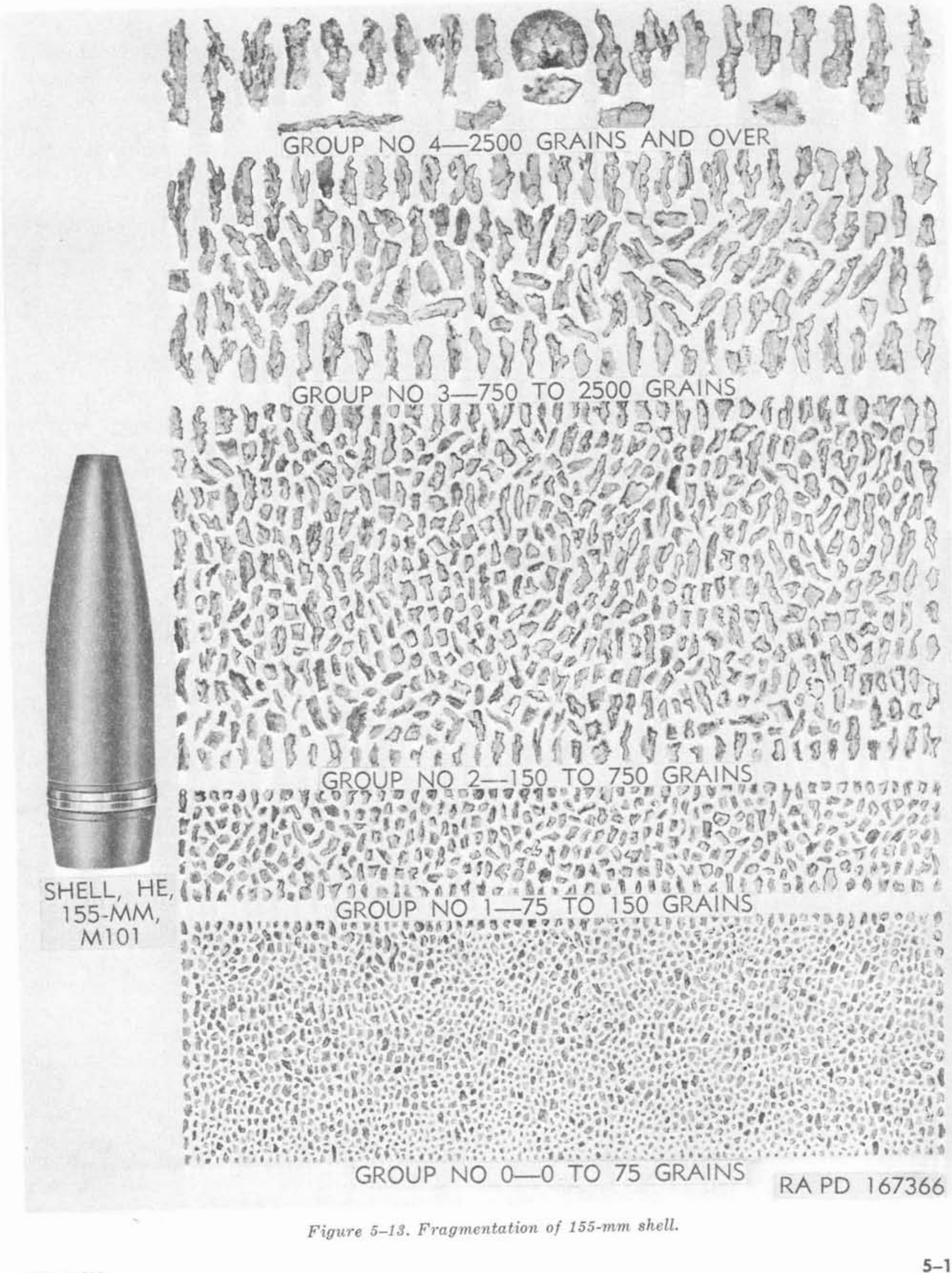


Figure 5-13. Fragmentation of 155-mm shell.

ammunition. Compound detonators and blasting caps have a booster charge of a noninitiating high explosive, such as tetryl, RDX, or PETN adjacent to the lead azide or similar charge. This permits use of less of the sensitive explosive and the development of a detonating wave having a higher rate than that developed when the initiator alone is used. In shell and bombs, the booster principle is applied by the use of a relatively small *booster*. This consists of a steel-encased, cylindrical charge of an explosive such as tetryl inserted in the bursting charge of the shell or bomb. The booster charge is initiated by a detonator inserted at one end. By means of the booster, there is impressed on the bursting charge a detonation wave having a velocity of perhaps 7,500 meters per second instead of that of perhaps 5,000 meters per second obtainable with lead azide. As many of the bursting charge explosives have rates of detonation less than that of tetryl and are less sensitive to initiation than tetryl, the advantage of using a booster charge is apparent.

d. Pressed TNT, which is distinctly more sensitive to initiation than cast TNT, was used as a booster charge for cast TNT and amatol until replaced by the more sensitive and effective tetryl. During World War II, tetryl was replaced to some extent by 65–35 and 70–30 tetrytol, waxed PETN, and waxed RDX. The relative initiating values of such booster explosives can be determined by means of the explosive train test. In this test, an explosive to be boostered is loaded into one end of a steel tube by pressing or casting. One or more disks of half-hard brass, each having a thickness of 0.017 inch and a diameter essentially that of the internal diameter of the steel tube, are placed in the tube and pressed firmly against the end of the explosive charge. On top of the disks is place a booster casing having a cavity with an ellipsoidal cross section and loaded with a weighed quantity of the booster explosive. This is exploded by means of a detonator; and whether the explosive beyond the disk or disks undergoes high order detonation is noted. Repeated tests are made until there is determined the maximum number of disks that can be present and not prevent detonation of the boostered explosive. Five tests are made with this number of disks and five tests with one more disk. The greater the initiating action of the booster explosive, the greater is the number of disks through which detonation can be transmitted. Tests made in this manner have indicated tetryl and RDX to be equally effective and more so than picric acid in initiating other high explosives. The test can be used also to determine the effectiveness of detonators in initiating high explosives.

5–5. Sympathetic Detonation

a. It has long been known that an explosion sometimes results in the detonation of other explosives some distance from the first, without the projection of burning embers or missiles. Experimentation has shown that the distance at which a charge of explosive can be detonated by another charge of the same explosive increases with increase in size of the initiating charge. Such explosion by transmission of a detonation wave through air is known as *sympathetic detonation* or *explosion by influence.*

b. As a detonation wave reaches the end of a column of explosive, the pressure wave is projected into the air beyond, where it undergoes rapid decrease in velocity and pressure to extinction. If this wave traveling through air impinges on another mass of explosive before pressure and velocity have decreased too much, sympathetic detonation ensues. Since the wave traveling through air is not under confinement, expansion of the gases representing the pressure front will be in all directions and the rate of pressure decrease should be as the cube of the distance.

c. Two type of commercial blasting explosives were found to have the relationships shown in table 5–4 for sympathetic detonation.

Table 5–4. Maximum Distances for Sympathetic Detonation

Weight of charge, pounds	*Maximum distance in feet for sympathetic detonation*	
	Explosive A	*Explosive B*
25	2	9.5
50	3	13.5
100	4	19
200	6	21
400	8	26
800	10.5	
1,600	14	
2,400	15	

From these data it appears that the relationship is—

$$\frac{W}{L^{2.25}} = k$$

Where W is the weight of the explosive in pounds, L the maximum distance in feet, and k a constant peculiar to the explosive. The exponent of L has been determined experimentally. For explosive A, k is approximately 4, while for explosive B, it is approximately 0.18. If the maximum distance for sympathetic detonation by a known charge of an explosive is known, the above formula can be used for deriving the k value of the explosive and then for deriving the maximum distance values for other weights of charge.

d. The interposition of solid barriers, such as wood or concrete, decreases the maximum distance for sympathetic detonation of one charge of explosive by another. This effect is very significant when small charges are involved, but is of much less importance with very large charges.

e. Sensitivity to sympathetic detonation or the power to cause sympathetic detonation can be determined by means of the gap test. This was developed for testing dynamites and has been used chiefly in this connection, but can be applied to other high explosives. Cylindrical cartridges of explosive 8 inches in length and 1.25 inches in diameter are made up by pressing or casting equal weights of the explosive into paper shells. Two of these are suspended vertically, so that there is an air space between them and their axial lines are coincident. The air space between the cartridge is always a multiple of 1 inch. A detonator is embedded axially in the lower end of the lower cartridge and used to initiate detonation of this cartridge. By repeated tests, with varying spaces between the two cartridges, there is determined the maximum distance at which the upper cartridge is detonated by the lower. This will be 1 inch less than the minimum distance at which three successive tests fail to result in explosion of the upper cartridge.

5-6. Power

a. General. The total work capacity of an explosive is a function of the total available heat liberated at the instant of detonation, but its power is determined by the rate at which the heat energy is liberated. In the past, measurement was made of the maximum pressure developed by detonation and this was considered an expression of the work function of the explosive. However, such tests were made with explosive charges having relatively low loading density values and it was then necessary to calculate by extrapolation the pressure produced by an explosive in its own volume.

b. Heat of Explosion. The heat of explosion of an explosive can be determined with high density of charge in a calorimetric bomb. The procedure is similar to that used for determining heat of combustion, except that no oxygen is used and detonation instead of combustion is insured by the use of a detonator. After the bomb and contents have cooled to room temperature, the volume of gas and the weight of

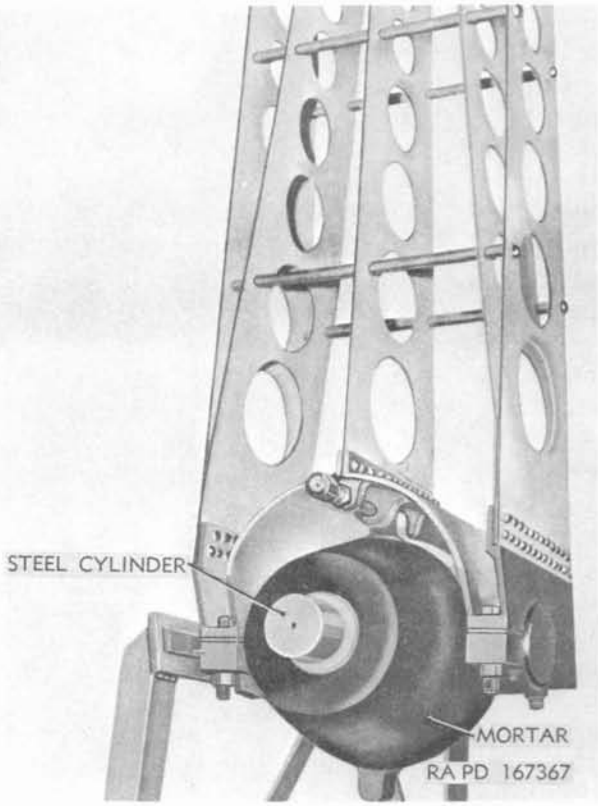


Figure 5-14. Ballistic pendulum—front view.

water produced are measured. The observed heat of explosion is corrected by subtracting the heat of condensation of the water produced. The measured volume of gas is corrected by adding the volume at standard temperature and pressure of the water formed when in a gaseous condition. The corrected heat of explosion, expressed as calories per gram, is considered to represent the useful work capacity of the explosive in fundamental terms. Such values for the various military explosives are given in tabular form in appendix A.

c. Ballistic Pendulum Test. A simpler but empirical test measuring the relative power of an explosive is the ballistic pendulum test, which utilizes the principle of equality of physical action and reaction. The equipment (figs. 5-14 and 5-15) consists of a pendulum bar suspended on knife edges and supporting a steel mortar that weighs 622 pounds. The outer chamber of the mortar has a diameter of 4.875 inches and a length of 5 inches. The inner or explosion chamber is 2 inches in diameter and 5.5 inches in length. In the outer chamber, there is placed a loosely fitting steel cylinder that weighs 36.6 pounds and has an axial perforation large enough to permit the passage of detonating fuze or electric detonator wires. A quadrant, graduated in degrees, is arranged so that any swing of the mortar may be measured. Ten grams of TNT is formed into a cartridge by wrapping in tin foil, an electric blasting cap being located centrally in the charge. The charge is placed in the explosion

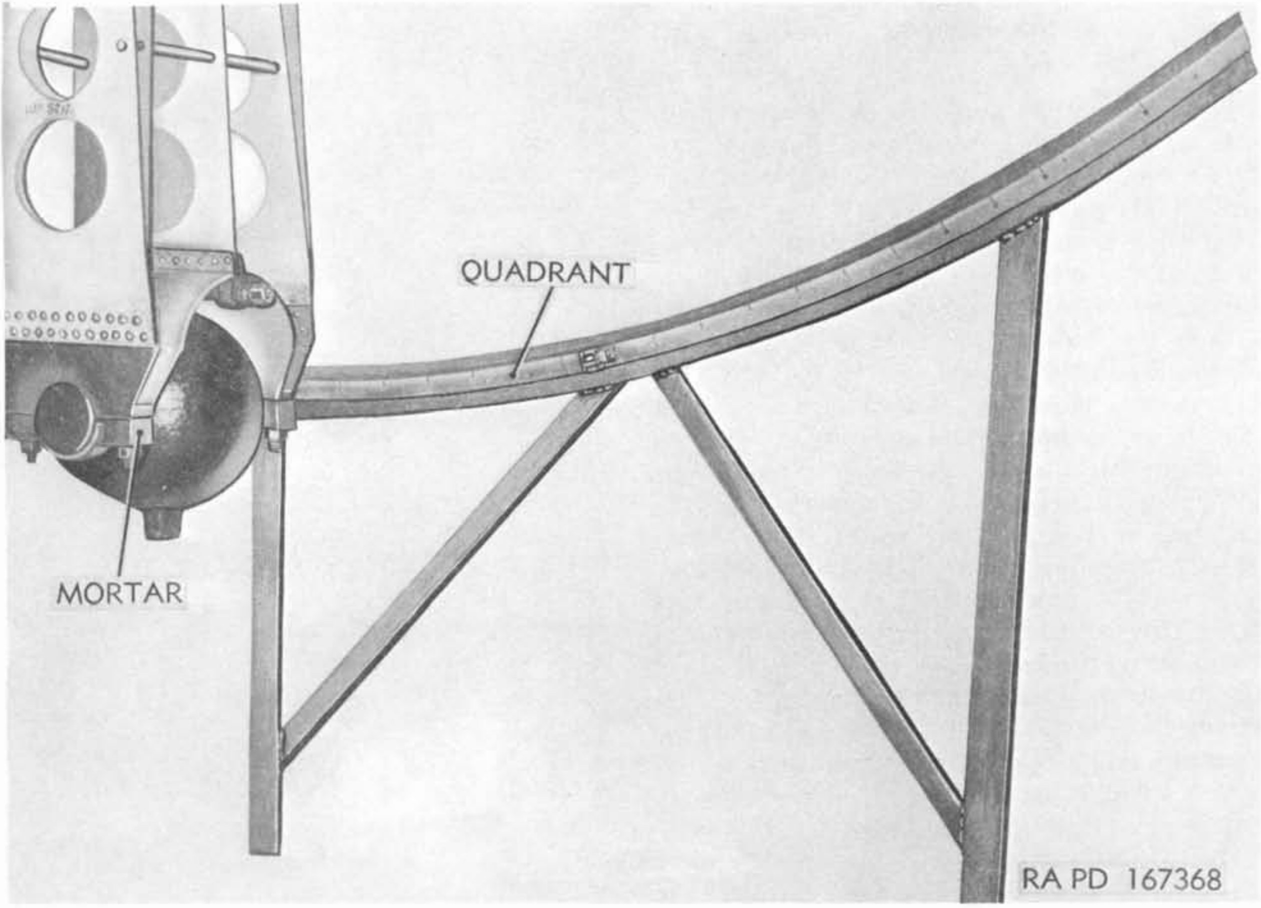


Figure 5-15. Ballistic pendulum—rear view.

chamber and detonated after the steel cylinder has been placed in the outer chamber. The maximum deflection or swing of the mortar is measured in degrees. The results of several such tests with TNT are averaged. By trial, determination is made of the number of grams of an explosive to be compared with TNT, which causes a deflection within 0.5 degree of the average value for 10 grams of TNT. Several more tests of the explosive under comparison are made with the same weight of charge, and the angular deflection results are averaged. From the average values for TNT and the other explosive and the weights of these used, the weight of the second explosive required to cause the same deflection as 10 grams of TNT is calculated. When 10 is divided by this, the relative TNT value of the second explosive is obtained. Ballistic pendulum test values of various explosives are given in tabular form in appendix A.

d. Trauzl Lead Block Test. This test measures the distension effect when 10 grams of an explosive under moderate confinement is detonated in a lead cylinder (fig. 5-16). The cylindrical block is cast of pure lead, so as to be 200-mm long and 200-mm in diameter, with an axial hole 125 mm long and 25-mm in diameter at one end. The volume of the hole is determined before use in the test by filling with water from a graduated burette. A 10-gram sample of the explosive is wrapped in a piece of tin foil of trapezoidal shape, with a width of 70-mm and sides 130 and 150-mm long, so as to make a cartridge 22 mm in diameter. An electrical blasting cap is placed centrally in the charge, which then is placed at the bottom of the hole in the test block. Forty milliliters of Ottawa sand are placed in the hole and tamped lightly. Forty milliliters more of sand are added and this is tamped much more thoroughly. After the charge has

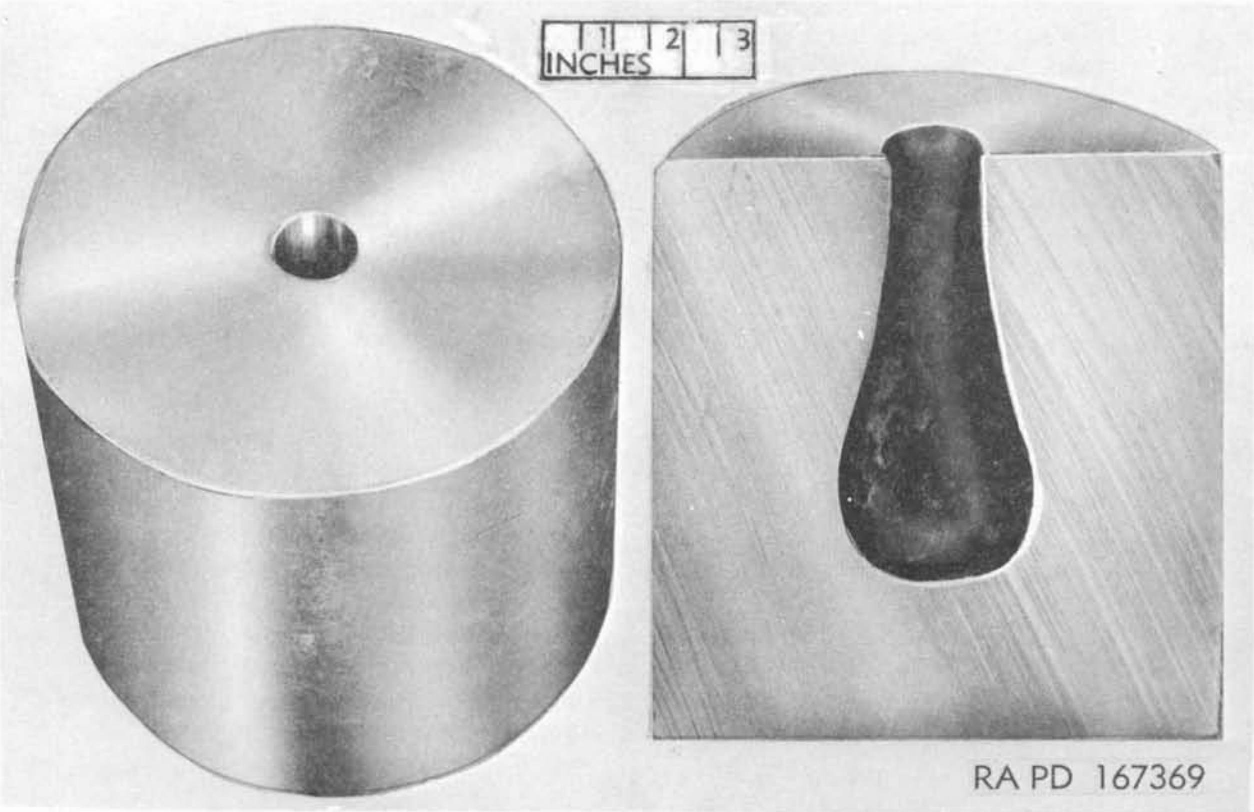


Figure 5-16. Trauzl lead block test.

been detonated, the volume of the hole is determined; and the distension is calculated by subtracting from this the volume of the hole before the detonation. Three such tests are made and the results are averaged. Test values for explosives are given in tabular form in appendix A.

e. Correlation of Test Values. If the heat of explosion, ballistic pendulum test, and Trauzl lead block test values from a number of explosives are compared with each other, a general agreement can be seen; and if the data are plotted against each other, approximate curves can be obtained. However, each of the tests has shortcomings and the results of two or more tests are required for proper evaluation of the relative power of an explosive. Heat of explosion is not strictly representative of power, because of the varying added effects in use of after-burning of some of the products of detonation. Ballistic pendulum test values may not be quantitatively proportional to power, because of the measurement of angular rather than linear displacement of the test mortar. Evidence has been found that, because of the relatively light confinement, the distension effects of the most powerful explosives in the Trauzl lead block test are not directly proportional to power. Nevertheless, the three tests do afford a means of evaluating relative explosive power when all three test results are available.

5-7. Blast Effect

a. More serious damage can be done to industrial installations, habitations, etc., by the blast effect of bombs than by their fragmentation. It was found that the blast effects of a bomb are comparable with those of a severe earthquake, since walls are overthrown, roofs collapsed, machinery thrown out of line, etc. This action is more or less uniform over a circular area; whereas, damage done by bomb fragments consists merely of holes in walls and roofs, damage to machinery at the point of impact, etc., at random points in the lines of travel of the fragments. The discovery that certain explosives are markedly superior to TNT and amatol, with respect to blast effect, permitted the use of bombs with maximum blast effect and afforded a basis for the bombing operations that represented one of the major tactical advances of World War II.

b. When a bomb charge detonates in approximately 0.0001 second to form gases at pressures of the magnitude of 700 tons per square inch and at temperatures from 3,000° to 4,500° C., the metal casing of the bomb expands very rapidly to approximately 1.5 times its original diameter before breaking into fragments. Of the total energy liberated by the detonation of the charge, as much as half may be used for expanding the casing prior to rupture and imparting velocity to the fragments of the casing. The remainder of the available energy is expended in compression of the air surrounding the bomb and is responsible for the blast effect produced.

c. Upon release from confinement by fragmentation of a bomb casing, the gaseous products of detonation expand suddenly and compress the layer of surrounding air, so that it is under high pressure and moves outward with high velocity. This layer of compressed air moving outward is bounded by an extremely sharp front less than 0.001 inch thick, called the *shock front,* in which the pressure rises abruptly. The shock front moves outward with an initial velocity much greater than that of sound (1,100 fps), but, after advancing a short distance, the velocity decreases rapidly and the abruptness of the pressure rise also decreases. This decrease in velocity, generally, is much greater than the decrease in velocity of the casing fragments caused by air friction. Consequently, at some point, the advance of the shock front lags behind the movement of the fragments. This difference between the changes in velocity is due to a fragment having constant cross-sectional area, while the surface area of the expanding shock front increases in accordance with the equation—

$$A = 4\pi r^2$$

where r is the distance from the point of detonation to the shock front.

d. The gaseous products of detonation, moving outward as a strong wind behind the shock front, are prevented by their own inertia from decreasing in velocity as rapidly as the pres-

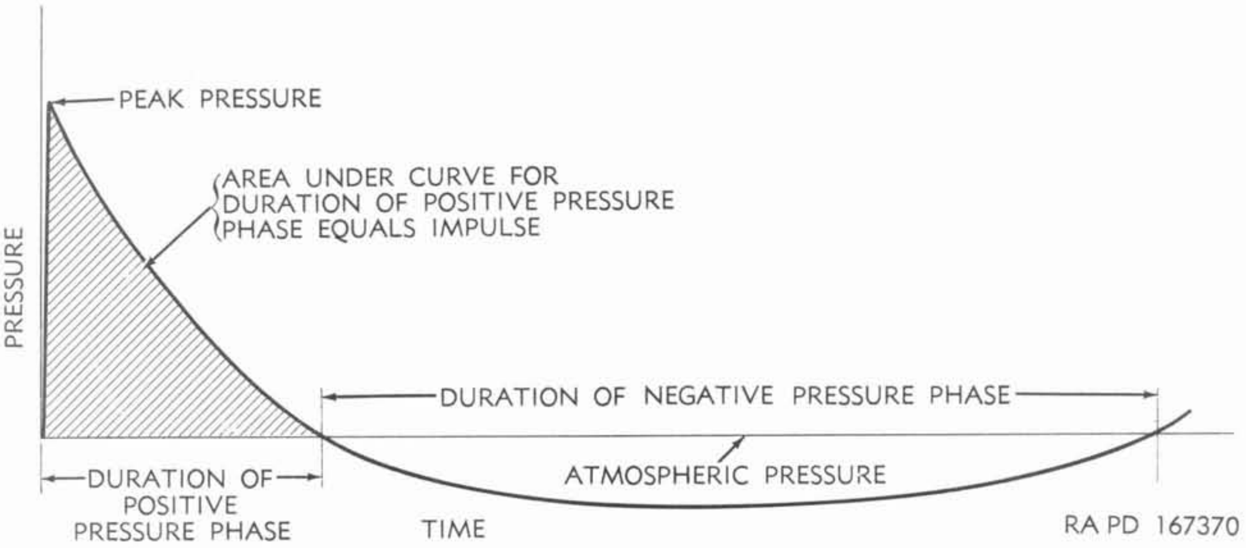


Figure 5–17. Phases of a blast wave.

sure at the point of detonation decreases. As a result, there is produced a rarefaction effect at this point and the pressure decreases. This condition of reduced pressure also moves outward, trailing the shock front. When the pressure becomes less than atmospheric, the wind reverses in direction and blows backward toward the point of detonation. Any light object, such as a leaf, when struck by the shock front, is rapidly carried away from the point of detonation and then is blown back almost to its original position when the wind reverses. The shock front, the high pressure area behind it, and the following rarefaction form a complete wave, to which the terms *blast* and *shock wave*, generally, are applied (fig. 5–17).

e. While the duration of the positive pressure portion of the blast wave is about one-fourth that of the portion at less than atmospheric pressure, the pressure at the shock front is many times the decrease in pressure below atmospheric. The duration of the positive phase varies with the explosive charge and its distance from the point of origin. In one case, it is of the order of 0.006 second at a distance 50 feet from the bomb. A wall struck by such a blast wave is first pushed forward by the short, sharp, hammer-like blow of the positive pressure phase and then pulled backward by the much longer negative phase. Whether the wall falls forward or backward depends upon circumstances.

f. In expressing the blast effect of an explosive charge, it is necessary to use two criteria instead of one—*peak pressure* and *impulse*. Peak pressure is the pressure increase at the shock front or the highest pressure in the shock wave minus atmospheric pressure. Impulse is mathematically equal to the area under the time-pressure curve for the duration of the positive phase (fig. 5–17). This is approximately half the peak pressure multiplied by the duration of the positive phase. Peak pressure represents a measure of the maximum force exerted against a surface by a blast wave, since force is equal to the product of pressure and area. Impulse represents a measure of the force multiplied by the duration.

g. As the shock front moves outward, the peak pressure decreases and the duration of the positive pressure phase increases. The rates of these changes vary with the magnitude of the peak pressure, but the net effect is decrease in impulse. With increase in distance from the bomb, impulse decreases approximately as $\frac{1.}{R}$

h. In general, two criteria must be satisfied in order to demolish any structure. Both peak pressure and impulse must exceed certain minimum values, which depend upon the type of structure. Window glass requires a moderately high peak pressure but only a low impulse value, since the positive pressure need not last long to cause fracture. On the other hand, a brick wall withstands only a small peak pressure, but the pressure must be of relatively long duration and therefore the impulse value must be high. The blast from almost any charge that develops sufficient peak pressure will have sufficient impulse to break glass. Most bomb charges that develop sufficient impulse to demolish a brick wall also develop sufficient peak pressure to do so. It may therefore be said that structures that are strong and light in weight, with respect to the area presented to the shock front, require high peak pressure but no great impulse for demolition; while heavy but relatively weak structures require considerable impulse but not such a high peak pressure.

i. Blast waves are reflected from solid surfaces, but not in the same manner as sound and light waves are reflected. When the incident and reflected waves intersect on the ground, they do not make equal angles with the ground, as do sound waves. At small angles of incidence (the angle between the normal to the blast front and the normal to the reflecting surface), the angle of reflection is smaller than the angle of incidence, and the pressure behind the reflected wave is greater than behind the incident wave. Such reflection of a blast wave from a bomb that has detonated above the ground gives rise to an increased pressure wave at ground level. When the angle of incidence (α) is greater than an extreme angle (α_e) (40° for strong to a value approaching 90° for very weak shocks), the incident and reflected waves do not intersect on the ground. This is known as irregular or Mach reflection. Reflection that occurs when the angle of incidence occurs at the extreme angle is known as the extreme regular reflection, whereas, reflection occurring where the angle of incidence is smaller than the extreme is known as regular reflection (fig. 5–18). When the incident angle exceeds the extreme angle, the reflected and incident waves intersect at some point above the ground. The lower portion of the reflected and the incident waves fuse into one wave, which is known as the Mach stem (M, fig. 5–18). The point of intersection being known as the triple point (TP, fig. 5–18). The resulting

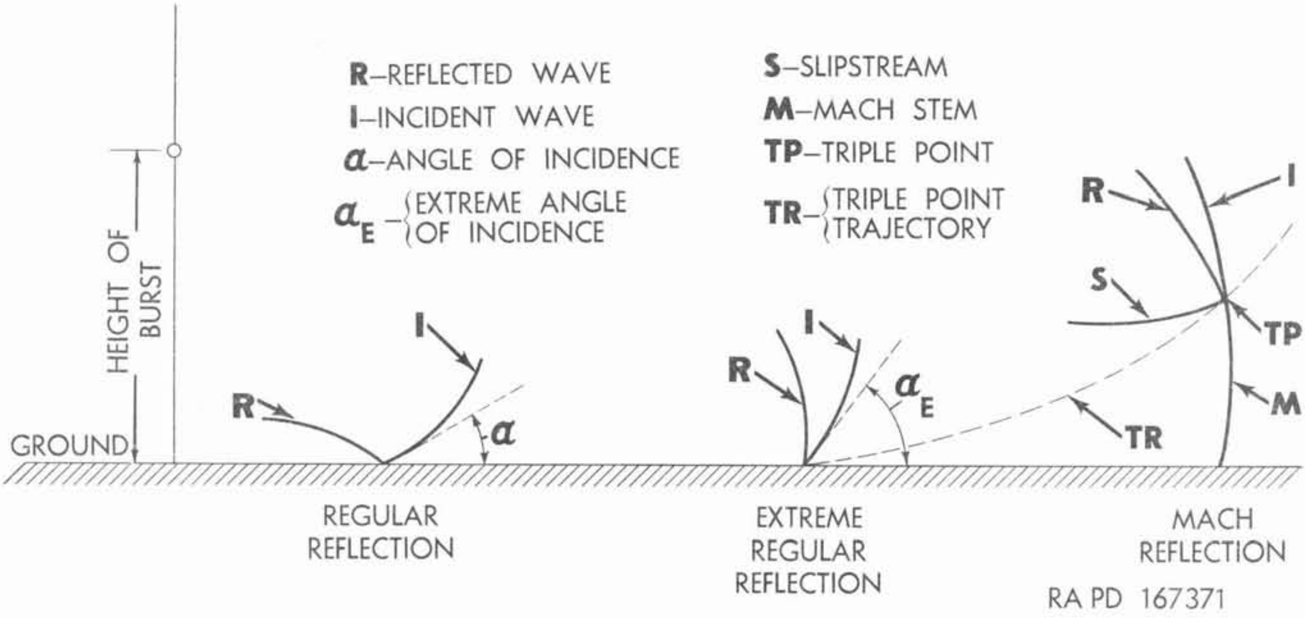


Figure 5–18. Mach reflection of blast wave.

existence of three waves, termed the Mach reflection, causes a density discontinuity and there is formed a region of high pressure termed the Mach region. The pressure behind the front of the Mach region is approximately twice that of the incident wave. The top of this high-pressure region, the triple point, travels away from the reflecting surface. As pressure and impulse appear to have their maximum values just above and below the triple point, respectively, the region of maximum blast effect is approximately that of the triple point. By controlling the height above ground at which a bomb is detonated, it is practicable to control the region of maximum blast effect and insure maximum damage. At distances that are large, compared to the height of the burst, the incident and reflected waves have fused and proceed outward as a single shock. After the point of complete fusion of the waves, the shock wave appears as though it had come from double the charge detonated on the ground, and at large distances, the Mach stem in the neighborhood of the ground becomes perpendicular to it.

j. Blast effects are enhanced by confinement, due to the reflection of blast waves by the confining surfaces. A blast wave traveling through a tunnel, corridor, trench, or even a street decreases in intensity much more slowly than in the open. If a bomb detonates within a building, there is considerable reflection of the blast wave from the walls, even if these are demolished. The rapid reflection of the wave from various walls, in such a case, results in a multiple punch effect on another wall. The overall effect of confinement is increased in the *vulnerable radius of demolition* of the bomb charge. The analogous *vulnerable radius of visible damage* (as detected by aerial observation) is also used to evaluate the effects of bombing.

k. The underwater and underground shocks produced by an explosive charge are more comparable with its blast effect without confinement than with confinement.

l. The TNT and binary explosives used for loading bombs do not contain sufficient oxygen to oxidize all their carbon to carbon dioxide and hydrogen to water and so do not have maximum liberation of heat. It has been found that the oxygen of the atmosphere reacts with the gaseous products of detonation and so increases the heat liberated, although the amount of oxygen utilized in this manner is never as much as that required for complete oxidation. On this basis, there have been developed explosives containing metallic aluminum in finely divided form, such as tritonal and torpex. The aluminum in these increases the blast effect of the charge by highly exothermic afterburning.

m. Relative blast efficiency values for various explosives used in bombs are given in appendix A.

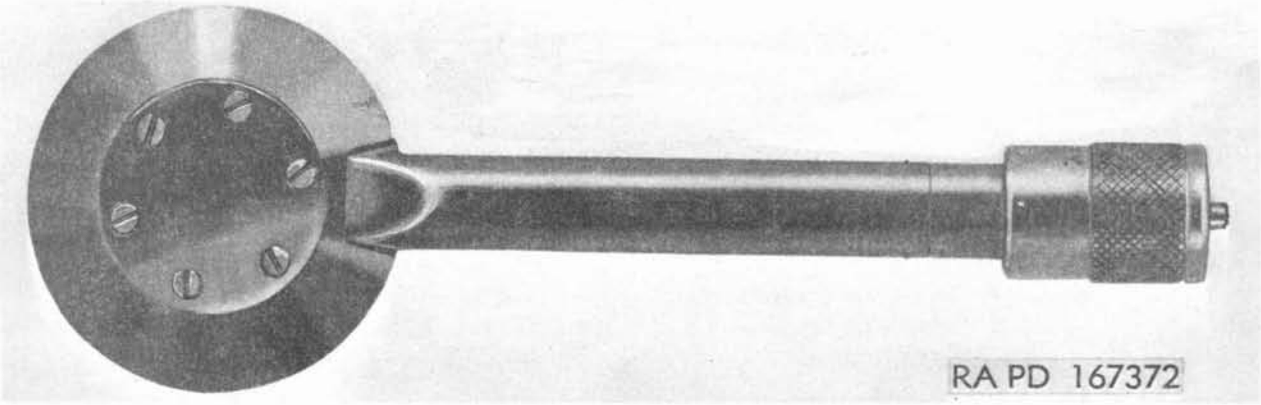


Figure 5–19. Piezo-electric gage for measurement of blast pressure.

5-8. Cratering Effect

a. If an explosive is buried deeply in the earth and exploded, pressure is exerted almost entirely on the earth about it and causes movement effects over a distance known as the *radius of rupture*. If the depth of burial is less than the radius of rupture, the gaseous products of explosion blow through the surface of the ground and there is formed a roughly circular depression known as a *crater*. The crater is considerably greater in volume than the explosive charge producing it. The apparent depth and diameter of the crater are determined by the amount of loose earth that falls back into the cavity and around the edge of the true crater. The true depth is the distance from the original ground level to the bottom of the crater when loose earth is removed. The true diameter is the distance between opposite edges of the crater at original ground level when loose earth is removed.

b. An explosion on the surface of the ground produces a shallow crater, which may have a greater diameter than that produced when the explosive is buried. This is due to the scouring action of the gases projected downward from the explosive charge.

c. In addition to the size of the explosive charge, determining factors in crater formation are the type of ground cratered and the type of explosive used. A soft, low-density soil is lifted, scoured, and blown away more easily than a firm or rocky earth formation. An explosion of a moderate charge in soft, swampy ground produces a relatively huge crater, while in a rock formation only a small crater will be formed. An explosive with a low rate of detonation, that produces a large volume of gases may dig a larger crater than TNT, if the charge is well below the surface and the ground is not too hard and rocky. If the charge is on the surface of the ground, a low-rate explosive produces a smaller crater than TNT, because of the lesser scouring action of the gases resulting from their lower velocity.

d. The volume of a crater in cubic feet, V, produced *on* average soil by commercial dynamite, where Q is the weight of dynamite in pounds, is given by the formula—

$$V = 0.4\, Q^{\frac{8}{7}}$$

This fits the production of a crater 31 feet in diameter and 9 feet in depth (3,200 cubic feet) by a 2,400-pound charge of dynamite. By way of contrast, a 4,000-pound bomb containing 2,400 pounds of high explosive has produced a crater 60 feet in diameter and 20 feet deep (26,000 cubic feet). The explosion at Oppau, Germany of 9,000,000 pounds of ammonium nitrate produced a crater 400 feet in diameter with a true depth greater than 90 feet. Because of the low rate of detonation of ammonium nitrate, the volume of the crater was but 10,000,000 instead of the 75,000,000 cubic feet indicated by the above formula. Military bursting charge explosives, therefore, are much more effective cratering agents than commercial blasting explosives, and the above formula is not applicable to them.

e. When the explosive is buried in the ground at a depth greater than the radius of rupture, the compression effect downward and horizontally and the lifting upwards with subsequent subsidence results in a cavity in the surface known as a *camouflot*. If a proper charge of TNT is buried so that a *common crater* is formed, the ratio of the true diamter of the crater in feet, D, to the depth of burial in feet, L, will be two. Based on experience, such crater formation can be estimated by the formulas—

$$L^3 = \frac{2\,W}{Z} \text{ and } D^3 = \frac{16\,W}{Z}$$

where W is the weight of TNT in pounds and Z is a constant having values of 0.054, 0.066, 0.084, and 0.10 for light earth, common earth, hard sand, and hardpan or heavy clay, respectively. If W is calculated from either of these formulas, an overcharge is added, this being 100 percent for 25 pounds, 50 percent for 25 to 100 pounds, 25 percent for 100 to 250 pounds, and 10 percent for more than 250 pounds. Use of an explosive other than TNT requires further modifications of the formulas.

f. Use of cratering operations in the field has led to the development of special cratering explosives. These are not to be confused with

Figure 5-20. Earth crater from explosion.

the explosives having high rates of detonation used for producing earth shocks.

g. As cratering effect is considered to measure to some extent the demolition effects of general purpose bombs, these are sometimes general purpose bombs, these are sometimes tested for size of crater produced. The formula—

$$V = 4.13\ W$$

where V is the volume of the crater in cubic feet and W is the weight of the explosive component in pounds, has been found to hold for 500 and 1,000-pound bombs loaded with TNT.

5-9. Munroe Effect

a. The practical application of the blast effect and Munroe effect of explosives represent the major advances in the use of explosives during World War II. While the blast effect was utilized to enhance demolition at a distance, the Munroe effect, through the use of shaped charges, enabled the concentration of explosive action, so as to have great effect in penetrating steel armor, concrete, etc.

b. In 1888, C. E. Munroe discovered that if a block of nitrocellulose with letters countersunk into its surface is detonated with its lettered surface against a steel plate, the letters are indented into the plate. On the other hand, if the letters were raised above the surface of the nitrocellulose, the detonation of this repro-

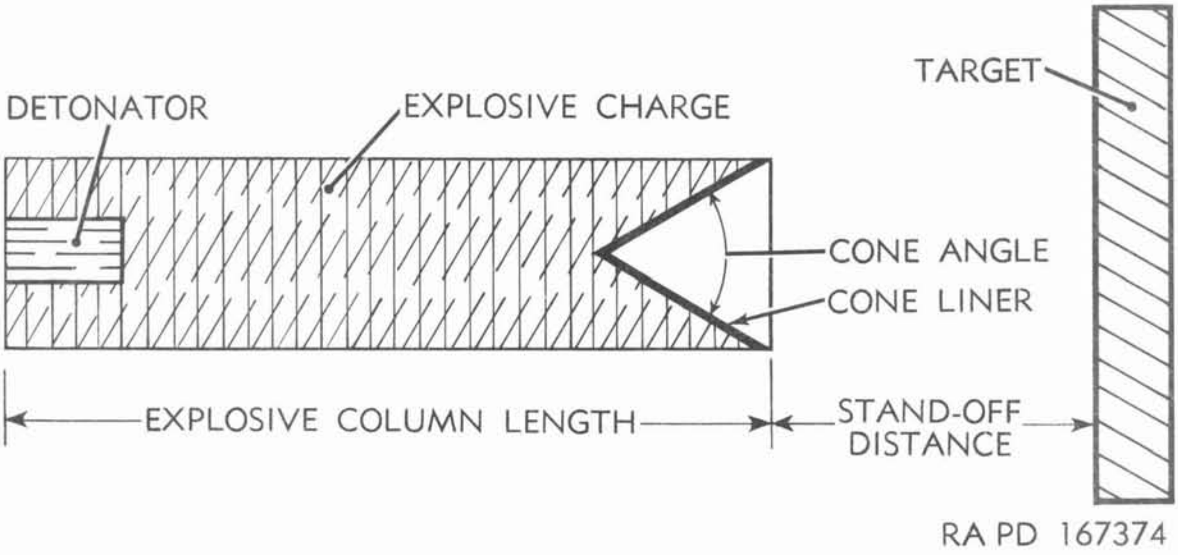


Figure 5-21. Shaped charge and target.

duced the letters in relief on a steel plate. From this it can be seen that the greatest effect is produced where the explosive is not in direct contact with the steel plate. This might be considered as showing the enhanced effects of detonation waves from different directions meeting and reinforcing each other. Munroe found that by increasing the depth of the depression or cavity in the explosive he was able to increase its effect on a steel plate. In spite of later investigaitons by other workers, it was not until World War II that military use was made of the principle. The end of that conflict found it in use by all the warring nations.

c. As used, a shaped charge consists of the essential parts shown in figure 5-21. The cone liner may be of metal, glass, or other inert material. If no cone liner is employed, such a charge has essentially the same effect as a lined charge, if the two are in direct contact with the steel target. In such a case, the action of the unlined charge is ascribed to not only incident and reflected shock waves, but also to a third particular type of shock wave formed as a result of the collision of shock waves coming from the conical wall of the cavity. The action of such a charge, then, is that of a mass of dense gas moving with great velocity. On the other hand, the focused disruptive action of a lined charge in contact with a steel target is due to the jet formed by the solid, collapsed liner. If detonation of unlined and lined charges takes place with the charge at an appropriate distance from the target, the unlined charge is less effective, while the effectiveness of the lined charge is increased. Under such conditions the effect of the cavity in the unlined charge merges into the general blast effect of the charge.

d. The mechanism of the lined charge appears to be that of collapse of the liner, due to the reflection and focusing of the detonating wave as it becomes incident to the cone wall. If the stand-off distance is optimum, collapse of the liner can be complete, before it reaches the target. In figure 5-22, A shows the initial state of a cone of 40 degrees angle, B the beginning of collapse of the cone, and C further collapse. In the next phase, D, a small jet emerges from the base of the cone while collapse continues. As shown by E, there follow elongation of the jet and complete collapse of the cone. As shown by F, the jet from a steel cone then breaks up into small particles followed by the *slug* or major portion of the complete collapsed cone. The mechanism of collapse

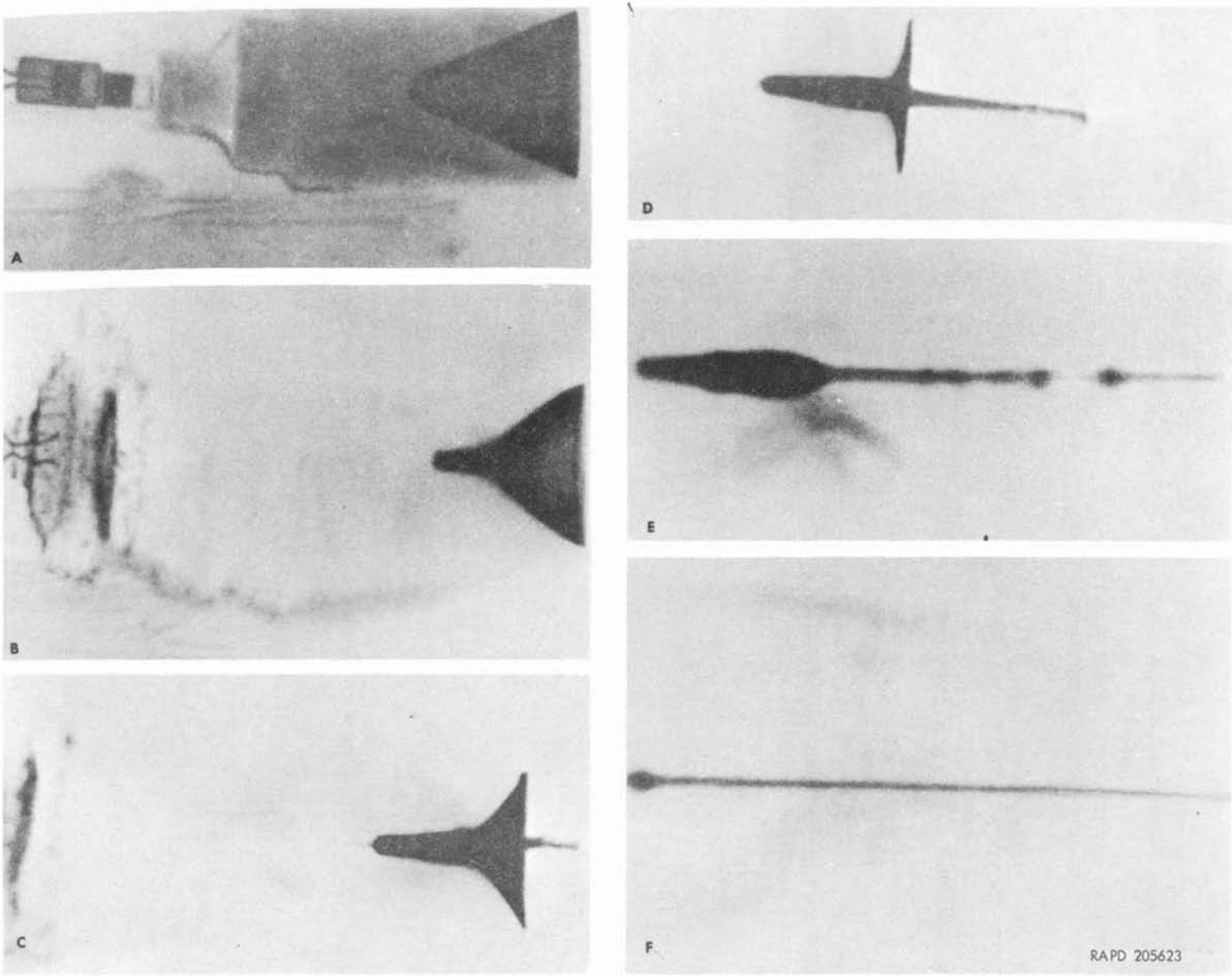


Figure 5–22. Collapse of shaped charge liner.

of liners of large apex angle or hemispherical shape probably differs somewhat and resembles turning inside out.

e. The long, narrow jet of moving particles ejected along the axis of the cavity has a velocity gradient from the tip to the rear, the particles at the tip moving fastest and the slug at a relatively low rate. Small portions of the jet maintain high velocity over a relatively long range. The jet particles, and not the slug, constitute the penetrating agent. The high-velocity jet particles exert pressures on the target measured in terms of several hundred thousand atmospheres. Such pressures so greatly exceed the yield strength of the target material that it is literally pushed aside from the path of the jet by plastic flow. Even though material from the jet is captured by the target the diameter of the hole produced is always greater than the diameter of the jet.

f. The penetrating action of a shaped charge is affected by a number of factors. The explosive used is of great importance; and, while the depth of penetration is indicated to be more closely related to the detonation pressure than the rate of detonation, in general, the greater effect is produced by the explosive having the greater rate of detonation. Very little effect is produced by explosives having rates of detonation of 5,000 meters per second or less. TNT has a relatively low rate of detonation; and other castable explosives with greater rates of detonation, such as composition B, pentolite, and ednatol are much superior to TNT when used in shaped charges.

g. The type of material used as a liner and its thickness are important. Increase in thickness of the liner up to 1-mm for charges about 1⅝ inches in diameter causes increase in depth of penetration, but further increase in thickness has no significant effect on depth of penetration until it reaches a critical thickness, above which performance deteriorates. Comparative test with liners of different metals gave results (table 5-5) that indicate that, in general, the depth of penetration is greatest with metal of the greatest density. However, ductility also plays a major role in penetration.

Table 5-5. Effect of Liner Material on Shaped Charge Efficiency

Liner material	*Density*	*Depth at optimum stand-off* (in)*	*Diameter of entrance hole (mm)*
Copper	8.8	7.5	18
Steel	7.8	5.0	16
Zinc	7.2	4.5	22
Aluminum	2.7	5.0	23

*In armor-plate targets.

As the depth of penetration decreases and the diameter of the hole increases, the volume of the hole is practically unchanged. The hole volume correlates with the total energy of the explosive.

h. The *stand-off distance,* or distance between the target and the base of the conical cavity required for maximum penetration effect, varies with the metal used as a liner. With a given liner, there is an optimum stand-off distance above and below which less penetration effect is obtained. As stated previously, the jet is the penetrating agent; and as stand-off distance is brought into existence and increased, there is more time in which the jet can become extended. The results of this are increase in depth of penetration and decrease in diameter of the hole produced. However, after a certain stand-off distance, the jet has a tendency to break up both axially and radially. This prevents ductile drawing of the jet (in the case of continuous jets such as Cu) and increases the cross-sectional area and results in bifurcation and/or polyfurcation of the jet. Thus penetrating effect is decreased. Optimum stand-off distance must be determined experimentally for each type of shaped charge design.

i. Various cone angles and other shapes of cavities have been studied and used. Hemispherical cavities have been found to produce more shallow but wider holes than conical cavities. A helmet-shaped cavity (fig. 5-23) produces greater penetration than either the simple cone or hemisphere alone and was used by the Germans in their counterpart of the bazooka. As it has been found that a charge with an axial cavity above the liner gives in creased penetration (due to the prejet from this portion of the charge), a so-called flashback tube is attached to the apex or pole of the liner, such as the combination hemispherical liner with flashback tube (fig. 5-23) used in the projectile or the 57-mm recoilless gun. A further extension of this combination of cavities is the bottle-shape. In these two designs, with a hole throughout the charge, the jet from the liner of the cylindrical part of the cavity is formed first and this is followed by jet and slug from the liner of the hemispherical portion of the cavity.

j. The loading of shaped charge ammunition requires great precision, if maximum and reproducible penetration effects are to be obtained. Misalinement of the cavity axis with the axis of the explosive charge causes decrease in the penetration value of the jet. Uneven thickness of the metal liner, formation of a nonuniform layer of explosive at the base of the cavity, and voids or low-endsity regions in the explosive charge all have adverse effects on penetration value. These adverse effects are more pronounced for small than for large charges.

k. When used in projectiles that are subjected to rotation, there is much less penetration effect of a shaped charge than when it is fired statically. Investigation has shown that even low rates of rotation have a measurable effect and that this increases with increase in rate of rotation until a maximum effect is observed at certain point. Further increase in rate of rotation causes no further reduction in penetration value. Shaped charge projectiles, rotating at rates required for satisfactory flight stability, yield penetration effects but then only 50 percent or less of the effects obtained in static tests. Rotating shaped charges

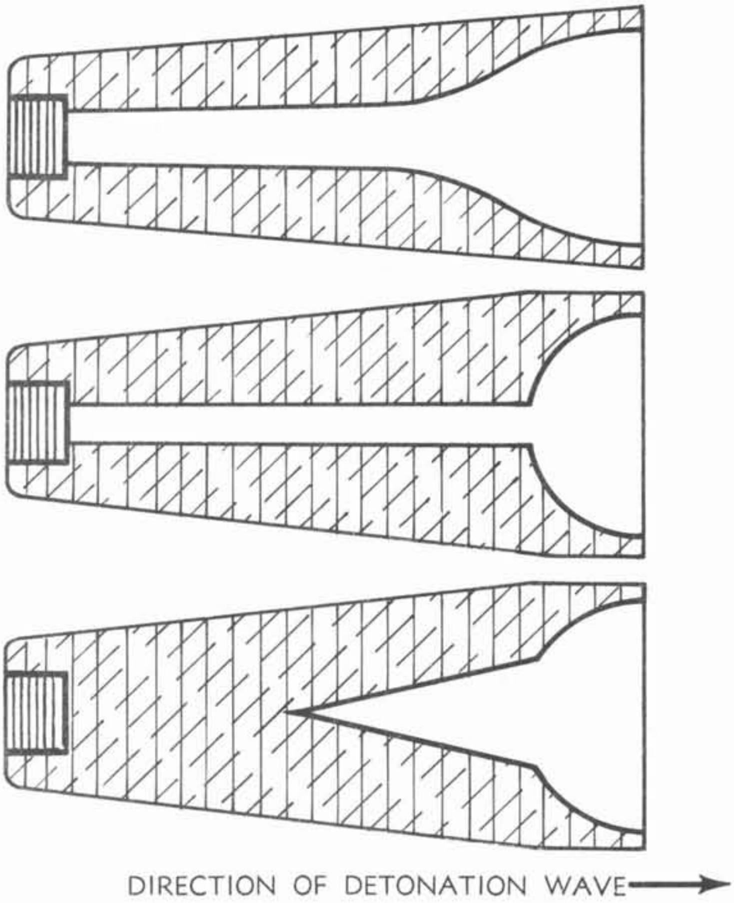


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Figure 5-23. Special forms of shaped charges.

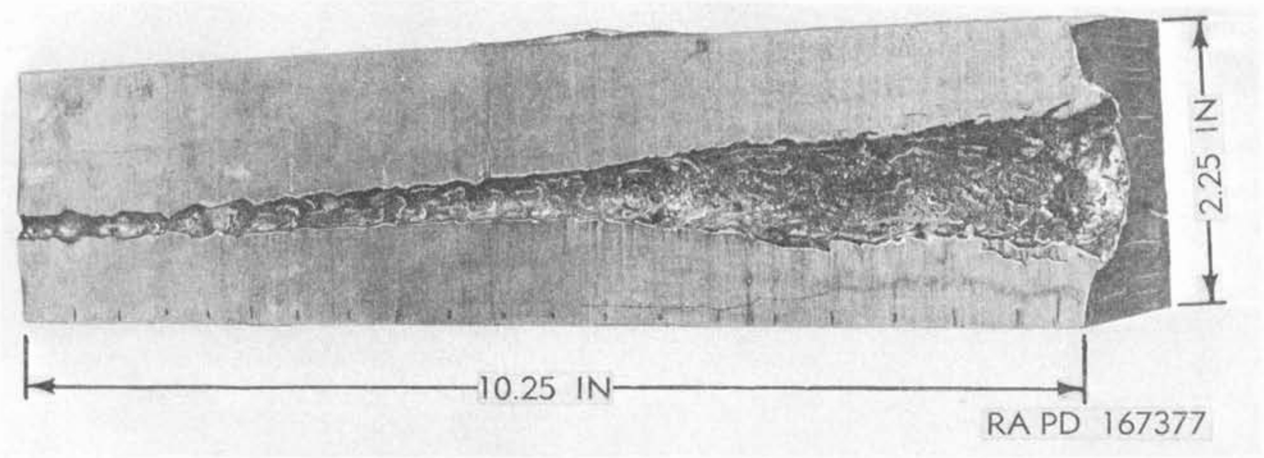

Figure 5-24. Penetration of steel by a shaped charge.

produce holes of greater diameter than static charges, but rotation does not affect the volume of the hole produced. The relative effect of rotation is approximately the same with both light and heavy cone liners; and changing the apex angle of the cone does not eliminate the reduction in penetration caused by rotation of the charge.

l. Determination of shaped charge effects is made by placing the assembly vertically, at a measured stand-off distance, above a pile of armor-plate steel plates 0.5 inch thick and detonating the charge. Great care must be taken to insure that the common axis of the charge and cavity is exactly perpendicular to the plane of the surface of the top plate. If a shaped charge is to be tested when under rotation, it is mounted at the end of an extension of the shaft of a motor capable of being run at selected speeds. After detonation of the charge, the depth of the hole, its average diameter at the top, and its volume are determined. Figure 5-24 shows in crossection a typical hole produced by a shaped charge.

m. Shaped charges are used in artillery and rocket projectiles, demolition charges, and antitank mines. In antitank artillery projectiles, shaped charges have the advantage in that their penetration effects are independent of terminal velocity at the target. Although their potential efficiency is much reduced by their rotation, such shaped charge projectiles can penetrate a greater thickness of armor plate than a corresponding shell with a conventional charge. The bazooka and M9A1 rifle grenade projectiles are not rotated and, therefore, function with maximum effectiveness. Having charges of only 0.5 and 0.25 pound, respectively, of 50-50 pentolite, they can penetrate 3 to 5 inches of armor plate. The M2A3 and M3 demolition shaped charges contain approximately 12 and 30 pounds, respectively, of 50-50 pentolite. The former is capable of penetrating 36 inches of reinforced concrete or, if the concrete is thicker, will produce a hole 30 inches deep and 2 to 3 inches in diameter. The M3 charge can penetrate 60 inches of concrete. Such demolition charges are used for destroying concrete pill boxes, knocking out bridge pillars, and severing supporting bridge spans. The Germans used a shaped charge antitank mine, which really was a demolition charge, since it was placed on enemy tanks or similar targets rather than being buried beneath the surface of the ground. With a shaped explosive charge of 2.5 pounds of an RDX-TNT mixture, it could penetrate armor plate 6 inches thick, producing a hole 1.25 inches in diameter and causing much flaking or spalling of steel from the interior of the tank wall.

n. Application of the shaped charge principle to other types of ammunition has been made or is under way, but these developments are not within the restricted scope of this manual. This applies also to developments in the direction of nullifying the effects of the shaped charge and protecting tanks, etc., from such effects.

CHAPTER 6

BLACK POWDER

6–1. Ignition, Fuze, and Fuse

a. General. Black powder is the generic name originally applied to a mixture of charcoal, sulfur, and potassium nitrate and now applied also to compositions containing bituminous coal instead of charcoal and sodium nitrate instead of potassium nitrate. In appearance it varies from a black, very fine powder to dense pellets which may be black or have a grayish-black color because of a graphite-glazed surface. The composition of black powder containing potassium nitrate, charcoal, and sulfur has remained essentially unchanged for 400 years, as any considerable modification of the 75:15:10 proportionality of the ingredients has been found to result in the powder burning more slowly or producing less effect. While no longer used as a military propellant, black powder finds application in the ignition of smokeless powder, time fuzes, saluting charges, squibs, smoke-puff charges, and catapult charges. Other uses are in ignition of rocket and missile propulsion units by means of black powder and black powder compositions used in rocket, Jato, and missile boosters and sustainers.

b. Composition and Combustion. Standard black powder contains 74.0±1.0 percent potassium nitrate, 15.6±1.0 percent charcoal, and 10.4±1.0 percent sulfur. Its autocombustion yields CO , CO, N , K_2CO_3, K_2SO_4, and K_2S as the principal products. As a result of combustion, there are liberated approximately 680 calories per kilogram of heat.

c. Burning Rate. The type of charcoal used in the manufacture of black powder is reflected by the burning rate of the powder, which burns much more rapidly when made from willow or alder charcoal than when made from oak charcoal. Increase in the percentage of nitrate, with corresponding decrease in percentage of charcoal, causes decrease in the burning rate, but this is unaffected by small changes in the proportion of sulfur present. The presence of more than 0.2 percent of moisture causes a decrease in the burning rate. Black powder made by simple mixing of the ingredients burns much more slowly than that in which the ingredients are incorporated or welded together by a wheel mill. When pressed, an incorporated black powder is not porous and the burning rate decreases with increase in density. When subjected to pressures of 25,000 and 75,000 psi, black powder has density values of 1.74 and 1.88, respectively. As manufactured, it has a particle density of 1.72 to 1.77. When confined, increase in gas pressure causes increase in burning rate, this being approximately 4 inches per second at a pressure of 25,000 psi.

d. Manufacture. Black powder is manufactured by pulverizing charcoal and sulfur together in a ball mill, whereby some of the sulfur is incorporated or worked into the cellular openings of the charcoal. The dampened material is mixed with finely ground nitrate and the moisture content is adjusted to approximately 4 percent. About 300 pounds of the mixture are placed in a wheel mill (fig. 6–1) having two wheels, each of which is 18 inches wide and weighs 8 or 10 tons. The milling operation, which is continued for 3 to 6 hours, increases the degree of incorporation. The milled material is pressed into cakes, using a pressure of approximately 6,000 psi. The cake is broken up by means of adjustable rolls having corrugated surfaces. The several rolls in the corning mill reduce the material to the desired grain size. Passage of this over shaker screens removes dust and fines, which are returned to the wheel mill. The coarse material

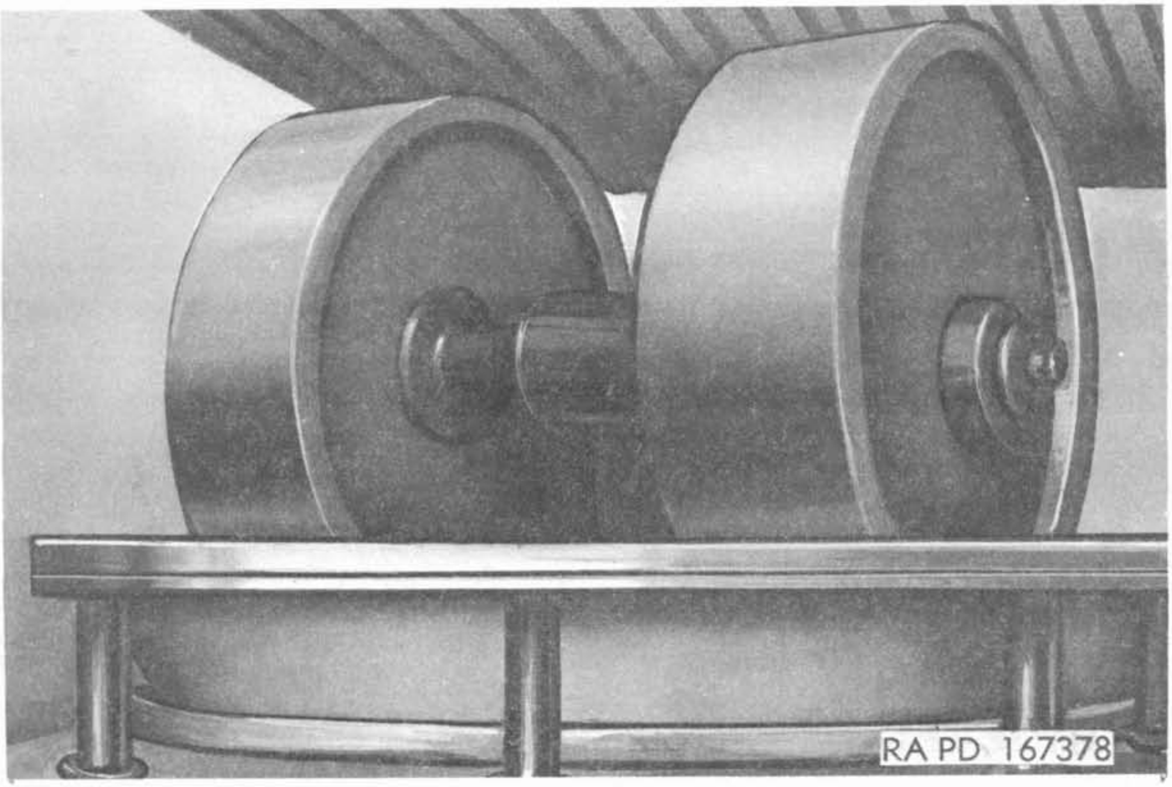

Figure 6-1. Black powder wheel mill.

is passed through the rolls a second time and is rescreened. In the blending house, the screened material is tumbled for about 8 hours in a rotating drum of hardwood. Because of friction, the powder is heated to as much as 65° C., and any moisture present is removed by evaporation. If the powder is to be glazed, graphite is added to the contents of the drum, after the moisture has been reduced to a certain point but before the powder is too dry. The dried powder is separated into standard granulations by sieving and then packed in metal drums holding 20 to 25 pounds. The manufacture of black powder is hazardous because of sensitivity to ignition by spark, and the separate operations are conducted by remote control in widely separated buildings. The corning mill operation is considered the most hazardous.

e. Granulations. Military black powder is manufactured in a range of grain sizes, each of which is identified by a designation—grade, symbol, or name—as shown in table 6-1.

f. Sensitivity. Black powder is less sensitive than tetryl, as judged by impact tests, and undergoes no ignition in the pendulum friction test with a steel shoe. In the sand test, it crushes no sand when ignited by a flame and only 8 grams when initiated by tetryl or PETN. Having an explosion temperature test value of 457° C., it is also relatively insensitive to nonradiant heat energy. Its high degree of accidental explosion hazard, therefore, is attributable to its great sensitivity to ignition by flame, incandescent particles, or electric spark. The ballistic pendulum test shows black powder to be 50 percent as powerful as TNT, but efforts to detonate it, by means of a booster explosive, have resulted in a maximum rate of decomposition approximately only 400 meters per second.

g. Stability and Hygroscopicity. In the ab-

Table 6-1. Granulations of Black Powder

Designation	Maximum percentages of grains retained on (R) and passed through (T) sieves of sizes 4, 6, 8, etc, as indicated.																			
	4	6	8	12	12	14	16	16	20	20	25	30	40	40	45	50	100	100	140	200
	R	R	T	R	T	R	R	T	R	T	T	T	R	T	R	T	R	T	T	T
Grade A-1	3.0		5.0																	
Grade A-2	3.0				5.0															
Cannon		3.0			5.0															
Grade A-3				3.0				5.0												
Grade A-3a				3.0						5.0										
Musket						3.0					5.0									
FFG							3.0					5.0								
Grade A-4							3.0							5.0						
Shell							3.0									5.0				
FFFG									3.0							5.0				
Grade A-5													3.0					5.0		
Fuze																				
FFFFG															3.0				5.0	
Grade A-6																	5.0		15.0	
Grade A-7																	3.0		50.0	
Meal																	5.0			50.0

sence of moisture, black powder is of a high order of stability, its ingredients being essentially nonreactive with each other even at 120° C. Heating black powder above 70° C., because of the rapid increase in the volatility of sulfur above that temperature, results in change in composition or uniformity of composition. Black powder is undesirably hygroscopic, this being due even more to the charcoal present than the hygroscopic nitrate also present. When used for military purposes, black powder is required to contain not more than 0.7 percent of moisture and, generally, is dried so as to contain only 0.2 to 0.3 percent of moisture immediately before loading. While the presence of moisture does not cause black powder to become unstable, in the presence of moisture it reacts with and corrodes metals such as steel, brass, and copper.

h. Ignition Black Powder. Black powder used for igniting charges of smokeless powder has the standard composition and is, generally, granulated so that at least 92 percent of the powder passes through a No. 4 sieve and is retained on a No. 8 sieve. The grains are glazed with graphite. The large number of finely divided, incandescent solid particles, produced by the burning of black powder, make it a better igniting material for smokeless powder than finely divided smokeless powder itself. The potassium nitrate and sulfur, used in the manufacture of this type of black powder, are required to be of high purity. The charcoal used is required to be made from a specified wood and contains a limited percentage of volatile matter. As a further control in the quality of the charcoal, the finished black powder is required to have an ash content no greater than 0.80 percent.

i. Black Powder for Fuzes. Black powders used for loading the time-train rings of fuzes have the standard composition in some cases, the granulations in these cases being such that at least 97 percent passes through a No. 140 sieve or at least 97 percent passes through a No. 40 sieve and not more than 5 percent passes through a No. 100 sieve. The grains of fuze powder are not glazed with graphite. Fuze powder having the standard composition is required to have a certain burning rate when loaded in a standard M54 fuze and tested under specified conditions. Such powder burns too rapidly for use in some fuzes, so a slow-burning powder is used. This has the composition—

	Percent
Potassium nitrate	70.0 ± 1.0
Semibituminous coal	14.0 ± 1.0
Sulfur	16.0 ± 1.0

This powder, which in use may be blended with powder having the standard composition, is required to contain not more than 0.75 percent of moisture and be of such granulation that at least 98 percent passes through a No. 140 sieve. When loaded in a specified fuze and burned, it is required to have a specified burning time, with a maximum dispersion in burning time of not more than 2.0 seconds. Coal powder is blended and mat milled. The characteristics of this composition depend largely on those of the coal used, and the volatile matter, fixed carbon, ash, and sulfur contents of this are specified. Like fuze powders having the standard composition, slow-burning fuze powder is not glazed with graphite. As coal has only the vestiges of a cellular structure, the manufacture of slow-burning fuze powder does not result in the same degree of incorporation as is obtained with the standard composition. Because of this and the differences in composition, the slow-burning powder burns only 60 percent as rapidly as the standard composition and does so with less uniformity.

j. Black Powder for Fuzes. Black powder used in the manufacture of time blasting or safety fuze may have the standard black powder composition, be a modification of the proportions of the ingredients of this, or contain inert diluents such as graphite, brick dust, or borax. The most common type of fuze burns at a rate of about 1 foot in 40 seconds. This low burning rate is ascribable to the design of the fuze as well as the composition used. The powder used is manufactured by the same process as that having the standard composition. It is produced as a finely granular, free-flowing powder which is compacted during the loading operation.

k. Analysis. The moisture content of black powder is determined by heating a 2-gram sample at 70° C. for 4 hours, cooling, and finding the loss in weight. The nitrate content is determined by extracting a 10-gram sample with hot water, drying the residue at 70° C. for 4 hours, cooling, and considering the loss in weight as nitrate and moisture. The residue is extracted with carbon disulfide, dried at 100° C. for 1 hour, and cooled. The loss in weight represents the sulfur present. The weight of residue after this extraction represents the charcoal or coal present in the black powder. This is ignited in a muffle furnace until all carbonaceous matter has been burned away. After cooling, the nonvolatile mineral matter is weighed and considered to represent the ash content of the powder.

6–2. Sodium Nitrate Black Powder

a. When sodium nitrate is substituted for potassium nitrate in black powder and the proportions of the ingredients are changed somewhat, there is obtained a composition that is more hygroscopic but burns more slowly and is cheaper than standard black powder. The sodium nitrate black powder used for military purposes and blasting operations has the following composition—

	Percent
Sodium nitrate	72 ± 2
Charcoal	16 ± 2
Sulfur	12 ± 2

The grains of powder are glazed with graphite.

b. For commercial use, sodium nitrate black powder is sieved to sizes designated as CCC, CC, C, F, FF, FFF, and FFFF. The average diameter of the CCC particle is about 14-mm, while that of the FFFF particle is only 1 to 2-mm. Sodium nitrate black powder for military use is granulated in three classes. Class A powder is of such fineness that at least 97 percent passes through a No. 12 sieve, at least 45 percent passes through a No. 16 sieve, and at least 95 percent is retained on a No. 40 sieve. This class is used in saluting charges. Class B powder is used in practice bombs and is of such granulation that at least 97 percent passes through a No. 4 sieve and at least 95 percent is retained on a No. 16 sieve. Class C powder, which is used for torpedo impulse charges, is required to pass through a sieve having 9/16-inch openings and be retained on a sieve having$^{3}/_{8}$ -inch openings. Black powder used in rocket or missile igniters has designated granulation depending on speed of ignition and the quality of brisance effect.

c. Sodium nitrate black powder has a specific gravity of 1.74 to 1.82 and is slightly less sensitive to impact than black powder containing potassium nitrate. The heat of explosion and gas volume values of both are approximately equal, but since the sodium nitrate powder burns the more slowly, it has even less brisance than the potassium nitrate powder. They are of the same order of stability. Both compositions are manufactured by the same process.

6–3. Compositions for Squibs

a. Squibs being designed to produce a voluminous flash of flame, there is used a finely branular composition that is only slightly compressed. This is loaded into a tube, with one end closed by a moistureprooving paper disk. Ignition of the squib composition is effected at the other end.

b. Squibs for military use are caused to function by the heat developed by an electrical resistance wire. This may ignite a charge of either potassium nitrate or sodium nitrate black powder or an ignition composition containing—

	Percent
Potassium chlorate	58
Diazodinitrophenol	48
Nitrostarch	2

This ignites the main charge of black powder. In some cases, the black powder charge is ignited by a matchhead composition containing—

	Percent
Potassium chlorate	30
Antimony sulfide	20
Dextrin	50

6–4. Black Powder Substitutes

Today, black powder is used primarily as a propellant ignition material. In some instances, however, black powder is used in such other applications as expelling charges and spotting charges.

a. Benite.

(1) Benite, developed at Picatinny Arsenal, as used in igniter compositions of artillery primers or in base igniter bags for separate-loading ammunition, is in the form of extruded strands consisting of the ingredients of black powder (KNO_3, Charcoal, Sulfur) in a matrix of nitrocellulose. The ratio of black powder ingredients to nitrocellulose is 60/40, proportions of black powder ingredients in Benite being the same as in black powder.

(2) Benite is manufactured by a solvent extrusion process, using essentially the same mixing, extrusion and drying facilities employed in the manufacture of double- and triple-base cannon propellant, with the exception that cannon-powder cutting equipment cannot be used. Stability of Benite, as measured by the standard short-term heat test, is improved by the direct substitution of Diphenylamine in the formulation.

(3) The processing of Benite involves use of an Ether-Alcohol solvent system. The feasibility of using a multi-orivce plate die in extrusion of Benite strands and drying Benite strands on a dowel was demonstrated. Benite colloid does not detonate under severe conditions of convnement and, therefore, should be considered a Class 2 explosive hazard (Fire Hazard) for extrusion. Automated high-capacity equipment of the type found in the food and plastics industries may be adapted to Benite manufacture.

b. Eimite. Eimite is another substitute for black powder in artillery primers.

c. Boron-Potassium Nitrate. Boron Potassium Nitrate is used in many instances to replace black powder. This material has an energy content of over 1600 calories per gram as against less than 1000 for black powder.

d. Mox Type Mixtures. Mox type compositions, developed beginning in 1950, consist of: varying percentages of such oxidizers as ammonium perchlorate, potassium nitrate, barium nitrate; metal powders including aluminum, magnesium or copper oxide; and small quantities of other ingredients (i.e., wax, stearates, and graphite). The oxidizers are generally coated with RDX, TNT or mixtures of both. Appropriate amounts of the ingredients are blended together for at least 30 minutes in the dry state. Although classified as explosives, these mixtures are used for specialized applications. The most common composition, MOX 2B, is used as a spotting charge in place of black powder.

CHAPTER 7

UNITED STATES HIGH EXPLOSIVES

7-1. Initiating Agents

a. Initiating agents used in military ammunition include priming compositions and initial detonating agents.

b. Priming compositions are physical mixtures of materials that are very sensitive to impact or percussion and, when so exploded, undergo very rapid autocombustion. The products of such an explosion are hot gases and incandescent solid particles. Priming compositions are used for the ignition of initial detonating agents, black powder igniter charges, propellants in small arms ammunition, etc.

c. Initial detonating agents are high explosives that are so sensitive to heat, impact, and friction that they undergo detonation when subjected to a flame or percussion. They are used to initiate detonation of the less sensitive high explosives comprising bursting charges, demolition high explosives, and dynamites. Most initial detonating agents have distinctly lower rates of detonation and brisance values than those of the explosives they are used to initiate. As they include azides, fulminates, and diazo-, nitro-, and nitroso-compounds, many of the initial detonating agents used are less stable than the noninitiating military explosives. Many compounds that have satisfactory initiating characteristics are too unstable or sensitive for use in military ammunition and, consequently, those actually in use are limited in number.

7-2. Initial Detonating Agents

a. Lead Azide.

(1) Lead azide, $Pb(N_3)_2$, is a salt of hydrazoic acid, HN_3, and has a molecular weight of 291.258. Lead azide exists in two forms: orthorhombic (α), density 4.71; and monoclinic (β), density 4.93. The pure compound, as usually prepared, consists of colorless, needle-like crystals. Formation of the too sensitive orthorhombic form is prevented by the very slow mixing of very dilute solutions of the lead nitrate and sodium azide from which lead azide is prepared. It can also be produced in the form of finely divided amorphous aggregates by the rapid mixing of relatively concentrated solutions of lead nitrate and sodium azide.

(2) The azides of lead, silver, and mercury were first prepared by Curtius in 1890 and 1891. The hazards involved in the manufacture and handling of the pure material delayed its practical use for many years; but it has been used in this form in some foreign countries since 1920. Military commercial use of "dextrinated" lead azide, an impure form, has been made in the United States since about 1930. This form will be that considered in the discussion in (3) through (18) below.

(3) Dextrinated lead azide is manufactured in the form of rounded aggregates, which have no observable crystal faces under a magnification of 50 diameters (figs. 7-1 and 7-6). It is white to buff in color and contains approximately 93 percent lead azide, 4 percent lead hydroxide, and 3 percent dextrin and impurities. It is used in detonators and priming compositions.

(4) The dextrinated and crystalline forms of lead azide have apparent density values of approximately 1.5 and 0.8, respectively, the absolute density of

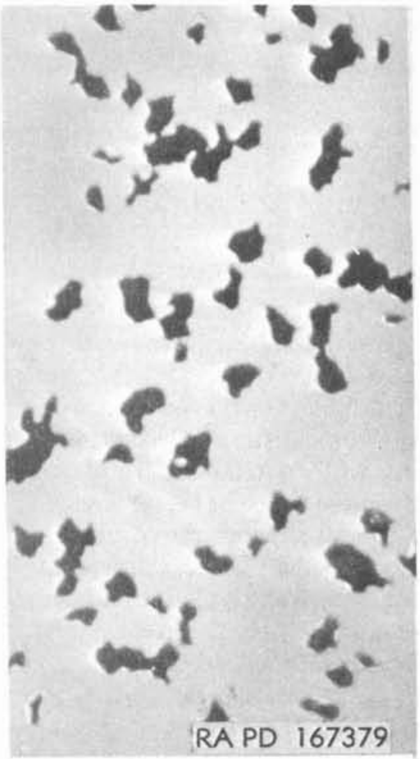

Figure 7–1. Lead azide crystals, 100 ×.

the dextrinated form being 4.38. When subjected to various pressures, the two types of leal azide have been found to have density values given in table 7–1.

Compressed lead azide has been reported to have a specific heat value of 1.55 calories per gram per degree Centigrade.

Table 7–1. Density of lead azide when pressed

Pressure, psi	*Density of lead azide, gram per milliliter*	
	Dextrinated	*Crystalline*
3,000	2.56	
5,000	2.72	3.00
10,000	2.94	3.37
15,000	3.07	3.51
25,000		3.72
50,000		4.05

(5) Pure lead azide is soluble in water to the extent of only 0.02 percent at 18° C. and 0.09 percent at 70° C. Dextrinated lead azide may be soluble in cold water to the extent of 1 percent, because of the solubility of the dextrin present. Both forms are almost insoluble in ether or acetone at their boiling points. Cold ethanol has little if any solvent action on lead azide, and the material can be stored when wet with a mixture of equal volumes of water and ethanol. Pure lead azide is somewhat soluble in an aqueous solution containing 10 percent of ammonium acetate. It is insoluble in ammonium hydroxide solution.

(6) When exposed to an atmosphere of 90-percent relative humidity at 30° C., dextrinated and crystalline lead azide are hygroscopic to the extent of 0.8 and 0.03 percent, respectively.

(7) Lead azide is decomposed by a 10 percent solution of sodium hydroxide, with the formation of lead hydroxide and sodium azide. A solution of ceric ammonium nitrate decomposes lead azide, with the quantitative liberation of nitrogen. When dissolved in aqueous ammonium acetate, lead azide reacts with potassium dichromate to form potassium azide and insoluble lead chromate. Dry lead azide does noto react with or corrode steel, iron, nickel, aluminum, lead, zinc, copper, tin, or cadmium. It does not affect coatings of acidproof black paint, baked oil, NRC compound, or shellac or steel surfaces that have been phosphatized. In the presence of moisture, it corrodes zinc and copper; and, in the latter case, it forms the extremely sensitive and dangerous compound copper azide. For this reason, lead azide is not loaded so as to be in direct contact with uncoated copper or its alloys; aluminum, generally being used for detonator shells, etc.

(8) Dextrinated and pure lead azide have calculated activation energy values of 23.4 and 41.3 kilogram-calories, respectively, per gram molecular weight. Upon detonation, pure lead azide liberates 367 calories of heat

and 308 milliliters of gas per gram, the lead produced being in the gaseous form. These values are slightly less than the corresponding values for mercury fulminate. Because of the nonexplosive diluents present, the heat produced by dextrinated lead azide is somewhat less than that produced by explosion of the pure compound.

(9) Dextrinated lead azide is manufactured as follows: 164 pounds of lead nitrate and 8.25 pounds of dextrin are dissolved in 245 gallons of water and the pH of the resulting solution is adjusted to 5.4 by the addition of sodium hydroxide. The solution then is heated to 71° C. and stirred vigorously. While agitation is continued, there is added slowly 233 gallons of an aqueous solution containing 64 pounds of sodium azide and 1.6 pounds of sodium hydroxide. The reactions taking place are—

$$Pb(NO_3)_2 + 2\ NaN_3 \rightarrow 2\ NaNO_3 + Pb(N_3)_2$$

$$Pb(NO_3)_2 + 2\ NaN_3 \rightarrow 2\ NaNO_3 + Pb(OH)_2$$

At the same time that insoluble lead azide and lead hydroxide are precipitated, dextrin also separates out. This acts as a binding agent for the particles of solid matter precipitated, and there are formed nodular aggregates, some of which are retained on a No. 270 sieve while others pass through a No. 325 sieve. The precipitated material is caught on a cloth and washed with water until the washings are free from nitrate. The purified product is kept in a wet condition until used.

(10) Several grades of lead azide are manufactured for military use. The grades are required to be white to blue in color, be free of needle-shaped crystals longer than 0.1 mm, have a minimum lead azide content of 91.5 percent, have a total lead content of 68.5 to 71.15 percent, be free of acidity, have a maximum solubility of 1.0 percent in cold water, and cause the complete detonation of tetryl when used in the sand test. The requirement that it be free of needle-shaped crystals longer than 0.1 mm prevents the presence of very sensitive crystals, which are believed to detonate when broken by the application of pressure. The other requirements assure the desired composition, its noncorrosiveness, and its effectiveness as an initiating agent.

(11) Dextrinated lead azide is less sensitive to impact than mercury fulminate, lead styphnate, diazodinitrophenol, tetracene, or crystalline lead azide. The small aggregates that pass through a No. 325 sieve are slightly less sensitive than those that pass through a No. 230 sieve and are retained on a No. 270 sieve. When wet with water for 95 percent ethanol, lead azide has the comparative sensitivity values shown by table 7-2.

Table 7-2. Sensitivity of Dry and Wet Lead Azide

Composition, percent				Pendulum friction test			
				Fiber shoe		Steel	shoe
Lead azide	Water	Ethanol	Impact, test.* inches.	Trials	Detonations	Trials	Detonations
100.......	....	....	4	1	1	....	....
80.......	20	....	9	10	0	4	1
80.......	....	20	4	1	1	....	....
75.......	25	....	9	10	0	12	0

* Picatinny apparatus with 2-kilogram weight.

(12) When pure lead azide is heated at 245° to 250° C., it decomposes into nitrogen and lead without explosion. In the explosion temperature test, temperatures of 340° and 345° C. are required to cause the explosion of dextrinated and crystalline lead azide, respectively, in 5 seconds, while a temperature of approximately 390° C. is required to cause the explosion of either in 0.1 second. These temperatures required for explosion are much greater than the corresponding values for mercury fulminate, lead styphnate, diazodinitrophenol, and tetracene and reflect the greater difficulty in igniting lead azide in practical use.

(13) When subjected to the sand test, dextrinated lead azide is indicated to be 95 percent as brisant as the pure crystalline material, but only 75 percent as brisant as mercury fulminate and 37 percent as brisant as diazodinitrophenol. Dextrinated and pure lead azide are indicated to have rates of 3,900 and 4,300 meters per second, respectively, when pressed at 3,000 psi (density values 2.62 and 2.65). When pressed so as to have a density of 4.0, the rates of detonation are indicated to be 5,000 to 5,200 meters per second.

(14) Trauzl lead block tests have shown dextrinated lead azide to be 89 percent as powerful as the pure compound, but only 80 percent as powerful as mercury fulminate.

(15) Lead azide is an excellent initiating agent for high explosives. While not superior to mercury fulminate for detonating the less sensitive explosives, such as TNT and picric acid, it is markedly superior as an initiator for the more sensitive explosives, such as tetryl, RDX, and PETN. Unlike diazodinitrophenol, it cannot initiate the detonation of explosive "D" or cast TNT.

(16) The stability of dextrinated as well as pure lead azide is exceptional. This is indicated by 100° C. heat and vacuum stability tests and also by storage tests. It has been found to be unchanged with respect to purity or brisance after storage for 25 months at 50° C. or under a water-ethanol mixture at ordinary temperature. Storage at 80° C. for 15 months caused no decrease in brisance and, after such storage, a priming

composition containing lead azide showed no decrease in sensitivity to stab action.

(17) Lead azide is not considered to be particularly toxic; but inhalation of the dust should be avoided, as this causes a headache and distention of the blood vessels. It has been recommended that the lead azide content of air be prevented from exceeding 0.2 milligram per cubic meter in order to avoid undue toxicity.

(18) Lead azide is sensitive to shock initiation and it has replaced mercury fulminate. Its superior initiating action, complete stability from a practical viewpoint, low cost, and the greater availability of its raw materials make lead azide the most important initial detonating agent for military use. It also is used extensively in the manufacture of commercial blasting caps.

b. Mercury Fulminate.

(1) Mercury fuliminate, $Hg(ONC)_2$, is a salt of an acid that is too unstable to exist in the free state. The mercury salt has a molecular weight of 284.65 and is white when pure but, as manufactured, it is only 98 to 99 percent pure and is grayish. It forms octahedral crystals, usually truncated, only the smaller crystals being fully developed (figs. 7-2 and 7-6). It crystallizes from water with half a molecule of water of crystallization, but lacks this as manufactured.

(2) First prepared by Johann Kunckel von Löwenstern (1630-1703), the preparation and properties of mercury fulminate were first described in 1800 by Howard. It was not until 1867 that the compound was used as initial detonating agent, when Nobel used it to tetonate nitroglycerin. Because of its sensitivity to flame and percussion, it was for many years the most important material used in detonators and as an ingredient of priming compositions. Since 1930, it has been replaced extensively by lead azide, but is still used to some extent in military ammunition and commercial blasting caps.

Figure 7-2. Mercury fulminate crystals, 125 x.

(3) Mercury fulminate has a crystal density of 4.42 and an apparent density of 1.35 to 1.75. When subjected to pressures of 3,000, 10,000, 20,000, and 50,000 psi, density values of 3.0, 3.6, 4.0, and 4.3 are obtained. Pressure at 25,000 to 30,000 psi or more causes the desensitization of mercury fulminate to such an extent that the dead-pressed material merely ignites and burns when subjected to contact with a flame. Dead-pressed mercury fulminate is detonated if it is covered with a layer of loose or slightly compressed fulminate and this is ignited. Mercury fulminate in compressed form is reported to have a specific heat value of 1.1 calories per gram per degree Centigrade.

(4) Soluble in water to the extent of only 0.01 percent at 15° C. and only slightly soluble in cold ethanol, mercury fulminate can be stored under water or, when there is danger of freezing, under a mixture of equal volumes of water and ethanol or methanol. It is dried easily and is hygroscopic to the extent of only 0.02 percent when exposed to an atmosphere of 90 percent relative humidity at 30° C. It is soluble in aqueous ammonium hydroxide or potassium cyanide and in pyridine, and can be recovered from such solutions by treating the ammonium or potassium solution with acid or by adding water to the pyridine solution.

(5) Mercury fulminate reacts with concentrated hydrochloric acid to form hydroxylamine, $H_2N.OH^2$, and formic acid, HCOOH. It reacts with sodium thiosulfate in aqueous solution, with the formation of mercury tetrathionate—

$$\begin{gathered} Hg(ONC)_2 + 2\ Na_2S_2O_3 + \\ H_2O \rightarrow HgS_4O_6 + 2\ NaOH + \\ NaCN + NaNCO \end{gathered}$$

If allowed to stand, a secondary reaction occurs with the information of sulfate and thiocyanate—

$$\begin{gathered} HgS_4O_6 + 2\ NaOH + NaCN \\ + NaNCO \rightarrow HgSO_4 + \\ Na_2SO_4 + 2\ NaNCS + H_2O \end{gathered}$$

The first reaction can be used for determination of the purity of mercury fulminate. Even in the presence of 0.5 percent of moisture, pure mercury fulminate does not react with any of the ordinary metals. However, the standard grade of mercury fulminate may contain as much as 1 percent of free mercury and, if this impurity is present to an appreciable extent, the mercury fulminate may cause slight corrosion of copper or its alloys by amalgamation.

(6) Mercury fulminate is manufactured in relatively small quantities. A solution of 1 pound of redistilled mercury in 8 to 10 pounds of nitric acid of specific gravity 1.4 is heated at 55° to 60° C. and then poured into a large balloon flask containing 8 to 10 pounds of 95-percent ethanol. The flask rests in a bath of running cold water. After 1.5 hours the reaction is complete; and

when the reaction mixture has cooled to room temperature, all of the fulminate has settled out. This is caught on a cloth screen and washed with cold water until free of acid and fine impurities termed "fulminate mud. The washed fulminate is transferred to cloth bags and stored wet until used. The yield is 120 to 130 parts per 100 parts of mercury. Ethyl nitrate ($C_2H_5.ONO_2$), ethyl nitrite ($C_2H_5.ONO$), and nitroethane ($C_2H_5NO_2$) also are produced. The intermediate products of oxidation and nitration involved in the preparation of mercury fulminate are as follows:

$CH_3.CH_2OH \rightarrow CH_3.CHO \rightarrow$
Ethanol Acetaldehyde

$CH_2(NO).CHC \rightarrow CH(:NOH).CHO \rightarrow$
Nitrosoacetaldehyde Isonitrosoacetaldehyde

$CH(:NOH).COOH \rightarrow$
Isonitrosoacetic acid

$C(NO_2)(:NOH).COOH \rightarrow$
Nitroisonitrosoacetic acid

$O_2H.CH:NOH \rightarrow C:NOH \rightarrow Hg(ONC)_2$
Formonitrolic acid Fulminic acid Mercury fulminate

The product so obtained is not more than 99 percent pure. It can be purified so as to have a fulminate content of 99.75 percent or more by dissolving the impure material in concentrated ammonium hydroxide, filtering the solution, cooling the filtrate, and reprecipitating slowly, adding concentrated nitric acid with rapid agitation, while keeping the temperature below 35° C. Conditions can be established so that the precipitated fulminate is of essentially the same granulation as the impure material. The precipitated pure fulminate is washed with distilled water until free of acid. The yield is 80 to 87 percent.

(7) Mercury fulminate for military use consists of one grade. The requirements applying are—

Appearance:	Crystals of sparkling appearance that are white, gray, or light gray with a yellowish tint.
Granulation:	A maximum of 15 percent retained on a No. 100 and a maximum of 75 percent passing through a No. 200 sieve.
Sand test:	0.400 gram shall crush a minimum of 44 grams of sand.
Mercury fulminate:	Minimum, 98.0 percent.
Acidity:	None.
Insoluble matter:	Maximum, 2.0 percent.
Free mercury:	Maximum, 1.0 percent.
Chlorine:	Maximum, 0.05 percent.

The requirement, with respect to chlorine content, is due to the occasional use of a small quantity of cupric chloride, $CuCl_2$, for the purpose of improving the color of the product. The chloride is added to the solution of mercury in nitric acid. While this improves the color, it also decreases the purity of the product. The insoluble matter represents decomposition products, which are formed as the result of side reactions.

(8) Mercury fulminate is more sensitive to impact than lead azide and lead styphnate. Being of the same sensitivity to impact as diazodinitrophenol and tetracene, these three compounds are the most sensitive initiating explosives used in military ammunition. Mercury fulminate is more sensitive

to heat and friction than lead azide and lead styphnate and has a higher explosion temperature test value (210° C.) than diazodinitrophenol and tetracene. Comparative values for the sensitivity of these last two compounds to friction are not available. The sensitivity of mercury fulminate to percussion is one of its most advantageous characteristics.

(9) As measured by the sand test, the brisance of mercury fulminate is greater than that of lead azide, but only 50 percent that of diazodinitrophenol. This relationship is in agreement with the relative order of the rates of detonation of the three compounds.

(10) Mercury fulminate is a distinctly more powerful explosive than lead azide, as indicated by Trauzl lead block tests, and this is confirmed by the higher heat of explosion.

(11) Although mercury fulminate is not as efficient an initiator of detonation as lead azide and diazodinitrophenol, it is entirely satisfactory when used in conjunction with tetryl, RDX, or PETN, the most generally used booster explosives. During the half century it was almost the only initial detonating agent in practical use, its initiating efficiency was considered satisfactory.

(12) The relatively poor stability of mercury fulminate has been its most disadvantageous characteristic and the main reason for efforts to replace this initiator with a superior substitute. The usual stability tests are not applicable to mercury fulminate, due to explosion in a relatively short time at temperatures above 85° C. When mercury fulminate deteriorates, it is chiefly with the production of a nonexplosive solid rather than gaseous products of decomposition. It has been found that when its purity has been reduced to approximately 92 percent, the initiating efficiency of mercury fulminate is practically destroyed, although the material will explode when ignited. When purity has been reduced to 95 percent, the stability of mercury fulminate must be considered to be seriously impaired.

Table 7-3. Deterioration of Mercury Fulminate

Storage temperature °C.	Time required to reduce purity to					
	95 percent			92 percent		
	Days	Months	Years	Days	Months	Years
80	0.5	------	------	1	------	------
50	------	8	------	------	11	------
30-35	------	------	1.7	------	------	5.8
20	------	------	7	------	------	9
10	------	------	8	------	------	10

Table 7-3 gives a summary of the times of storage at various temperatures required to cause deterioration to 92 and 95 percent purity. The importance of the purity of mercury fulminate, with respect to its stability, can be recognized from the data in table 7-3 and the fact that mercury fulminate of 99.75 percent purity has been found to require storage at 50° C. for 24 months to become deteriorated to 92 percent purity.

(13) Mercury and its compounds are of recognized toxicity. The handling of mercury fulminate is not unduly hazardous from a toxicity viewpoint, but should be done with minimum contact with the skin. Its dust should not be inhaled and it has been recommended that, in order to avoid undue toxicity, the air in loading plant buildings should contain not more than 0.1 milligram of mercury fulminate per cubic meter.

c. Diazodinitrophenol (DDNP).

(1) Diazodinitrophenol, 4,5-dinitrobenzene-2-diazo-1-oxide, dinol, diazol or DDNP is a compound having the structure

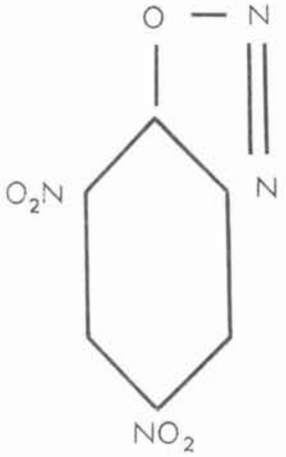

Figure A. DDNP

which forms greenish yellow to brown tabular crystals (figs. 7-3, 7-6). It has a molecular weight of 210.108. It is used extensively in commercial blasting caps and has found some use in military priming compositions and detonators. DDNP was first prepared by Griess in 1858, but it was not until 70 years later that it was developed commercially as an initial detonating agent.

(2) DDNP has a crystal density of 1.63 and in the loose form has an apparent density of 0.5 to 0.9. When pressed at 3,000 psi, it has a density of about 1.2. It is not desensitized when subjected to a pressure of 130,000 psi.

(3) The solubility of DDNP in various liquids is shown by table 7-4. It is soluble to some extent also in nitroglycerin, nitrobenzene, aniline, pyridine, and concentrated hydrochloric acid. DDNP is nonhygroscopic in an atmosphere of 90-percent relative humidity at 30° C.

(4) DDNP does not react with water at ordinary temperatures, but is desensitized by it. A 0.5-percent solution of sodium hydroxide decomposes DDNP, with the liberation of nitrogen. It is darkened rapidly by exposure to sunlight, this effect probably representing oxidation at the surface.

Figure 7-3. Diazodinitrophenol crystals, 50 x.

Table 7-4. Solubility of Diazodinitrophenol

	Solubility, grams per 100 grams of solvent at—	
	25° C.	50° C.
Water	0.08	
Benzene	0.09	0.23
Methanol	0.57	1.25
Ethanol	0.84	2.43
Ether	0.04	
Chloroform		0.11
Ethylene chloride	0.29	
Acetic acid	1.40	
Ethyl Acetate		2.45
Acetone	6.0	

(5) The process used commercially for the manufacture of DDNP is not available, but it can be prepared by the diazontization of picramic acid by means of sodium nitrite and hydrochloric acid. The reactions involved are—

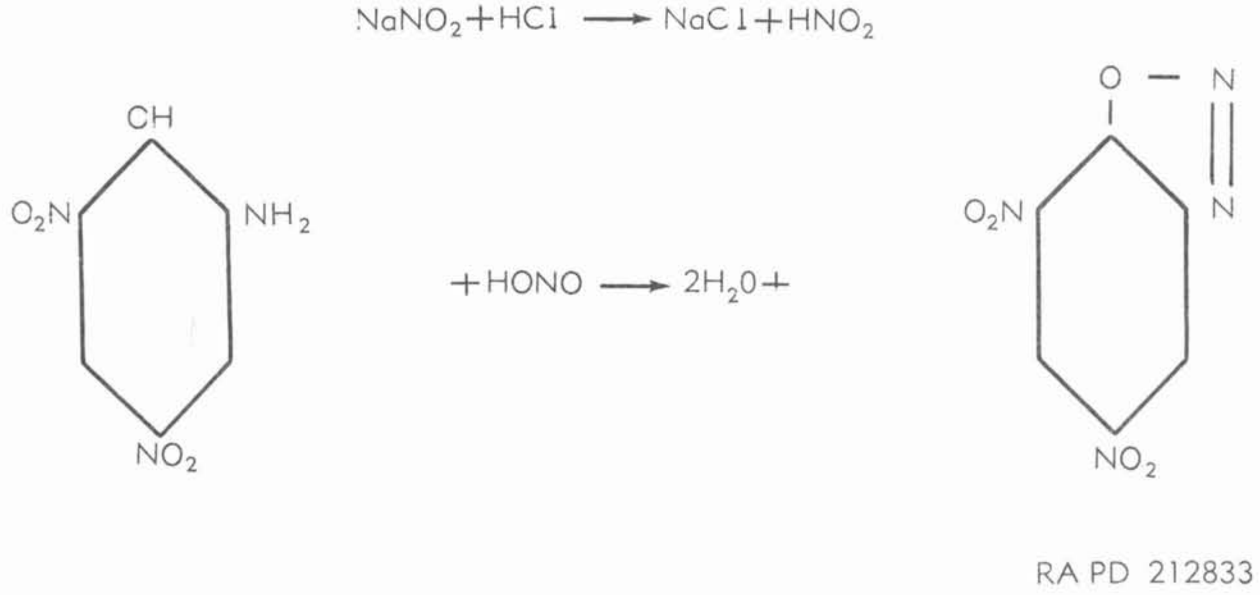

Figure B. DDNP.

Picramic acid may be prepared by evaporating a mixture of an alcoholic solution of ammonium picrate and ammonium sulfide and purifying the product. Ten grams of picramic acid are suspended in 120 milliliters of a 5-percent aqueous solution of hydrochloric acid. The mixture is cooled with an ice bath and stirred rapidly. A solution of 3.6 grams of sodium nitrite in 10 milliliters of water is added all at once and stirring is continued for 20 minutes. The dark brown granular material that separates is caught on a filter and washed with ice water until the washings give no test for hydrochloric acid or sodium chloride. If this material is dissolved in hot acetone and to the agitated solution there is added a large volume of ice water, the DDNP is precipitated as a bright yellow amorphous powder. Recrystallization from a solvent is used to produce the tabular crystals that comprise specification grade material. After manufacture, it is kept wet with water until used.

(6) But one grade of DDNP is used for military purposes. This complies with the following requirements:

Color: Greenish yellow to brown.
Form: Tabular crystals having a maximum length of 0.2 mm.
Granulation: 100 percent shall pass through a No. 100 US standard sieve.
Bulk density: Minimum, 0.4 g/ml.
Acidity: Maximum, 0.01 percent as hydrochloric acid.
Sand test: 0.40 gram shall crush not less than 33 grams of sand.

(7) DDNP is as sensitive to impact as mercury fulminate and much less sensitive to friction. It has approximately the same sensitivity to friction as lead azide. The explosion temperature test value of 180° C. for DDNP shows it to be much more easily ignited than even mercury fulminate. Water effectively desensitizes DDNP, as it is not detonated by a No 8 blasting cap when wet with water. Although DDNP detonates when struck a sharp blow, it burns with a flash,

if ignited, when unconfined and even in quantities of several grams. However, even slight confinement causes the transition of burning into detonation. If pressed into a blasting cap shell with a reinforcing cap, and a piece of black powder safety fuse is crimped in the shell, a charge of DDNP undergoes detonation when ignited. It is said that a spark falling into the open end of such a blasting cap causes only ignition and flashing of the DDNP.

(8) As indicated by sand test values, DDNP is twice as brisant as mercury fulminate and of the same order of brisance as TNT. It is to be noted that DDNP and TNT at a density of 1.58 have the same rate of detonation of 6,900 meters per second. Tests with 1-gram samples in small Trauzl lead blocks have shown DDNP to be three times as powerful as mercury fulminate.

(9) By means of the sand test, it has been found that DDNP is a better initiator of detonation than mercury fulminate or lead azide for the less sensitive high explosives. The most marked evidence of this is the ability of DDNP to initiate the detonation of explosive D and cast TNT. For initiation of the more sensitive high explosives, DDNP is not superior to lead azide. Comparative values showing relative initiating efficiencies are given in table 7-5.

Table 7-5. Efficiency of Initiating Compounds

	Minimum detonating charge, gram of initiator required to detonate—		
	Tetryl	*TNT*	*Explosive "D"*
Lead azide	0.10	0.26	([a])
Mercury fulminate	0.19	0.24	([a])
Diazodinitrophenol	0.12	0.15	0.28

[a] Not detonated.

(10) DDNP is not as stable as lead azide, but is markedly superior to mercury fulminate in this respect, as indicated by 100° C. vacuum stability and heat test results. Storage tests have shown dry DDNP to withstand storage at 50° C. for at least 30 months, as compared with 9 months for mercury fulminate. When stored under water, DDNP is of unimpaired brisance for 24 months at ordinary temperature and for 12 months at 50° C. The stability of DDNP, therefore, is considered satisfactory for commercial and military use, and DDNP as well as lead azide has replaced mercury fulminate in blasting caps to a large extent. It is used to some extent in loading fuze detonators and the manufacture of priming compositions for military use.

d. Lead styphnate.

(1) Lead styphnate or lead 2,4,6-trinitroresorcinate is a compound having a molecular weight of 450.30 and the structure—

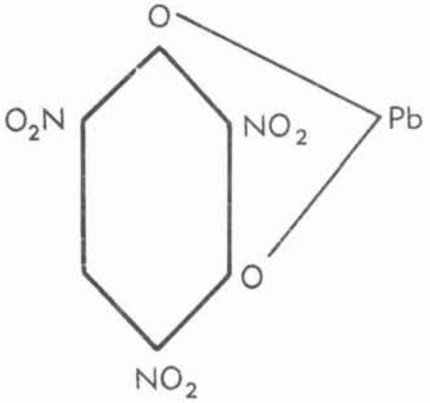

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Figure C. Lead styphnate.

It forms light orange or reddish-brown rhombic crystals containing a molecule of water of crystallization. First described in 1914 by von Herz, it is a relatively poor initiator of detonation but has found use as a cover charge for lead azide and as an ingredient of priming compositions.

(2) The crystal density of lead styphnate is 3.02, but the apparent density of the uncompressed material is only 1.4 to 1.6.

(3) Lead styphnate is soluble in cold water to the extent of only 0.04 percent and has a hygroscopicity value of only 0.05 percent when exposed to an atmosphere of 100 percent relative humidity at 25° C. It is less soluble in acetone and ethanol and is insoluble in ether, chloroform, carbon tetrachloride, carbon bisulfide, benzene, toluene, concentrated hydrochloric acid, and glacial acetic acid. It is somewhat soluble in a 10 percent aqueous solution of ammonium acetate.

(4) Lead styphnate is decomposed by concentrated nitric or sulfuric acid. When dissolved in ammonium acetate solution, it reacts with potassium dichromate to form potassium styphnate and insoluble lead chromate.

(5) Lead styphnate can be prepared by the reaction of lead nitrate or acetate with either sodium or magnesium styphnate in the presence of an acid. In commercial production, the magnesium salt is used. This is prepared by the reaction of magnesium oxide and styphnic acid (2,4,6-trinitroresorcinol). In one procedure, a boiling solution of lead nitrate is added to a boiling solution of sodium styphnate acidified with acetic acid. In another procedure, solutions of magnesium styphnate and lead acetate are caused to react at 70° C. to form a precipitate of basic lead styphnate, which is converted into the normal salt by the addition of dilute nitric acid. The insoluble product is filtered off and washed with water until free of acid. Like other initial detonating compounds, lead styphnate is kept in a wet condition until used. Photomicrographs of lead styphnate are shown in figures 7.4 and 7.6.

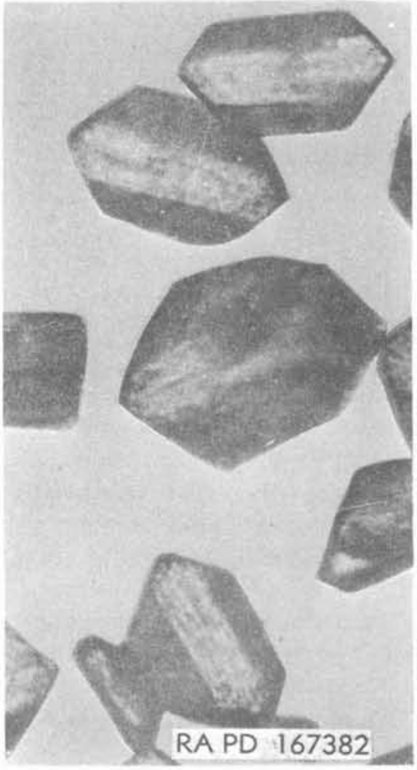


Figure 7-4. Lead styphnate crystals, 425 x.

(6) One grade of lead styphnate is used for military purposes. This must comply with the following major requirements:

Form: Cubic crystals not longer than 0.5 mm and free of needle-shaped crystals or crystal aggregates.

Purity: Minimum, 98.5 percent of $PbO_2C_6H(NO_2)_3.H_2O$.

Lead content: Maximum, 44.5 percent.

Apparent density: 1.5±0.1 grams per milliliter.

pH of a 1 percent solution: 5.0 to 7.0.

Ammonium acetate insoluble matter: Maximum, 0.3 percent.

Ether-soluble matter: Maximum: 0.1 percent.

Granulation: Not less than 99.5 percent through a No. 100 sieve.

Brisance: Minimum, 50 percent of that of TNT, as determined by the sand test.

(7) Lead styphnate is slightly less sensitive to impact than mercury fulminate or diazodinitrophenol, but is more sensitive than lead azide. It is less sensitive to friction than mercury fulminate or lead azide. It has an explosion temperature test valve (282° C.) less than that for lead azide but much greater than those for mercury fulminate and diazodinitrophenol. Although it detonates with a loud noise when brought into contact with a flame, lead styphnate apparently detonates at less than its maximum rate when ignited. The calculated activation energy for the decomposition of lead styphnate is 61.5 kilogram-calories per gram molecule as compared with a value of 25.2 kilogram-calories for mercury fulminate. It is much more easily ignited by an electrical spark than is mercury fulminate, lead azide, or diazodinitrophenol.

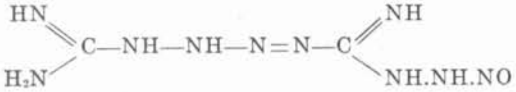
(8) When subjected to the sand test, with ignition by a black powder fuse, the brisance of lead styphnate (10.5 grams) is much less than that of lead azide (16.7 grams). However, when initiated with a small charge of mercury fulminate or lead azide, its brisance (24.0 grams) is greater than that of lead azide or mercury fulminate (22.1 grams). When initiated with blasting caps, lead styphnate pressed to a density of 2.9 has a rate of detonation of 5,200 meters per second, which is greater than the rate of detonation of either lead azide or mercury fulminate at the same density. Trauzl lead block tests, with large and small blocks, show lead styphnate to be more powerful than lead azide and probably equal to mercury fulminate.

(9) In spite of its favorable rate of detonation and power characteristics, lead styphnate is a relatively poor initiator of detonation. While it detonates straight 60 percent dynamite and sometimes detonates gelatin dynamite, it is incapable of initiating the detonation of any of the military high explosives except PETN. It initiates only unpressed PETN, and the minimum detonating charge is 0.55 gram as compared with 0.30 gram of mercury fulminate or 0.04 gram of lead azide. However, the ease of ignition of lead styphnate renders it suitable for use as an igniting charge for lead azide and as an ingredient of priming compositions. It has been used extensively abroad for the first purpose and to some extent in the United States for the second.

(10) Lead styphnate is of a high order of stability as indicated by vacuum stability tests at 100° and 120° C. The loss of 1.5 percent in the 100° C. heat test may be attributable to the removal of water of crystallization, which is present to the extent of 3.84 percent. Storage at 80° C. caused no change in its 120° C. vacuum stability test value, although the explosion temperature and sand test values were increased slightly. These changes also may be attributable to loss of water of crystallization. The stability of lead styphnate may be considered to approach that of lead azide and be better than that of mercury fulminate or diazodinitrophenol.

E. TETRACENE

(1) Tetracene, 4-guanyl-1-(nitrosoaminoguanyl)-1-tetrazene, is a compound having the structure



which is prepared as crystals forming a colorless or pale yellow, fluffy material. It has a molecular weight of 188.16. It was first prepared in 1910 by Hoffman and Roth and has been used as an ingredient of priming compositions.

(2) As prepared, tetracene has an apparent density of only 0.45, and when subjected to a pressure of 3,000 psi the density is increased to 1.05.

(3) Tetracene is practically insoluble in water, but is hydroscopic to the extent of 0.8 percent when exposed to an atmosphere of 95 percent relative humidity at 30° C. It can be stored wet with water or a mixture of water and ethanol. It is insoluble in ethanol, ether, benzene, and carbon tetrachloride. It is soluble in strong hydrochloric acid, and from this solution it is precipitated as the hydrochloride by the addition of ether. This, on treatment with sodium acetate or ammonium hydroxide, yields tetracene.

(4) Boiling water decomposes tetracene, with the liberation of 2 molecules of nitrogen per molecule of tetracene. On hydrolysis with sodium hydroxide, it yields ammonia, cyanamide, and triazonitrosoaminoguanidine, $N_3C(:NH).NH.NH.NO$. Tetracene reacts with an excess of silver nitrate to form the double salt $C_2H_7N_{10}OAg.AgNO_3.3H_2O$ and forms explosive salts, such as the perchlorate.

(5) Tetracene is prepared by the reaction of aminoguanidine and nitrous acid.

$$H_2N.C(:NH).NH.NH\boxed{H + HO}N\boxed{O + H_2}N.C(:NH).NH.NH\,H + HO\,NO \rightarrow 3\,H_2O + H_2N.C(:NH).NH.NH.N:N.C(:NH).NH.NH.NO$$

In practice, this is brought about by the reaction of 1-aminoguanidine hydrogen carbonate or sulfate with sodium nitrate in the presence of acetic acid. If a mineral acid is used, guanyl azide, $N_3.C(:NH).NH_2$, is formed; and if the acetic acid is in excess, there is formed 1,3 ditetrazolyltriazine.

Tetracene may be prepared as follows: 34 grams of aminoguanidine carbonate is dissolved, with warming, in a mixture of 2,500 milliliters of water and 15.7 grams of glacial acetic acid. After the solution has been filtered and cooled to 30° C., there is added 27.6 grams of solid sodium nitrite, which is dissolved by stirring. After standing for 3 or 4 hours, the solution is shaken to start precipitation and the mixture is allowed to stand for about 20 hours longer. The precipitate of tetracene is separated and washed with water until free of acid.

(6) No standard grade of tetracene for military use has been established. Photomicrographs of the material prepared as described above are shown by figures 7–5, and 7–6.

(7) Tetracene is as sensitive to impact as mercury fulminate and diazodinitrophenol and has a lower explosion temperature test value (154° C.) than any of the other initiating compounds considered herein. It is this ease of ignition and its relatively high heat of explosion and gas volume that render it useful in priming compositions and, along with lead azide, in explosive rivets. When exposed to contact with a flame, tetracene undegoes mild explosion with the production of much black smoke. Its sensitivity is such that it is extremely

Figure D. Ditetrazolyltriazine.

Figure 7.5. Tetracene crystals, 125 x.

subject to dead-pressing. In the sand test the loose material crushes 13.1 grams of sand, but when loaded under a pressure of 3,000 psi, it crushes only 2.0 grams of sand. However, tetracene pressed at 3,000 psi crushes 21.1 grams of sand when initiated with mercury fulminate. This maximum brisance is equal to that of mercury fulminate and is reflected by its relatively high Trauzl lead block test value (157 milliliters) as well as its high heat of explosion.

(8) Unpressed tetracene, when ignited by a flame, can cause the detonation of loose or pressed PETN, but pressed tetracene does not detonate PETN. While unpressed tetracene can cause low-order detonation of tetryl, it has no such effect on TNT, even when primed with mercury fulminate, Tetracene therefore does not have sufficient initiating efficiency to permit its use as such with military high explosives.

(9) Tetracene is relatively stable at temperatures not greater than 75° C. However, in the 100° C. heat test, it undergoes extreme decomposition in the first 48 hours of the test. Sufficient data are not available to permit a definite evaluation of the stability of tetracene, but its reactivity with boiling water and slightly high 75° C. international test value (0.5 percent) indicate an order of stability approximating that of mercury fulminate.

7–3. Priming Compositions

a. The ideal priming composition would consist of a single chemical compound of uniform particle size that would undergo very rapid autocombustion, but not detonation when subjected to friction or impact. Lead dinitroresorcinate has been found to have these characteristics, but its sensitivity is less than that desirable and the compound has not been standardized. The next most desirable type of composition would be a mixture of nonexplo-

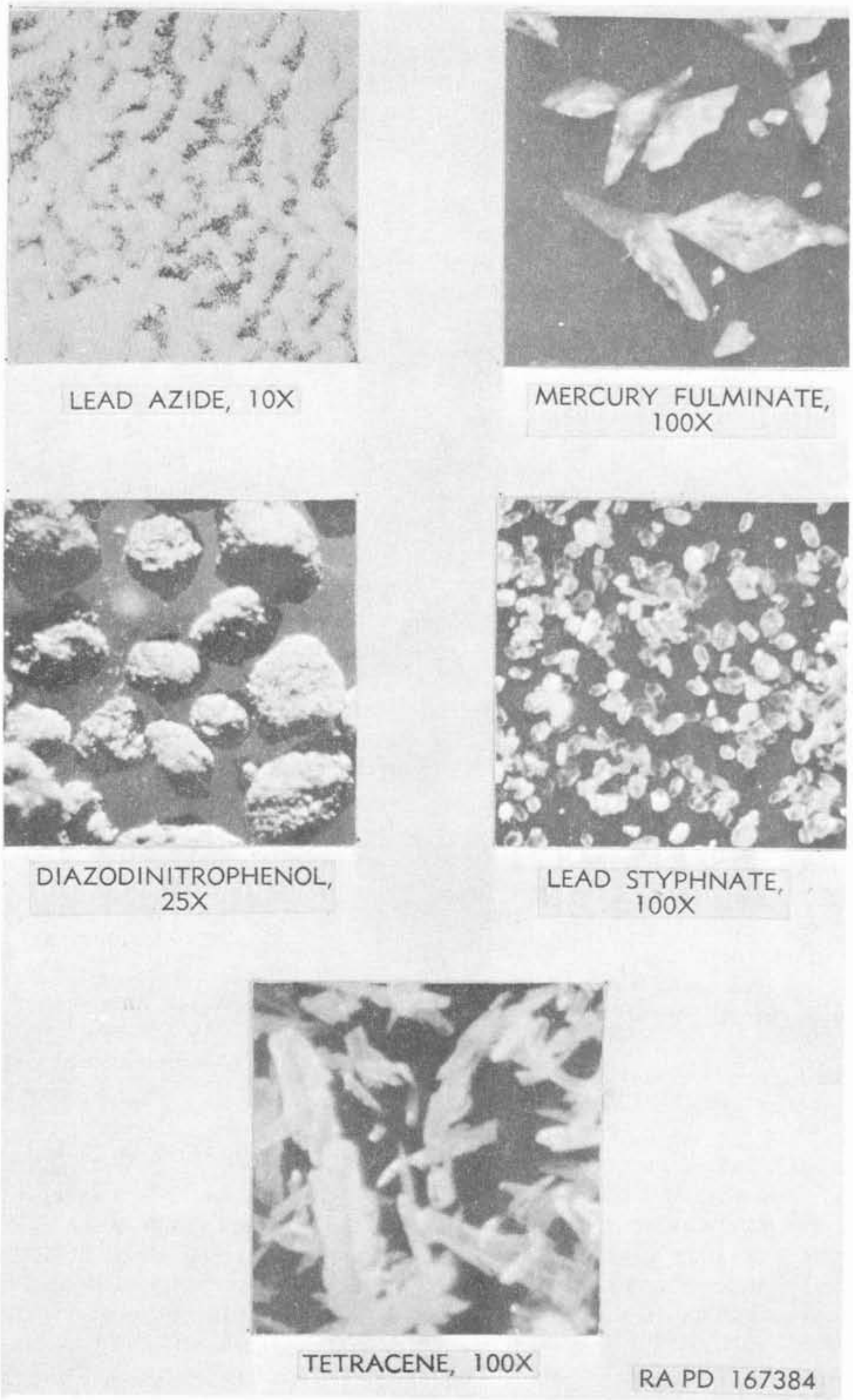


Figure 7–6. Initial detonating agents.

sive compounds that sensitize each other to ignition and rapid burning. Such compositions have been developed and used. Most military priming compositions consist of mixtures of one or more initial detonating agents with oxidizing agents, fuels, sensitizers, and binding agents.

b. The oxidizing agents used in priming compositions are potassium chlorate and barium nitrate; while the fuels are lead thiocyanate, carbon black, antimony sulfide, and calcium silicide. The last two serve also to sensitize the composition to friction or percussion. Carborundum and ground glass also are used as sensitizers. The explosive ingredients generally are the sensitive initiating compounds, lead azide, mercury fulminate, lead styphnate, and diazodinitrophenol; but TNT and PETN also are used in compositions containing no initial detonating compound. Shellac, gum arabic, and gum tragacanth are the chief binding agents used and serve also as fuels. Binding agents permit the use of priming compositions in the form of caked masses, instead of the powdery physical mixtures formed from the chief ingredients of the compositions.

c. Individual charges of priming composition are very small, being of the order of 0.05 to 0.2 gram. Because of this and the sensitivity of the compositions to friction, they are manufactured in small lots of about 5 pounds or less. If the composition is to be used in a dry condition, the ingredients are placed on a triangular sheet of rubber having a cord attached to each corner and located behind a barricade. By raising the corners alternately, the ingredients are mixed thoroughly. A final lifting operation discharges the mixture through a screen into a rubber container. If the composition is to be wet with a binding agent, the ingredients are mixed in a small barricaded doughmixer. The pasty product so obtained is used in loading operations and volatile solvent present is removed by evaporation after being loaded in primers.

d. The sensitivity of priming compositions varies widely, and careful control of the granulations of the ingredients of each composition is necessary. Nonuniformity of composition, due to mechanical segregation of the ingredients, can cause great variations in sensitivity and consequent failure to function. This is particularly apt to occur if one of the ingredients, such as lead azide and mercury fulminate, is of much greater density than the other ingredients. A binding agent prevents such segregation. The sensitivity of priming compositions is not determined so as to be comparable with that of other explosives. In practice, the composition is loaded in primer cups, which are placed in a test apparatus equipped with a firing pin. A steel ball is allowed to fall from a predetermined height and strike the firing pin. This may cause explosion of the composition by percussion or impact, depending upon the type of primer used. By repeated trials, there are determined the maximum height of fall causing no explosion and the minimum height of fall required for explosion in all of a prescribed number of tests. By such a test procedure, the range of sensitivity of a composition is determined. This is of particular importance is a material that is to be caused to function by a mechanical method.

e. The rate of burning of a priming composition and the volume of gases and weight of solid particles produced determine the effectiveness of the composition as an ignition agent. The bursts of flame from primers loaded with various compositions have been found to have ecective durations from 400 to 750 microseconds and total durations from 650 to 1,500 microseconds. The volume of gas produced has been found to be of the order of 25 milliliters per gram of composition, at standard temperature and pressure. The proportion of the weight of the composition converted into incandescent solid particles varies considerably, but may be as much as 70 percent.

f. Priming compositions used in military ammunition are of good stability, except for those containing mercury fulminate. Those containing fulminate undergo deterioration and desensitization if stored at slightly elevated temperatures. Storage at 30° to 35° C. (85° to 95° F.) for 5 years seriously impairs for functioning of such compositions. The other compositions, containing nonexplosive materials only or lead azide, lead styphnate, TNT, or PETN are of a high order of stability. 100° C. vacuum stability test values reflect this difference in stability, fulminate compositions liberating about 10 milliliters and the other compositions about 0.3 milliliter of gas per gram of composition.

g. The simplest priming composition, one containing no explosive ingredient, is a mixture containing 55 percent potassium chlorate and 45 percent lead thiocyanate. Potassium chlorate, which readily undergoes decomposition with the liberation of oxygen

$$KClO_3 \rightarrow KCl + 1\tfrac{1}{2}O_2$$

is nonexplosive as shown by impact, pendulum friction, and sand tests. Lead thiocyanate, the fuel in the composition, has a marked sensitizing egect on the chlorate; and the mixture has an explosion temperature test value of only 240° C.

h. The simple mixture of chlorate and thiocyanate is not suitable for wide application, because of its relative insensitivity. A group of compositions that may be considered as derived from the chloratethiocyanate composition by the inclusion of explosive ingredients is shown by table 7-6.

Table 7-6. Chlorate-Thiocyanate Compositions Containing Explosive Ingredients.

	I *Percent*	*II* *Percent*	*III* *Percent*	*IV* *Percent*	*V* *Percent*
Patassium chlorate.	53	37.05	53	53	35
Lead thiocyanate.	25	38.13	25	25	17
Antimony sulfide.	17		12	17	30
Barium nitrate		8.68			
Ground glass		10.45			
Calcium silicide					15
TNT	5	5.69			3
PETN			10		
Lead azide				5	
	100	100.00	100	100	100

Photomicrographs of samples of these compositions are shown in figure 7-7. The first and third of these are loaded with the addition of a binding agent. The binder for the first composition is a mixture of gum tragacanth, gum arabic, glue, and thymol, in which the thymol serves as an antibacterial agent. A similar binding agent is used with the third composition.

i. A primer mixture used in detonators has the composition—

	Percent
Potassium chlorate	33.4
Antimony sulfide	33.3
Lead azide	28.3
Carborundum	5.0
	100.0

and a photomicrograph of this is shown in figure 7-7. In this composition, the antimony sulfide acts as both a fuel and sensitizing agent and the carborundum is also a sensitizing agent.

j. Priming compositions containing mercury fulminate as an explosive ingredient were the first such compositions developed, but now are used to only a limited extent, because of their relatively poor stability. Representative compositions of this class are shown in table 7-7.

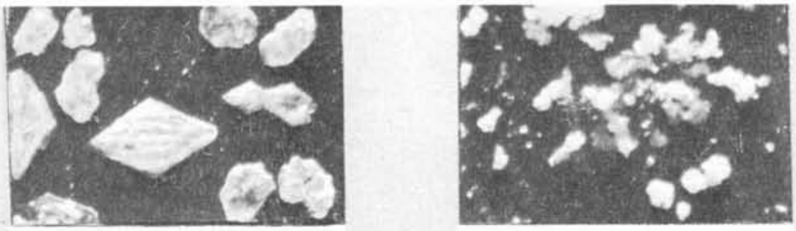
Table 7-7. Priming Compositions Containing Mercury Fulminate

	I *Percent*	*II* *Percent*	*III* *Percent*
Potassium chlorate	45	35	14
Antimony sulfide	23	30	21
Mercury fulminate	32	35	28
Ground glass			35
Shellac			2
	100	100	100

Photomicrographs of the first two of these are shown in figure 35. The first of the above compositions usually is loaded when dry but can be loaded when wet with ethanol. The second composition is used in a dry condition. The third composition is prepared with the shellac dissolved in ethanol and the composition is wet loaded.

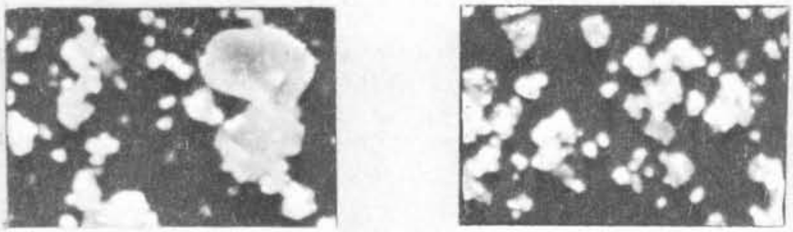
k. Priming compositions used in electric primers and electric squibs contain lead styphnate and diazodinitrophenol, respectively, as the explosive ingredients. These compositions are shown in table 7-8.

l. In certain items of ammunition such as pyrotechnic assemblies, primany ignition is brought about by frictional devices, in which a wire or rod is pulled through a perforated pellet of a priming composition.



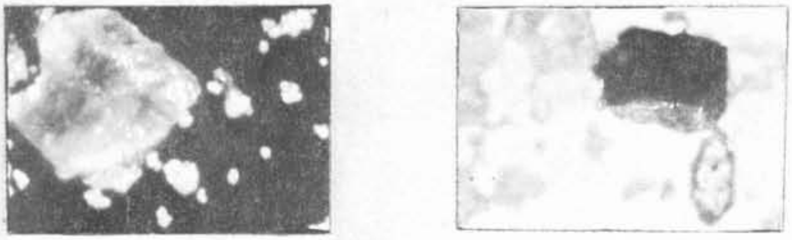
M-4 PRIMING COMPOSITION, 100X

M13 PRIMING COMPOSITION, 250X



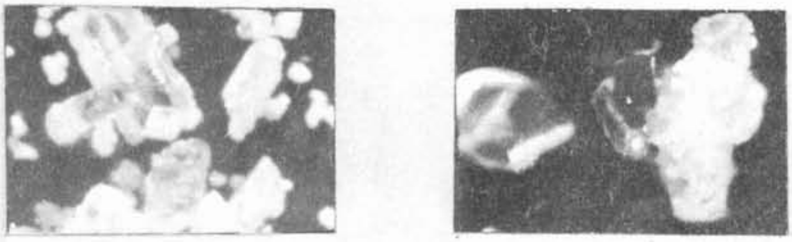
M26 PRIMING COMPOSITION, 100X

M42 PRIMING COMPOSITION, 250X



NO 70 PRIMING COMPOSITION, 100X

NO 74A PRIMING COMPOSITION, 150X



NO 90A PRIMING COMPOSITION, 100X

MK V PRIMING COMPOSITION, 100X

RA PD 167385

Figure 7-7. Priming compositions.

Table 7-8. Electric Primer and Squib Priming Compositions.

	Percent	*Percent*
Barium nitrate	44.4	
Potassium chlorate		60
Lead styphnate	39.5	
Diazodinitrophenol		20
Nitrostarch		5
Calcium silicide	14.1	
Carbon black	1.0	
Charcoal		15
Gum arabic	1.0	
	100.0	100

In some cases, the braided with is coated with a mixture of red phosphorus and shellac and the composition of the pellet is:

	Percent
Potassium chlorate	88
Charcoal	10
Dextrin	2
	100

In other cases the wire or rod is merely roughened and the pellet composition is:

	Percent
Potassium chlorate	56.2
Antimony sulfide	24.6
Sulfur	9.0
Ground glass	10.2
	100.0

7-4. Noninitiating High Explosives

a. Noninitiating high explosives comprise explosives that require initiation to detonation by another explosive and are used as booster and bursting charges and for blasting and demolition purposes. They may be divided into the following types:

(1) Single compound explosives.

(2) Binary explosives.

(3) Plastic explosives.

(4) Dynamites.

b. Single-compound high explosives include inorganic and organic compounds, with ammonium nitrate as the only important inorganic high explosive. The organic high explosive compounds are of the aliphatic, aromatic, and heterocyclic series and include nitrates, nitrocompounds, and nitramines. The aliphatic nitrates, such as nitroglycerin and nitrocellulose, were the first important high explosives and reflected the commercial availability of natural products such as glycerin, cellulose, and sugars. The development of the coal-tar industry resulted in the development of aromatic nitrocompounds, such as TNT and picric acid, as major military explosives. The rather recent development of bulk synthetic chemicals, such as ammonia, formaldehyde, acetaldehyde, and guanidine, has made practicable the development of the nitramine explosives RDX, haleite, and nitroguanidine as well as the nitrate PETN. It is probable that important future developments will be based on the nitration of glycols or polyhydroxy-compounds of synthetic origin. There are hundreds of known explosive compounds, but only a few of these can be used as military explosives, because of existing requirements with respect to sensitivity, brisance, stability, volatility, hygroscopicity, reactivity, cost, availability, etc.

c. Of the dozen standard noninitiating high explosives used for military purposes, only TNT melts at a temperature below 100° C. and can be melt-loaded with the use of low-pressure steam kettles. As melt-loading offers great advantages over press-loading, there have been developed binary explosives that consist of mixtures of TNT with another explosive, with a second explosive and a nonexplosive material such as aluminum, or aluminum alone. This has made available military explosives that are superior to TNT with respect to fragmentation and blast effect.

d. The distinct limitations, with respect to the proportions of other explosives that can be mixed with TNT and melt-loaded, the requirement for very brisant explosives that can be press-loaded without undue hazard, and the requirement for demolition explosives that can be moulded by hand have led to the development of two types of plastic explosives. One of these consists essentially of a very brisant explosive, desensitized with a wax, and suitable for press-loading in projectiles of small calibers. The second consists of a highly brisant explosive, such as RDX, and a binding agent that forms a putty-like mass.

e. Military operations sometimes involve blasting operations comparable with those involved in road-building, quarrying, removal of structures, etc. Blasting explosives similar to or identical with commercial dynamites are used for such operations.

f. The individual military explosives included in the four types (*a* above) are described herein.

7-5. Ammonium Nitrate

a. Ammonium nitrate, NH_4NO_3, has a molecular weight of 80.048 and forms colorless rhombic crystals at ordinary temperatures (figs. 7-8 and 7-9). First prepared by Glauber in 1659, it was first used as an explosive in 1867, when Ohlsson and Norrbin used it as an ingredient of what was essentially a dynamite. Although ammonium nitrate can be made to detonate under conditions of great confinement, it is so insensitive that it cannot be used alone as an explosive. It therefore has found use as an ingredient of binary explosives, dynamites, and cratering explosives.

b. Melting at 169.6° C., liquid ammonium nitrate forms cubic crystals on solidification. When cooled to 125.2° C., these undergo transition to the tetragonal system, and further cooling to 84.2° C., causes the crystals to become α-orthorhombic. At 32.3° C., there is transition to the β-orthorhombic form. This form persists to $-18°$ C., where the crystals become pseudohexagonal. Ammonium nitrate has no boiling point at atmospheric pressure, the compound decomposing when heated to 210° C.

c. Ammonium nitrate is extremely hygroscopic and is soluble to the extent of 118.3 and 871 grams per 100 grams of water at 0° and 100° C., respectively. It is soluble in ethanol and methanol to the extent of 3.8 and 17.1 grams, respectively, per 100 grams of solvent at 20° C.

d. The crystal density of ammonium nitrate is 1.725 at 25° C. Its apparent density varies widely with granulation and the purpose for which it is intended. That used for the manufacture of amatol has an apparent density of 1.06 or more.

e. The scratch hardness of ammonium nitrate crystals is slightly less than that of TNT and considerably greater than that of tetryl.

f. Ammonium nitrate has specific heat values of 0.397 and 0.428 calorie per gram per degree Centigrade at 0° and 100° C., respectively. Its heat of fusion is 18.23 calories per gram. The heat of formation of ammonium nitrate is 1098.46 calories per gram or 87.93 kilogram-calories per mole at constant pressure. When detonated, ammonium nitrate has a heat of explosion value of 346.3 calories per gram or 27.72 kilogram-calories per mole, and it liberates gaseous products to the extent of 980 milliliters per gram or 78.44 liters per mole, measured at standard temperature and pressure.

g. Manufactured by the neutralization of an aqueous solution of ammonia with nitric acid and evaporation of the solution, modern ammonium nitrate is a very pure product, since the ammonia is manufactured by the fixation of atmospheric nitrogen and the nitric acid is obtained by the catalytic oxidation of such ammonia. The final drying of the ammonium nitrate is accomplished in a graining kettle.

h. Ammonium nitrate is decomposed by strong alkalies with the liberation of ammonia and by sulfuric acid with the formation of ammonium sulfate and nitric acid. In the presence of moisture, ammonium nitrate reacts with copper to form tetraminocupric nitrate, $Cu(NO_3)_2.4NH_3$, which is of the same order of sensitivity to impact and brisance as lead azide. For this reason, tools of brass or bronze are not used in operations with explosives containing ammonium nitrate. It also corrodes iron, steel, brass, lead, and cadmium. When ammonium nitrate reacts with iron in the presence of moisture, ammonia is liberated. It has little if any effect on coatings of acid-proof black paint, shellac, baked oil, or NRC Compound.

i. Ammonium nitrate decomposes, when detonated, according to the reaction

$$NH_4NO_3 \rightarrow N_2 + 2\ H_2O + \tfrac{1}{2}\ O_2 + 27.72\ \text{kg cal}$$

but when heated under various conditions the most important modes of decomposition are

$$\underbrace{\begin{array}{l} NH_4NO_3 \rightarrow N_2O + 2H_2O + 10.7 \text{ kg cal} \\ 4NH_4NO_3 \rightarrow 2\ NO_2 + 8\ H_2O + 3\ N_2 + 96.0 \\ \quad \text{kg cal} \\ 4NH_4NO_3 \rightarrow 3\ NO_2 + 5\ H_2O + N_2 + 2\ NH_3 \\ \quad + NO - 84.88 \text{ kg cal} \end{array}}_{20_2 + 8\ H_20 + 4N_2 + 195.76 \text{ kg cal}}$$

In the last example, the decomposition is endothermic; and if the products of decomposition are heated, these react exothermically with explosive effect.

j. The grade of ammonium nitrate used in the manufacture of binary explosives is required to be at least 99 percent pure, contain not more than 0.15 percent of moisture, and have maximum ether-soluble, water-insoluble acidity, sulfate, and chloride contents of 0.10, 0.18, 0.02, 0.05, and 0.50 percent, respectively. It must contain no nitrite or free alkali. The granulation of ammonium nitrate is controlled, so that amatol of satisfactory fluidity when liquid and density when cast can be manufactured from it. Crystalline ammonium nitrate is shown in figures 7–8 and 7–9.

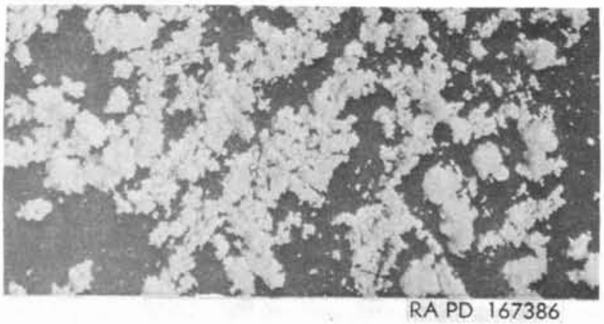


Figure 7–8. Ammonium nitrate crystals, 1½ x.

k. Ammonium nitrate is the least sensitive to impact of any of the military explosives, having an impact test value of 31 inches as compared with 17 inches for explosive D and 14 inches for TNT. At temperatures from 75° to 15° C., its impact test value is 27 or 28 inches, but the value for the molten material is only 12 inches. When subjected to the explosion temperature test, ammonium nitrate does not explode, and it is unaffected in the pendulum friction test with a steel shoe. It is not exploded in the rifle bullet impact test. In the sand test, only partial explosion of ammonium nitrate results even if boostered with a charge of tetryl or RDX. It has been found practicable to detonate larger charges, when properly confined, by means of a booster charge of tetryl but not by means of a lead azide or mercury fulminate blasting cap. The sensitivity of ammonium nitrate to initiation decreases with increase in loading density. If the density exceeds 0.9, charges of 1 to 3 pounds cannot be detonated completely by large booster charges, and larger quantities cannot be detonated completely at densities greater than 1.1. The admixture of up to 8 percent of nonexplosive carbonaceous material somewhat sensitizes ammonium nitrate to initiation. Certain very insensitive blasting explosives consist of ammonium nitrate, dinitrotoluene, and paraffin. It has been found that ammonium nitrate can be caused to detonate by heat alone, but the confinement must be such that a pressure of 2,500 psi or more can be developed prior to explosion.

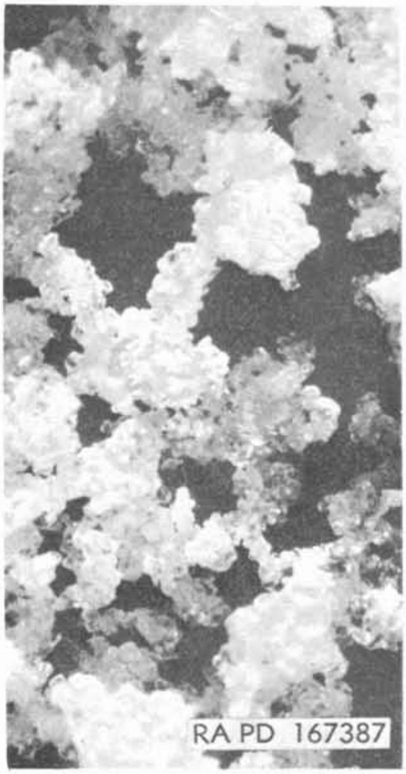


Figure 7–9. Ammonium nitrate crystals, 25 x.

l. Because of its insensitivity, the rate of detonation of ammonium nitrate is affected by its particle size, apparent density, degree of confinement, and the efficiency of the booster charge. Increase in rate is brought about by decrease in particle size, decrease in apparent density, and increase in confinement. Increase in the emperature of the charge from 15° to 140° C. has been found to result in an increase of 400 meters per second in the rate of detonation. Molten ammonium nitrate, because of its greater sensitivity, can be detonated with practically no confinement. Observed rates of detonation of ammonium nitrate under various conditions are from 1,100 to 2,700 meters per second.

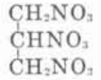
m. Both Trauzl lead block and lead cylinder compression tests indicate ammonium nitrate to be 55 percent as powerful as TNT, although their heat of explosion values would indicate ammonium nitrate to be only 37 percent as powerful. Because of its low rate of detonation, the brisance of ammonium nitrate is relatively low. Fragmentation tests in small shell of ammonium nitrate and TNT loaded with a density of 1.0 showed the nitrate to produce only 24 percent as many fragments as the TNT.

n. Ammonium nitrate is a very stable material even at 150° C., as indicated by the vacuum stability test at that temperature. It can be heated at 100° C. for 100 days without appreciable decomposition. Decomposition does not appear to begin until the compound melts. At 220° C., there are formed nitrous oxide, water, and nitrogen; and this method is used for the manufacture of nitrous oxide. If an organic material such as cellulose is present, decomposition of the mixture begins at 100° C. and is pronounced at 120° C. Admixture with TNT has little if any effect on the stability of ammonium nitrate at temperatures less than 120° C.

o. Ammonium nitrate is not toxic. It is a fire hazard since it is a powerful oxidizing agent and will increase the intensity of combustion of any flammable material mixed with or adjacent to it.

7–6. Nitroglycerin

a. Nitroglycerin, glyceryl trinitrate, or NG is a colorless liquid, which has the structure



$$\begin{array}{l} CH_2NO_3 \\ | \\ CHNO_3 \\ | \\ CH_2NO_3 \end{array}$$

and a molecular weight of 227.09. First prepared by Sobrero in 1864 or 1847, it was first used as an explosive by Nobel in 1864. It is used extensively in propellent compositions as well as in dynamites and for the shooting of oil wells.

b. Pure nitroglycerin freezes to form dipyramidal rhombic crystals, which represent a stable form melting at 13.2° C. Under some conditions, there are formed triclinic crystals, which represent a labile form melting at 2.2° C. The labile form gradually changes into the stable form after a week or two. Nitroglycerin at 145° C. decomposes with the ebullition of gaseous products of decomposition, so that it appears to boil; but it undergoes true boiling at 125° and 180° C. under pressures of 2 and 50 mm of mercury, respectively.

c. Nitroglycerin has a specific gravity value of $1.596^{\frac{20}{15}}$ and a refractive index value of $1.4732^{\frac{20}{D}}$. It has viscosity values of 0.36, 0.21, 0.094, and 0.068 poise at 20°, 30°, 50°, and 60° C., respectively. With vapor pressure values of 0.0015, 0.0075, and 0.06 mm of mercury at 20°, 40° and 60° C., respectively, nitroglycerin is volatile to the extent of 0.11 milligram per square centimeter per hour at 60° C.

d. The heat of explosion of nitroglycerin at constant pressure is 1,603 calories per gram or 368.4 kilogram-calories per mole. Its heat of detonation is 1,486 calories per gram or 337.4 kilogram-calories per mole and the gaseous products are 715 milliters per gram of 163.5 liters per mole.

e. Nitroglycerin is soluble in water to the extent of only 0.173, 0.191, 0.228, and 0.246 gram at 20°, 30°, 50° and 60° C., respectively. It is essentially nonhygroscopic when exposed to atmospheric humidity. Ethanol dissolves 37.5 and 54 grams of nitroglycerin per 100 grams of solvent at 0° and 20° C., respectively. Hot ethanol and nitroglycerin are miscible in all proportions. Nitroglycerin is miscible in all proportions with ether, acetone, glacial acetic acid, ethyl acetate, benzene, toluene, phenol, nitrobenzene, chloroform, ethylene chloride, and nitric esters, such as glycol dinitrate. Nitroglycerin has some solvent action on aromatic nitrocompounds, such as TNT and dinitrotoluene, and it is a gelatinizing agent for nitrocellulose.

f. Concentrated sulfuric acid decomposes nitroglycerin, with the formation of nitric acid. In the presence of metallic mercury, nitric oxide is formed quantitatively, and this reaction is the basis for determination of the purity of nitroglycerin by the nitrometer method. The reaction between nitroglycerin and aqueous sodium hydroxide is slow because of their immiscibility, but if ethanol is added, the reaction is rapid. The products are sodium nitrate, nitrite, formate, and acetate, with resinous material, oxalic acid, and ammonia being produced as by-products. Nitroglycerin is hydrolysed to a very slight extent by water, this being sufficient to produce less than 0.002 percent of acidity in 10 days at 22° C. or 0.005 percent of acidity in 5 days at 60° C. An aqueous solution of sodium sulfide decomposes nitroglycerin with the liberation of much heat, and this reaction can be used for the destruction of waste material.

g. Nitroglycerin is manufactured by the nitration of glycerin with a mixed acid containing about 40 percent nitric acid, 59.5 percent sulfuric acid, and 0.5 percent water. One part by weight of glycerin is added to about 4.3 parts of well-agitated mixed acid, the temperature of which is kept at 25° C. or less. When addition is complete, agitation and cooling are continued until the temperature is about 15° C. When the emulsion is allowed to stand in a separating tank, the nitroglycerin forms a supernatant layer containing about 8 percent nitric and 2 percent sulfuric acids. This is separated and agitated with water at a temperature as high as 43° C. After settling out, the nitroglycerin is washed further with water, once with a 2 percent solution of sodium carbonate and further with water, until the wash waters are free of alkali and the nitroglycerin is neutral to litmus. The product has a milky appearance because of its moisture content, but on storage in a heated building, the material becomes clear and the moisture content decreases to 0.4 percent or less. The yield of nitroglycerin is 230 ± 5 parts by weight per 100 parts of glycerin. In Europe the nitration and purification are carried out by the Schmid and Biazzi continuous processes, which also use glycerin and a mixed acid.

h. There is but one grade of nitroglycerin for military use. This must comply with the following requirements:

Moisture content: Maximum, 0.75 percent.
Acidity or alkalinity: Maximum, 0.002 percent.
Nitrogen content: Minimum, 18.40 percent.
82.2° C., KI Test: Minimum, 10 minutes.

The KI Test does not measure stability, but indicates the presence or absence of trace impurities not found in highly purified nitrogylcerin.

i. As measured by the Picatinny impact test, nitroglycerin is more sensitive to impact than mercury fulminate, and its great sensitivity to impact is generally recognized. It is sensitive to friction, as shown by the pendulum friction test. Increase in temperature increases sensitivity to impact markedly. Solid nitroglycerin is much less sensitive than the liquid compound, but many accidents have occurred when frozen dynamite was jarred while being thawed. Unconfined nitroglycerin in very small quantity burns without explosion; but if a fraction of a drop of the material is contained in a capillary glass tube and this is exposed to a flame, the nitroglycerin detonates with a loud report. Nitroglycerin is initiated to detonation by a black powder squib, but not uniformly so, and it therefore is almost as sensitive to initiation as mercury fulminate or lead azide. When compared with similar values for other explosives, the explosion temperature test value of nitroglycerin

(222° C.) does not indicate its sensitivity to initiation by heat. No data are available in connection with the sensitivity of nitroglycerin to initiation by initial detonating agents. However, the fact that 40 percent straight dynamite can be detonated by lead styphnate indicates a high degree of sensitivity, since lead styphnate will not detonate pressed PETN and this is very sensitive to initiation.

j. Nitroglycerin as such is not transported by common carrier, because of its sensitivity to shock. A mixture of 70 parts of nitroglycerin and 30 parts of acetone by weight is relatively insensitive and sometimes is transported by wagon or truck. Such a mixture can be detonated by a No. 8 blasting cap. Modified Bureau of Mines impact tests of various mixtures gave the sensitivity values shown by table 7–9.

Table 7–9. Impact Sensitivity of Nitroglycerin-Acetone Mixtures

Composition, percent		*Impact test, centimeters*
Nitroglycerin	*Acetone*	
100	0	16
90	10	23
80	20	41
75	25	60
73	27	64
70	30	100+

The nitroglycerin in such a mixture can be separated from the acetone by precipitating the nitroglycerin by addition of an excess of water or by evaporating the acetone with a current of air. An emulsion of 87-percent nitroglycerin and 13-percent water that has been stabilized with methyl cellulose also has been found to be sufficiently insensitive to permit safe handling. This mixture is not detonated by a blasting cap.

k. On the basis of sand test values, nitroglycerin is as brisant as RDX and surpassed in this respect only by PETN among the standard military explosives. When improperly initiated, nitroglycerin can undergo detonation at rates as low as 1,500 to 2,000 meters per second; but when properly initiated its normal rate is 7,700 meters per second. This is less than the rates of RDX and PETN at the same density. The temperature of explosion of nitroglycerin has been found to be 3,500° C. The Trauzl lead block test value for nitroglycerin (550 ml) is greater than that for any other military explosive and is reflected by its correspondingly highest heat of explosion value. The ballistic pendulum test values for the three compounds, however, indicate RDX and PETN to be more powerful than nitroglycerin. In this connection, it is to be noted that nitroglycerin has a ratio of combined oxygen to oxygen required for complete combustion of 105.9 percent, while RDX and PETN have corresponding ratios of 66.7 and 85.7 percent, respectively. In the Trauzl test, the samples are not in contact with air, while in the ballistic pendulum test, air surrounds the sample when placed in the explosion chamber. This would tend to increase the test value for explosives that are less than oxygen-balanced but have no effect on the test value for nitroglycerin.

l. Nitroglycerin is quite stable at temperatures less than 50° C., as has been shown by storage tests over a period of years. At higher temperatures, the rate of decomposition increases rapidly, and 100° C. vacuum stability test data show that at that temperature, it is the least stable of the standard military explosives of the noninitiating type.

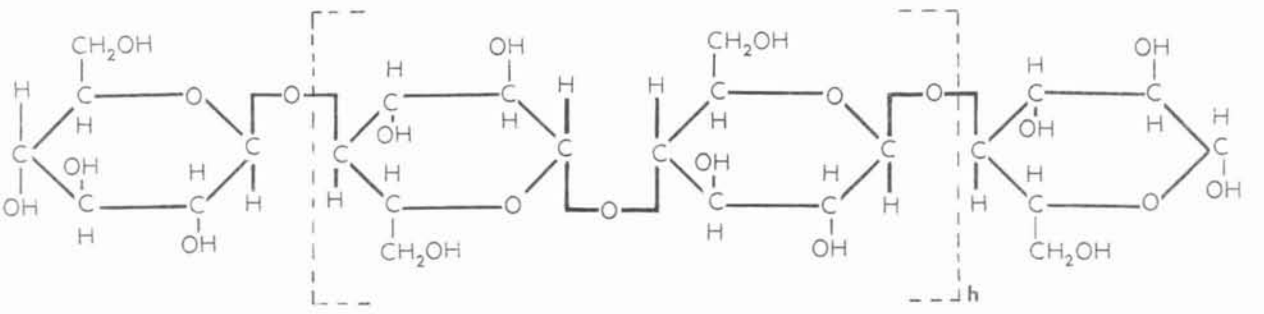
m. Because of its stability, low solubility in water, and neutrality, nitroglycerin does not cause significant corrosion of metals with which it may be in contact. Rust has been found to have a marked effect in increasing the decomposition of nitroglycerin, as indicated by 100° C. vacuum stability tests. The presence of more than a trace of free acid renders nitroglycerin quite unstable, decomposition with the appearance of red fumes taking place within a few days.

n. Nitroglycerin is readily absorbed through the skin into the circulatory system of the human body and vapors inhaled are absorbed by the blood. The effect is a severe and persistent headache, from which some relief can be obtained with strong black coffee or caffein citrate. Workers in constant contact with nitroglycerin usually develop an immunity that can be maintained only by almost daily contact. The toxicity of nitroglycerin does not cause organic deterioration, and workers do not appear to be affected by exposure over lengthy periods. Nitroglycerin is used medicinally for coronary ailments, the normal dosage being 0.0005 gram.

o. Nitroglycerin is the most hazardous explosive manufactured in relatively large quantities, and extreme precautions are taken to insure safety during its manufacture and use. Barricaded buildings, emergency drowning tanks, remote control of operations, rubber hose connections, rubber-tired transfer buggies, etc., are examples of these precautions.

7-7. Nitrocellulose

a. Nitrocellulose, cellulose nitrate, or NC is a mixture of nitrates obtained by nitrating cellulose. Cellulose is a long chain polymer of anhydroglucose units ($C_6H_{10}O_5$), usually represented by the following structural formula:



RA PD 212836

Figure E. Cellulose.

The number of anhydroglucose units (deg of polymerization or DP) varies appreciably. Cotton linters and wood pulp used for preparation of military grades of nitrocellulose have a DP of approximately 1,000 to 1,500. Cotton fiber is practically pure cellulose, and cellulose of equal purity can be separated from wood.

b. The hydrogen of the three hydroxyl (OH) groups can be replaced by NO_2 groups, in which case the nitrated anhydroglucose unit can be represented by the formula $C_6H_7O_5(NO_2)_3$, with a formula weight of 297.14 and a nitrogen content of 14.14 percent. Such complete nitration is difficult to accomplish, but material containing 14.0 percent of nitrogen can be prepared without difficulty. If the nitration is carried out under various conditions of acid concentration, temperature, and time of nitration, products containing from a few percent to 14.0 percent of nitrogen are obtained. These differ widely in their solubility characteristics and the viscosity values of their solutions. In cellulose nitrate containing less than 14.14 percent of nitrogen, the nitrate groups probably are distributed at random in the three possible positions in the anhydroglucose unit, so that nitrocellulose with a nitrogen content corresponding to that of cellulose dinitrate would represent cellulose, in which the nitration of the individual anhydroglucose units had been carried to the point where an average of two of the three replaceable hydrogens had been substituted. The dinitrate and mononitrate would have nitrogen contents of 11.11 and 6.75 percent, respectively.

c. Three types of nitrocellulose are recognized and distinguished by name. These are described in (1) through (3) below.

(1) Pyroxylin or collodion, which is soluble in a mixture of ether and ethanol, contains from about 8 to about 12 percent of nitrogen. The pyroxylin used for the manufacture of cell-

uloid contains 11.0 to 11.2 percent nitrogen, while that used in the manufacture of blasting explosives has a nitrogen content of 11.5 to 12.0 percent. The pyroxylin used for military purposes contain 12.20±0.10 percent of nitrogen. Pyroxylin is distinguished from the other types of nitrocellulose, also, by its partial solubility in ethanol.

(2) Pyrocellulose is a type of nitrocellulose which has a nitrogen content of 12.60±0.10 percent and is completely soluble in a mixture of two parts of ether and one part of ethanol. Pyrocellulose was developed by Mendeleev and, when colloided with ether-ethanol, formed the first smokeless powder for artillery used in the United States. Pyrocellulose for military use is manufactured from cotton linters or wood cellulose obtained commercially from wood pulp.

(3) Guncotton contains 13 percent or more of nitrogen. The guncotton used for military purposes contains a minimum of 13.35 percent. Only 6 to 11 percent of this type of nitrocellulose is soluble in ether-ethanol mixture; but it is completely soluble in acetone, as are pyroxylin and pyrocellulose.

d. In the manufacture of nitrocellulose propellants, there sometimes are used mixtures of pyrocellulose and guncotten termed *blended nitrocellulose.* These are designed to have desirable solubility and viscosity characteristics, as well as a specified nitrogen content of 13.15 ±0.05 or 13.25±0.05 percent.

e. The several types of nitrocellulose have thermochemical characteristics, which have been found to have linear relationships with nitrogen contents. A summary of these characteristics is shown by table 7-10.

Table 7-10. Thermochemical Characteristics of Netrocellulose

Nitrogen content, percent	*12.60*	*13.15*	*13.35*	*14.0*
Heat of combustion, cal/g:				
At constant volume	2415	2345	2320	2237
At constant pressure	2409	2338	2313	2228
Heat of formation, cal/g: At constant volume	616.7	575.5	560.8	512.5
Heat of explosion, cal/g: At constant volume, with:				
Water liquid	936	1017	1046	1140
Water gaseous	855	935.5	965	1058
Gas produced, ml/g: At constant volume, with:				
Water liquid	744	721.6	713.5	687
Water gaseous	918.5	892.7	883.2	853

f. Nitrocellulose is not truly soluble in any solvent so far as is known, it being partially or completely dispersed in a colloidal form by some solvents. Water has no such solvent action on any type of nitrocellulose. Ethanol dissolves about 30 percent of pyroxylin, but has relatively little action on pyrocellulose or guncotton. An ether-ethanol mixture dissolves pyroxylin or pyrocellulose completely, from 6 to 11 percent of guncotton, and only 1 to 2 percent of nitrocellulose having a nitrogen content of 14.0 percent. Acetone dissolves practically all of any type of nitrocellulose. Other solvents, such as ethyl acetate, nitroethane, and propylene oxide, have solvent action similar to that of acetone. Many so-called gelatinizing agents, such as dibutylphthalate, triacetin, and centralite, have a similar action when used alone or diluted with an ether-ethanol solution. Camphor disperses pyroxylin when the two are subjected to heat and pressure, the product being celluloid. Liquid nitric esters, such as nitroglycerin, are colloiding agents for nitrocellulose, and even aromatic nitrocompounds have been found to have some such effect. TNT, dinitrotoluene and mononitrotoluene have increasing effects in the order given, although toluene, like benzene, has no solvent action on nitrocellulose.

g. Nitrocellulose is somewhat hygroscopic.

Although the fineness of the material has some effect on hygroscopicity, pyrocellulose, guncotton, and 14-percent nitrogen nitrocellulose absorb moisture to the extent of approximately 3, 2, and 1 percent, respectively, when exposed to an atmosphere of 90 percent relative humidity at 30° C. This is an important property of nitrocellulose, as it deteriorates much more rapidly in the presence of moisture; and the absorption of moisture by a nitrocellulose propellant causes significant change in the ballistic value of the propellant.

h. Like other nitrates, nitrocellulose is decomposed by concentrated sulfuric acid, with the liberation of nitric acid or, in the presence of mercury, nitric oxide. As the latter reaction is quantitative, it permits determination of the nitrogen content of nitrocellulose by means of the nitrometer. Nitrocellulose undergoes saponification when heated with potassium hydroxide, with the formation of some potassium nitrate and degradation products of cellulose.

i. For military use, the manufacture of nitrocellulose in the United States is carried out by the mechanical dipper process, which has displaced other more hazardous processes. In the mechanical dipper process, about 32 pounds of dried cotton linters or wood pulp cellulose is added to a charge of about 1,500 pounds of mixed acid at a temperature of 30° C. The mixed acid is contained in a nitrator equipped with paddles, so designed that the cellulose is immediately drawn below the surface of the mixed acid. The nitration reaction is exothermic and the temperature is kept from rising above 34° C. The addition of the cellulose to the mixed acid requires about 4 minutes. Nitration, with agitation of the mixture, is continued for 20 minutes more. The slurry is then discharged through a bottom valve into a centrifuge, where most of the spent acid is removed. The acid-wet crude nitrocellulose then is forked through an opening in the bottom of the wringer into a drowning basin, where it is rapidly submerged by a heavy stream of water. It now is ready for the necessary purification process. A procedure for nitrating cellulose, which is used in Great Britain and Canada, is the Thomson displacement process. An earthenware pan with a perforated false bottom is charged with cellulose and mixed acid. A perforated plate placed on top keeps the cotton submerged during nitration and a layer of water over the plate absorbs the nitric acid fumes. After the nitration has been completed, the spent acid is run off from the bottom as cold water is added on top.

j. The mixed acid used has a composition dependent upon the type of cellulose nitrated, the degree of nitration desired, and the season of the year. During warm weather, it is necessary that the nitric acid content be slightly greater than during the winter, because of a tendency toward denitration of the mixed acid during warm weather. Representative mixed acid compositions are shown in table 7–11.

Table 7–11. Compositions of Mixed Acids for Nitrating Cellulose

	For pyrocellulose from—		*For guncotton from—*	
	Cotton linters Percent	*Wood pulp cellulose Percent*	*Cotton linters Percent*	*Wood pulp cellulose Percent*
Sulfuric acid	59.2	57.0	60.5	59.5
Nitric acid	21.5	23.5	24.5	28.5
Nitrosylsulfuric acid	3.5	4.4	4.0	3.0
Water	15.8	15.1	11.0	9.0
	100.0	100.0	100.0	100.0

Variations in composition are introduced, in order to obtain products having different solubility and viscosity characteristics than those of nitrocellulose produced with the acid compositions shown.

k. Crude nitrocellulose contains cellulose, some sulfate esters, and nitrates of partly oxidized or lydrolyzed cellulose originally present or produced by side reactions during the nitration. It is probable that forms of cellulose nitrate, less stable than others, are also present. The removal of these impurities is important if nitrocellulose of maximum stability is to be produced. This is accomplished by lengthy hydrolysis under acid conditions and subsequent beating, poaching, and washing. The acid hydrolysis, termed the "sour boil," is carried out by treating the crude nitrocellulose with boiling water containing 0.025 to 0.50 percent acid calculated as sulfuric acid. Pyrocellulose and pyroxylin are subjected to 40 hours of boiling treatment, with three changes of water during this period. Guncotton is subjected to 60 hours of boiling treatment followed by two 5-hour boiling treatments, with change of water after each treatment. It is then cut and crushed by a pulping or beating, to liberate and free acid retained in the fiber canals or fine structure of the nitrocellulose. This, generally, is accomplished by means of a type of beater used in the paper industry and known as a Jordan engine. The beating operation is carried out with a large volume of water, just sufficient sodium carbonate solution being added to preserve a slightly alkaline reaction to phenolphthalein. Beating is continued until the nitrocellulose has been reduced to the desired degree of fineness, as determined by a settling test. After the slurry from the beater has been settled and decanted, the nitrocellulose is subjected to a poaching treatment that consists of one 4-hour, one 2-hour, and two 1-hour boiling treatments with settling, decantation, and the addition of fresh water after each treatment. In the 4-hour boiling treatment, sodium carbonate to the extent of 0.5 percent of the weight of dry nitrocellulose is added. The poaching treatment is followed by not less than two washes with cold water, each wash consisting of agitation of the nitrocellulose with fresh water for at least half an hour. After the final washing, the nitrocellulose is screened through slots not more than 0.022 inch wide, in order to remove any incompletely pulped fibers.

l. Nitrocellulose produced by the foregoing nitration and purification processes is of maximum stability. It generally is faintly alkaline as indicated by an electrometric pH test, and contains less than 0.01 percent of cellulose sulfate. Nitrocellulose is shown in figures 7–10 and 7–11.

m. Military grades of nitrocellulose are:

	Class	*Nitrogen, percent*
Grade A	Pyrocellulose	
Type I		12.60±0.10
Type II		12.60±0.15
Grade B	Guncotton	13.35 minimum
Grade C	Blended	
Type I		13.15±0.05
Type II		13.25±0.05
Grade D	Pyroxylin	12.20±0.10

In addition, there is a long-fiber nitrocellulose of the guncotton class, produced by nitrating long fiber cotton and purifying without a pulping operation. Long fiber nitrocellulose is required to have a nitrogen content of not less than 13.0 percent and is used in electric primers for cannon.

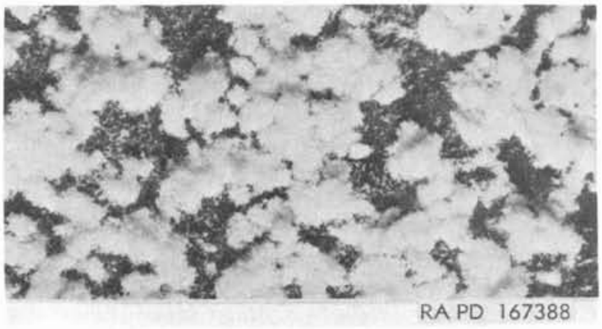


Figure 7–10. Nitrocellulose fibers, 1½x.

n. In addition to requirements with respect to nitrogen content, nitrocellulose must comply with certain requirements when subjected to the 65.5° C. KI and 134.5° C. heat tests. Grades A and D material must be not less than 99 percent soluble in ether-ethanol mixture. Grade A (type I), grade C, and grade D nitrocellulose must contain not more than 0.4 percent ash and not more than 0.4 percent of material insoluble in acetone. Requirements, with respect to fineness and viscosity, are those specified in the contract or order. Long fiber nitrocellulose must comply with similar requirements for ash and acetone-insoluble material and be not more than 15.0 percent soluble in ether-ethanol mixture.

o. Dry nitrocellulose is very sensitive to impact, friction, heat, and spark and is never handled in quantity, as such, in the United States. Impact tests show nitrocellulose to be almost as sensitive as mercury fulminate. While the explosion temperature test value for nitrocellulose (230° C.) is not particularly low, the rapid rate of decomposition of the material at temperatures greater than 100° C. and the exothermicity of such decomposition make the material very sensitive to ignition by a spark. Nitrocellulose is of the same order of sensitivity to initiation as lead azide, and it can be detonated even when wet. A nitrocellulose-water mixture (containing 40 percent water) confined in a steel barrel sometimes is detonated by a stick of dynamite, and the frequency of detonation is greater when the water is frozen. Tests have shown nitrocellulose (uniformly wetted with 35 percent ethanol) will also detonate, on occasion, when initiated with dynamite.

Figure 7-11. Nitrocellulose fibers, 75 x.

p. The brisance of nitrocellulose is directly comparable with that of TNT and less than that of tetryl, as determined by the sand test. The brisance of nitrocellulose increases with increase in the nitrogen content, and comparative values are given in table 7-12.

Table 7-12. Brisance of Nitrocellulose

Nitrogen content, percent	*Sand test, grams of sand*
12.60	45.0
13.15	48.0
13.35	49.0
14.0	52.3

The rate of detonation of guncotton (7,300 meters per second) is greater than that of TNT. As nitrocellulose propellants can be caused to detonate as well as burn, their brisance values are high. Trauzl lead block tests show guncotton to be more powerful than tetryl, but its heat of explosion is more comparable with that of TNT.

q. Nitrocellulose, even when highly purified, is much less stable than most of the noninitiating military high explosives, as judged by elevated temperature tests. It appears to undergo very slow decomposition even at ordinary temperatures, the rate of decomposition increasing 3.71 times with each increase in temperature of 10° C. The presence of moisture increases the rate of decomposition considerably and the presence also of free acid or alkali has an even more pronounced effect. Rates of decomposition of pyrocellulose under various conditions are shown by table 7-13.

Table 7-13. Decomposition of Nitrocellulose

Decomposition medium	*Percent available nitric acid liberated per hour at—*	
	50° C.	*97.5° C.*
Heat	0.0000045	0.0028
Water	0.0000111	0.0051
0.06 percent Nitric acid solution	0.0000325	--------
0.035 percent Nitric acid solution	--------	0.0088
0.035 percent Sodium carbonate solution.	0.0006870	0.1358
95 percent Ethanol	0.0000290	--------

From these data, it is apparent that alkali is more effective than acid in causing the deterioration of nitrocellulose.

r. The great care taken in the purification of nitrocellulose is due to the necessity for removing impurities that are much less stable than nitrocellulose itself. Cellulose sulfate is unstable, with respect to heat and moisture. Propellants made from nitrocellulose containing even comparatively small amounts of such esters give decreased 134.5° C. heat test values and deteriorate more rapidly. The little known nitrates of oxidized cellulose, also, are objectionable and cause increased instability of nitrocellulose, if not removed during the purification process.

s. The greater the degree of nitration of nitrocellulose, the less susceptible it is to hydrolysis. When subjected to the action of boiling water for 10 days, the several grades of nitrocellulose are hydrolyzed, as shown by table 7-14.

Table 7-14. Hydrolysis of Nitrocellulose

	Nitrogen, percent	*Percent available nitric acid liberated*
Pyroxylin	11.13	1.71
Pyrocellulose	12.62	1.22
Guncotton	13.44	1.03

The relationship shown by these data is nearly linear.

t. Vacuum stability tests of the different grades of nitrocellulose at various temperatures have shown the stability of nitrocellulose to decrease with increase in nitrogen content. Representative test values are given in table 7-15.

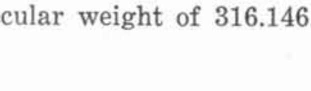
Table 7-15. Vacuum Stability Tests of Nitrocellulose

Nitrogen content, percent	Vacuum stability test at—					
	90° C.		100° C.		120° C.	
	Milliliters	Hours	Milliliters	Hours	Milliliters	Hours
12.60	0.17	40	1.0	40	11+	16
13.15	0.23	40	----	----	----	----
13.30	0.30	40	----	----	11+	16
13.45	0.42	40	1.5	40	11+	16
13.75	0.92	40	----	----	----	----
13.95	1.46	40	11+	24	11+	16

u. The practical problem of the storage life of nitrocellulose propellants, due to the inherent instability of nitrocellulose, is not as serious as it once was. Improved nitration and purification procedures and better control of the quality of the cellulose used have made practicable the production of nitrocellulose of improved stability. The formulation of propellant compositions with moistureproofing agents and more effective stabilizers has insured better protection of the nitrocellulose and longer prevention of acceleration of its rate of thermal decomposition.

7-8. Pentaerythrite Tetranitrate (PETN)

a. Pentaerythrite tetranitrate, PETN, penta, pentrit, or nitro pentaerythrite is an aliphatic nitrate having the structure



$$\begin{array}{c} \mathrm{CH_2O{-}NO_2} \\ \mathrm{O_2N.O.H_2C{-}C{-}CH_2O.NO_2} \\ \mathrm{CH_2O{-}NO_2} \end{array}$$

and a molecular weight of 316.146. As manufactured, it forms white prismatic needles, which crystallize from water as tetragonal crystals (figs. 7-12 and 7-13). PETN was first prepared in 1901 by Vignon and Gerin. The compound was not used on a practical basis until after World War I. It has been used extensively in admixture with TNT for the loading of small caliber projectiles and grenades and has been used to some extent in detonating fuse, boosters, and detonators. Foreign uses have included, also, the press-loading of mixtures of PETN and a wax.

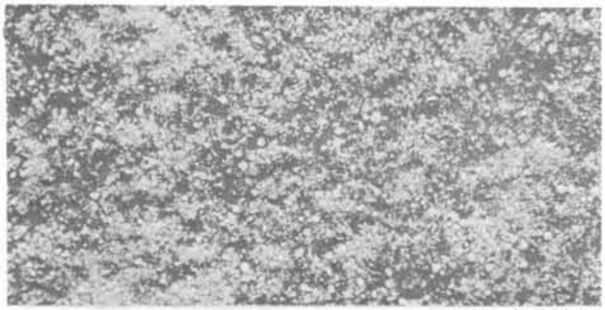


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Figure 7-12. PETN crystals, 1½ x.

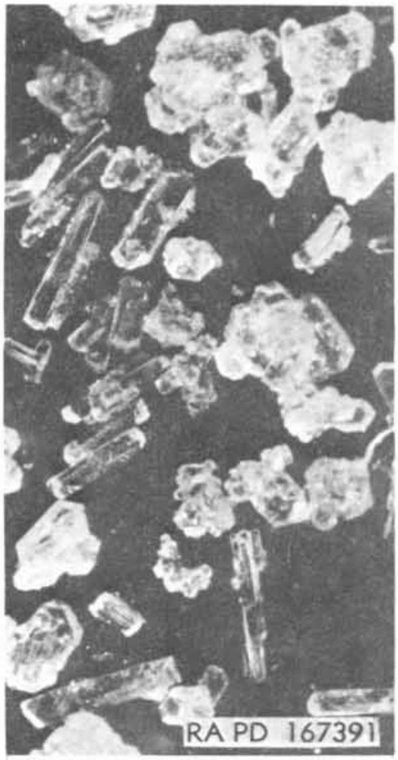

Figure 7-13. PETN crystals, 25 x.

b. Highly purified PETN melts at 141.3° C. It boils at 160° and 180° C. under pressures of 2 and 50 mm, respectively; at atmospheric pressure, it decomposes rapidly, and, in some cases, explosively at temperatures greater than 210° C. PETN crystals have a density of 1.765 and can be pressed to a density of 1.74 when subjected to a pressure of 40,000 psi. The crystals have a scratch hardness of slightly less than 2 on Mohs' scale and therefore are harder than TNT crystals and softer than RDX crystals. The compressed crystals have a specific heat value of 0.4 calorie per gram per degree Centigrade. The heat of combustion of PETN at constant pressure is 1,974 calories per gram and from this is derived a heat of formation value of 119.4 kilogram-calories per mole.

c. At 25° and 96° C., PETN is soluble in water to the extent of only 0.0043 and 0.018 gram, respectively, per 100 grams: and the compound is nonhygroscopic when exposed to an atmosphere of 90 percent relative humidity at 30°C. The solubility of PETN is some other solvents is shown by table 7–16.

Table 7–16. Solubility of PETN

° C	Solubility in grams per 100 grams of solvent at—					
	0	20	30	40	50	70
Methanol	0.19	0.46	0.71	1.17	1.87	
Ethanol	0.07	0.11	0.28	0.42	0.71	2.27
Ether	0.20	0.25	0.34			
Acetone	15.6	25.4	33.2	44.0	56.6	
Benzene	-------	0.30	0.45	1.17	2.05	5.71
Toluene	0.15	0.23	0.43	0.62	1.11	3.40
Carbon tetrachloride	-------	0.10	0.11	-------	0.12	
Methyl acetate	-------	12.9	17.0	21.7	28.0	
Cyclohexanol	-------	-------	<0.2	0.3	0.5	

d. PETN is decomposed much more slowly than is nitrocellulose by a boiling 2.5 percent solution of sodium hydroxide, several hours being required for complete decomposition. At 50° C., a solution of sodium sulfide decomposes PETN slowly, but a boiling solution of ferrous chloride decomposes it with fair rapidity. PETN does not reduce Fehling's solution even on boiling; in this respect it differs from some other aliphatic nitrates. At ordinary temperature or 50° C., dry PETN does not corrode copper, brass, aluminum, steel, stainless steel, cadmium, nickel, or zinc; in the presence of moisture, it causes slight corrosion of brass, cadmium and, zinc.

e. While PETN can be manufactured by treating pentaerythritol with nitric acid and adding concentrated sulfuric acid to complete the separation of the PETN, manufacture in the United States has been with nitric acid alone:

$$C(CH_2OH)_4 + 4\ HNO_3 \rightarrow C(CH_2NO_3)_4 + 4\ H_2O$$

This is accomplished by adding approximately 75 pounds of pentaerythritol to 350 pounds of 96 percent nitric acid in a nitrator, with continuous stirring and cooling of the acid. The pentaerythritol is added at such a rate that, with an initial acid temperature of 18° C., the temperature increases to and is maintained at 22° to 23° C. Stirring and cooling are continued for 20 minutes after addition of the pentaerythritol is complete. The acid solution then is added, with agitation, to about 850 pounds of cold water in a drowning tank. The precipitated PETN is caught on a glass-cloth filter and washed with water. It is then mixed with 1,300 gallons of cold water containing 2 pounds of sodium carbonate and separated from the slurry by refiltering. After being washed again with water, the PETN is dissolved in 440 pounds of 98-percent acetone heated to 50° C. and containing 14 ounces of ammonium bicarbonate. The solution is filtered and the PETN precipitated by the addition of cold water to the acetone solution. The precipitated solid is caught on a filter and washed with water to remove acetone. The water-wet material is considered the final product, as it generally is not dried before being used. The yield of PETN by this process is approximately 93 percent of the theoretical. The spent acid resulting from the drowning operation contains approximately 20 percent nitric acid. This is recovered and concentrated. The mother liquor, resulting from the precipitation of PETN, contains approximately 25 percent acetone, which also is recovered.

f. The purity of PETN produced by this process depends upon that of the pentaerythritol nitrated. Ordinarily, the pentaerythritol used for nitration contains 2 or 3 percent of dipentaerythritol and a small amount of tripentaerythritol:

$(HOH_2C)_3C.\ CH_2.O.CH_2.\ C(CH_2OH)_3$ and

$(HOH_2C)_3C.\ CH_2O.\ CH_2C(CH_2OH)_2.CH_2.O.CH_2\ C(CH_2OH)_3$

PETN produced on a large scale contains corresponding amounts of the hexanitrate and octanitrate of these compounds, respectively. But one grade of PETN is used for military purposes, and this complies with the following requirements:

Color: White or light buff.
Moisture[1]: Minimum, 40 percent.
Melting point: Minimum, 140° C.
Nitrogen content: Minimum, 17.50 percent.
Acetone insoluble[1]: Maximum, 0.10 percent.
Grit[1]: None.
Acidity of alkalinity[2]: Maximum, 0.005 percent.
120° C. Vacuum stability test: Maximum, 5 milliliters of gas from 2.3 grams in 20 hours.

Granulation:

Through sieve No.		*Class A*	*Class B*	*Class C*	*Class D*
30	Minimum	-----	-----	95	100
80	Minimum	100			
100	Maximum	-----	-----	-----	20
100	Minimum	85	95	-----	5
140	Maximum	55			
200	Maximum	30	80	30	
200	Minimum	-----	65		

Class A PETN is used in detonating fuse and boosters, class B is used in priming compositions, class C is used in the manufacture of pentolite, and class D is used in blasting caps and detonators. As pure PETN has a nitrogen content of 17.72 percent and a melting point of 141.3° C., the military grade is approximately 99 percent pure. The grit requirement is important, because of the sensitivity of PETN and known effect of gritty material in increasing sensitivity. The acidity or alkalinity requirement is important, since the presence of as little as 0.01 percent of either has been found to accelerate the deterioration of PETN to a marked degree. The granulation requirements are those found optimum for specific uses of PETN.

g. PETN is not as sensitive to impact as nitroglycerin or nitrocellulose, but is slightly more sensitive than RDX and distinctly more so than tetryl. It is less sensitive to friction than RDX and more sensitive than nitroglycerin, as judged by the pendulum friction test. Explosion temperature test values indicate PETN to be as sensitive to heat as nitroglycerin or nitrocellulose. However, the minimum temperature required for the explosion of PETN (215° C.) is greater than those required for the explosion of nitroglycerin (210° C.) and nitrocellulose (175° C.). Peculiarly, PETN is relatively insensitive to electric sparks, being much less sensitive than TNT or tetryl. However, it is more sensitive than RDX in this respect. PETN is more sensitive to initiation than nitrocellulose, RDX, or tetryl, as judged by the sand test. This is shown, also, by the fact that PETN with 35 percent of water present can be detonated by a No. 6 electric blasting cap, whereas RDX fails to explode if more than 14 percent of water is present. In spite of these somewhat inconsistent data, the overall sensitivity level of PETN is such as to make it one of the most sensitive military explosives of the noninitiating class.

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[1] Not applicable to Class C PETN used in coprecipitated pentolite.
[2] Not applicable to Class C PETN.

h. As measured by the sand test, PETN is the most brisant of the military high explosives, with RDX approaching it closely. Plate dent tests indicate PETN to be slightly less brisant than RDX, and the results of fragmentation tests of mixtures of PETN and wax and RDX and wax are in agreement with the plate dent test values. The slightly higher rate of detonation value of RDX, also, would indicate it to be the more brisant of the two. Ballistic pendulum and Trauzl lead block test values indicate PETN and RDX to be almost equal in power, although, PETN has a distinctly greater heat of explosion value than RDX. In general, it may be considered that PETN, RDX, and nitroglycerin are approximately equal in brisance and explosive power and, in these respects, are the most potent of the standard military explosives.

i. Vacuum stability tests at 100° and 120° C. show PETN to be more stable than nitrocellulose or nitroglycerin at elevated temperatures but distinctly less stable than RDX, tetryl, or TNT. It is quite stable at 100° C., as it withstands heating at this temperature for 100 hours without significant deterioration. Storage at 65° C. for 20 months does not cause instability or undue acidity; and after 24 months only slightly excessive acidity is developed. When 0.01 percent of free acid or alkali is present, storage for only 15 months at 65° C. is required to cause rapid acceleration of the rate of decomposition, as judged by the free acid content. As RDX, tetryl, and TNT are even more resistant to storage at 65° C., PETN is not as suitable for storage and use under tropical conditions.

j. PETN is not unduly toxic, since it is nearly insoluble in water and usually is handled while wet. It, therefore, cannot be absorbed through the skin and inhalation of the dust is improbable. Tests have shown that small doses of PETN cause decrease in blood pressure and larger doses cause dyspnea and convulsions.

k. Sheet PETN.

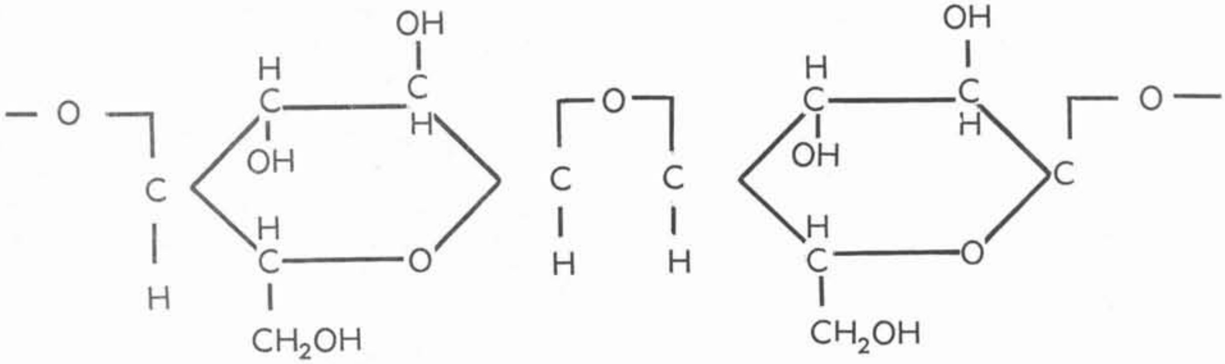
(1) A newly developed demolition charge consists of sheets of a highly flexible explosive which neither crazes or exudes when subjected to prolonged storage at high or low service temperatures. It is olive green in color and since it is unaffected by water submersion, can be initiated while underwater. Each sheet is prepared with pressure sensitive tape which can be quickly applied to most dry surfaces warmer than 32° F. It is considered to be superior to Comp C-4 demolition blocks in these respects:

- (*a*) Greater efficiency per unit weight because it covers a greater surface area.
- (*b*) Rapid attachment to target.
- (*c*) Flexibility at all temperatures.
- (*d*) Ease of cutting and forming to the desired shape.
- (*e*) Cleanliness in handling.

(2) This explosive is used commercially for explosive forming, cutting, or hardening of metals since it can be quickly applied in sheets of uniform thickness. It is composed of 63 percent PETN with pyrocellulose and plasticizer added.

7-9. Nitrostarch

a. Nitrostarch or starch nitrate is a mixture of nitrates obtained by nitrating starch. Its appearance is practically the same as that of the unnitrated starting material. The starch molecule consists of about 1,000 anhydroglucose units



RA PD 212837

Figure F. Nitrostarch.

having a configuration, which results in spiral arrangements of the units that are three-dimensional. The effect of this is the granular form of particle characteristic of starches instead of the long-chain threadlike molecules of cellulose formed by several thousand anhydroglucose units (fig. 7–14).

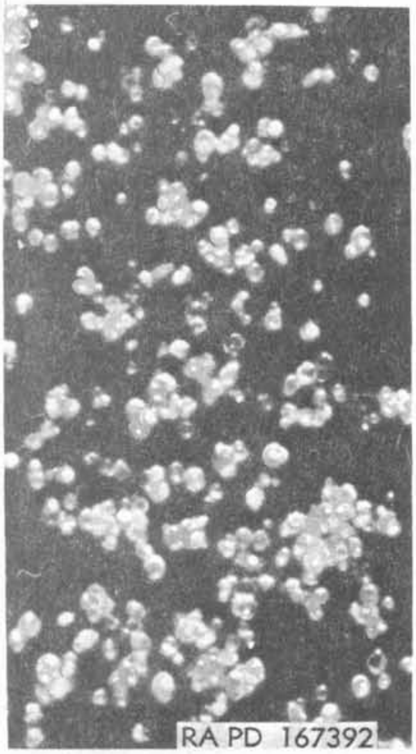

RA PD 167392

Figure 7–14. Nitrostarch granules, 100 x.

b. Starch was first nitrated by Braconnot in 1833, the product being termed *xyloidine*. Later European practice in the manufacture of nitrostarch consisted of dissolving the starch in strong nitric acid and precipitating the nitrate by the addition of sulfuric acid or spent acid from another nitrating operation. Like the nitrocellulose produced during the earlier years of its history, such nitrostarch was incompletely purified and of very poor stability. The pulverulent, dusty nature of the dry material was also considered a disadvantageous characteristic when compared with the fibrous structure of nitrocellulose. Cotton and wood are the major sources of supply of cellulose, and starch might logically be considered another major source of a comparable material. However, because of the relatively small size and form of its molecule, the physical characteristics of nitrostarch are such that it does not yield tough colloids like nitrocellulose when gelatinized. It therefore is much less suitable than nitrocellulose for the manufacture of propellants; and its use from a practical viewpoint is more or less restricted to that of an ingredient of detonating explosives. Like nitrocellulose, nitrostarch is an important ingredient of some commercial blasting explosives. It has also been used in the United States as an ingredient of military bursting charge explosives for grenades and trench mortar shell, demolition explosives, and a priming composition. Such military use has been chiefly during World Wars I and II and may therefore be considered to be essentially of an emergency nature.

c. From a chemical viewpoint, nitrostarch may be considered to be another form of nitrocellulose. It has nitrogen contents comparable with those of the various types of nitrocellulose and the thermochemical characteristics of nitrostarch are similar to those of nitrocellulose of the same nitrogen content. Nitrostarch, unlike starch, gives no color with iodine. It is decomposed by sulfuric acid in the presence of mercury, to yield nitric oxide quantitatively, and its nitrogen content therefore can be determined by means of the nitrometer. When dissolved in nitric acid and allowed to stand, nitrostarch is decomposed. Like nitrocellulose, it is saponified by aqueous alkali. It is insoluble in water, but is completely dispersed by acetone to form a colloidal 'solution. Ether-ethanol mixture has solvent action depending upon the nitrogen content of the nitrostarch, this action decreasing with increase in nitrogen content. Like nitrocellulose, nitrostarch can be gelatinized by a wide variety of agents such as esters. It is hygroscopic to the extent of 1 to 2 percent.

d. Nitrostarch is manufactured by the nitration of starch with mixed acid. The starch used is produced from corn or cassava and can be obtained from potatoes. This is purified to some extent by washing with a dilute sodium hydroxide or ammonia solution to remove fats and pectic acid and then with water. After being dried so that the moisture content is less than 0.5 percent, the starch is added to approximately four times its weight of mixed acid in a nitrator, the temperature being prevented from exceeding 38° to 40° C. The composition of the mixed acid varies with the degree of nitration desired, an acid for nitration to 12.75 percent nitrogen containing 38 percent nitric acid and 62 percent sulfuric acid. After nitration is complete, the contents of the nitrator are drowned in cold water and the nitrostarch caught on a filter. Purification is effected by washing with cold water, with the addition of ammonia during the preliminary washing, until all traces of free acid are removed. After separation on a filter or in a centrifugal wringer, the nitrostarch is dried on trays in a dry house, heated with air at a temperature of 35° to 40° C. The drying operation is the most dangerous of those involved in the manufacture of nitrostarch, as the dry material is sensitive to ignition by spark and burns with great violence.

e. There is no standard grade of nitrostarch used for military purposes, there being specified a commercial grade having a nitrogen content from 12.8 to 13.3 percent.

f. Nitrostarch is slightly less sensitive to impact than guncotton, having a test value of 5 inches, as compared with 3 inches for guncotton. The explosion temperature test value of nitrostarch (217° C.) is essentially the same as that for nitrocellulose (230° C.). The brisance and power of nitrostarch are similar to those of nitrocellulose of comparable nitrogen content. Nitrostarch is less stable than nitrocellulose, as judged by 120° and 134.5° C. heat tests; at ordinary temperatures, the two appear to be of similar stability as judged by long term storage tests.

g. Nitrostarch is used in explosive compositions chiefly as a substitute for nitroglycerin rather than nitrocellulose. When so used, it has the major advantage of making the composition nonfreezing and not subject to the desensitization that accompanies the freezing of nitroglycerin explosives. Nitrostarch explosives have been used as successfully in the Antarctic and Arctic regions as in temperate climates. Such explosives are free from the tendency to undergo leakage and the volatility effects on personnel in hot climates peculiar to nitroglycerin explosives.

7–10. Trinitrotoluene (TNT)

a. α or 2,4,6-trinitrotoluene, TNT, trotyl, tolite, triton, tritol, or trilite is the most important of the six isomeric trinitrotoluenes. It is an aryl nitrocompound having the structure

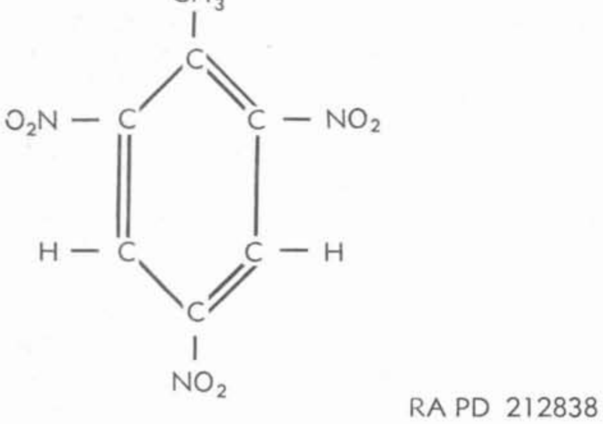


Figure G. TNT.

Figure 7-15. TNT crystals, 30 x.

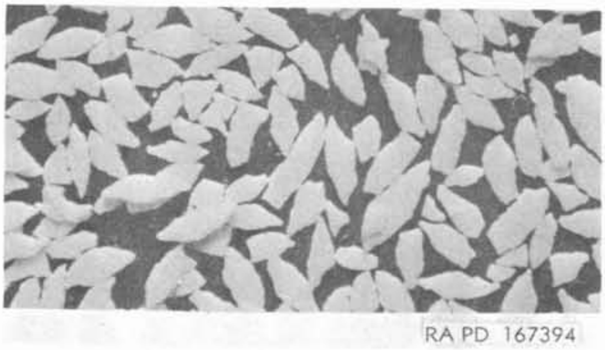


Figure 7-16. Flaked TNT, 1½ x.

and a molecular weight of 227.134. It forms colorless or light yellow rhombohedral crystals (figs. 7–15 and 7–17). It was first prepared by Wilbrand in 1863 and on an industrial scale in Germany in 1891. Beginning in 1901, Germany started to manufacture TNT on a commercial scale, and in about 1902, the German Army adopted it as standard filling for shells. Other countries slowly followed the German example, and by the beginning of World War II, nearly all of them used it. During World War I, its use was the maximum permitted by the available supply of toluene from the coke industry. The industrial development of synthetic toluene from petroleum just prior to World War II made available in the United States an almost unlimited supply of this raw material. Because of its suitability for melt-loading (figs. 7–16 and 7–18) and the formulation of binary explosives, also suitable for melt-loading, TNT was produced and used on an enormous scale during World War II and may be considered the most important military bursting charge explosives. Alone or as an ingredient of binary explosives, it has found wide application in shell, bombs, grenades, demolition explosives, and propellent compositions.

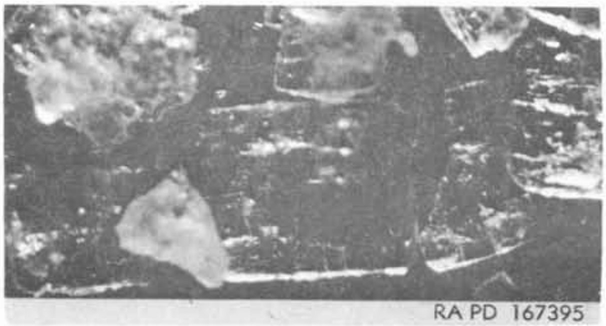


Figure 7-17. Crystalline TNT, 30 x.

b. Pure TNT has a freezing point of 80.75± 0.05° C. The freezing point is more reproducibly determined than the melting point and is used for judging the purity of TNT, since the impurities commonly present are soluble in molten TNT and cause depression of the freezing point. Unlike some other high explosives, TNT does not undergo partial decomposition when melted, and it has been found that TNT can be melted and solidified at least 60 times without significant decrease in its freezing point. At ordinary temperatures, TNT is essentially nonvolatile, its vapor pressure at 85° and 100° C. being only 0.053 and 0.106 mm, respectively. Under pressures of 2 and 50 mm, it boils at 190° and 245° to 250° C., respectively. It may be distilled in a vacuum without decomposition.

c. TNT has a crystal density of 1.654. When cast, it has a density of 1.56±0.01. When subjected to pressures of 3,000 5,000, 10,000, 15,000, 20,000, and 50,000 psi it has density values of 1.34, 1.40, 1.47, 1.515, 1.55, and 1.6, respectively. The density of liquid TNT is 1.465. The expansion in volume of TNT on melting is approximately 7 percent based on the crystal density. The viscosity of liquid TNT is 0.139 and 0.095 poise at 85° and 100° C., respectively.

d. The scratch hardness of TNT crystals is 1.2 on the Mohs' scale and therefore is intermediate between talc and gypsum. TNT has index of refraction values of $\alpha = 1.5430$, $\beta = 1.6742$ and $\gamma = 1.717$ for sodium light. When pressed to a density of 1.6, TNT has a compressive strength of 1,400 psi and a modulus of elasticity of 5.4×10^{10} dynes per square centimeter.

e. At temperatures of 0°, 20°, 50°, and 80° C. TNT has respective specific heat values of 0.309, 0.328, 0.353 and 0.374 calorie per gram per degree Centigrade. Its heat of fusion is 21.41 calories per gram. At 25° C., the thermal conductivity of TNT is 0.00054 calorie per second per centimeter per degree Centigrade. The coefficient of linear expansion of cast TNT varies somewhat with crystal size, but for crystals of medium size, it is 7.7×10^{-5} inch per inch per degree Centigrade over the range −40° to 60° C. The heat of combustion of TNT at constant pressure is 3,589.5 calories per gram, and from this is derived a heat of formation value of 16.37 kilogram-calories per mole.

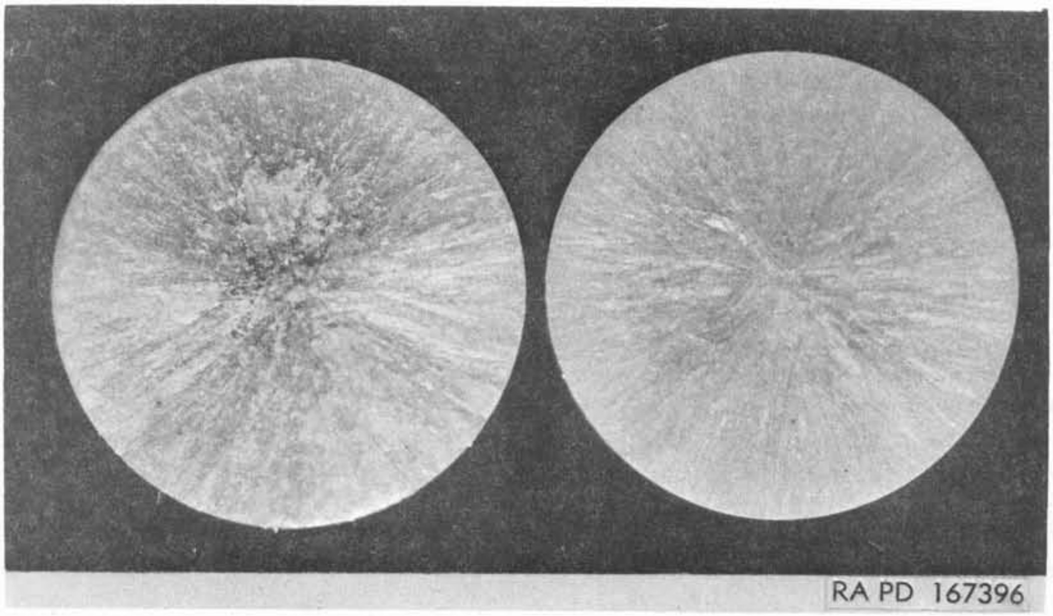


Figure 7-18. Cast TNT, 1½ x.

f. TNT is very slightly soluble in water (0.010, 0.013, and 0.1475 gram per 100 grams of water at 0°, 20°, and 100° C, respectively) and is hygroscopic to the extent of only 0.03 percent when exposed to an atmosphere of 90 percent relative humidity at 30° C. The solubility of TNT in other solvents is shown by table 7-17. In the past, ethanol, carbon tetrachloride, and sulfuric acid were used for the purification of crude TNT by recrystallization, but chemical purification by means of sodium sulfite has replaced recrystallization.

g. As compared with tetryl, picric acid, and other aryl nitrocompounds, TNT is relatively nonreactive; but it can form compounds by either association or reaction. With basic compounds, such an aniline, pyridine, diphenylamine, the toluidines, the naphthylamines, 2-methylindole, and carbazole, it forms molecular compounds having characteristic colors and melting points. Alkalies, alkoxides, and ammonia react with TNT to form dangerously sensitive compounds. A mixture of TNT and solid potassium hydroxide bursts into flame when heated to only 80° C. Sodium carbonate reacts with TNT to form a black solid, which is soluble in water or methanol, melts above 200° C. with decomposition, is as sensitive to impact and heat as tetryl, and is very unstable at 120° C. With formaldehyde and ammonium hydroxide, TNT reacts to form a black solid, which softens at about 90° C, is less brisant and sensitive to impact than TNT, and is unstable at 100° C. Potassium methylate and TNT react to yield a dark red powder, which inflames or explodes when heated at 130° to 150° C. and has been reported to explode spontaneously on standing at ordinary temperature. TNT and formaldehyde react with the formation of 2,4,6-trinitrophenylethanol. Sodium sulfide decomposes TNT completely, with the formation of nonexplosive products. Upon oxidation with chromic or nitric acid, TNT yields 2,4,6-trinitrobenzoic acid, which, on boiling in water, loses CO_2 and is converted to 2,4,6-trinitrobenzene. Exposure to sunlight, or ultraviolet light in the presence of oxygen causes progressive discoloration and decomposition with increase in sensitivity to impact. After exposure to ultraviolet light for 175 hours, a sample of TNT was brown in color, had an impact test value of 45 instead of 100 centimeters and a melting point of 79.8° instead of 80.7° C.

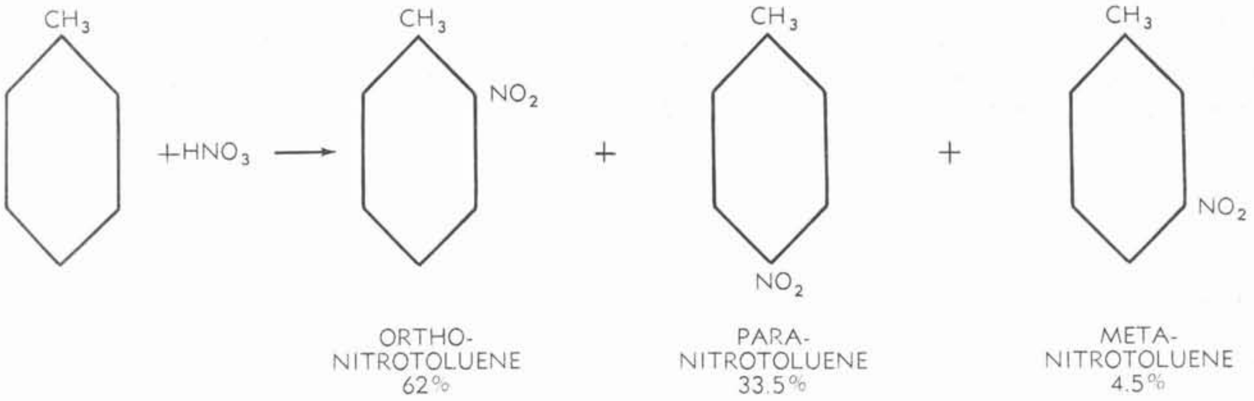
Table 7-17. Solubility of TNT

° C	Solubility in grams per 100 grams of solvent at—					
	0	20	25	30	50	75
Ethanol*	0.65	1.23	1.48	1.80	4.61	19.5
Ether	1.73	3.29	3.80	4.56		
Acetone	57	109	132	156	346	
Carbon tetrachloride	0.20	0.65	0.82	1.01	3.23	24.35
Chloroform	6	19	25	32.5	150	
Ethylene chloride	-------	18.7	22	29	97	
Benzene	13	67	88	113	284	
Toluene	28	55	67	84	208	
Carbon disulfide	0.14	0.48	0.63	0.85		
Methyl acetate	-------	72.1	80	99	260	
Triacetin	-------	-------	37.7			
Butyl carbitol acetate	-------	24				
Sulfuric acid	-------	4				

*95 percent.

h. TNT can be manufactured by one-, two-, or three-stage nitration processes, with toluene and mixed acid as the raw materials. All three have been used on a production basis, but the three-stage process has been found to have the advantages of maximum yield, greater purity of product, and greater ease of control of acid concentration and temperature conditions. Prior to World War II, each stage of nitration was carried out by adding the nitrating acid to the toluene, mononitrotoluene, or dinitrotoluene. During that conflict, there was developed the 'direct nitration process,' in which the toluene, mononitrotoluene, and dinitrotoluene are added to the mixed acid. This, with other improvements, resulted in reduction of the time required for a nitration cycle from 2 hours to 45 minutes and increase in the daily production of TNT from a manufacturing 'line' from 30,000 to 120,000 or 130,000 pounds. The nitration time for the mono and dinitrations was 45 minutes each and that for the tri-stage 90 minutes. However, by having two trinitrators for each dinitrator, it was possible to start a new batch in the mononitrator each 45 minutes, although the total time in the nitrators was 180 minutes. It was this development, along with that of synthetic toluene from petroleum, that permitted the potential production of TNT in the United States to be increased from approximately 700,000,000 to 3,600,000,000 pounds per year. This was accompanied by a reduction in cost from 15 to 6 cents per pound.

i. When toluene is nitrated in the mononitro-stage, the chief products are:



RA PD 212839

Figure H. Nitrated toluene products.

As these compounds have melting points of —10.6°, 51.3°, and 15.5° C., respectively, the product is an oily liquid at ordinary temperatures. When this is subjected to dinitration, the products are the following isomeric dinitrotoluenes (DNT):

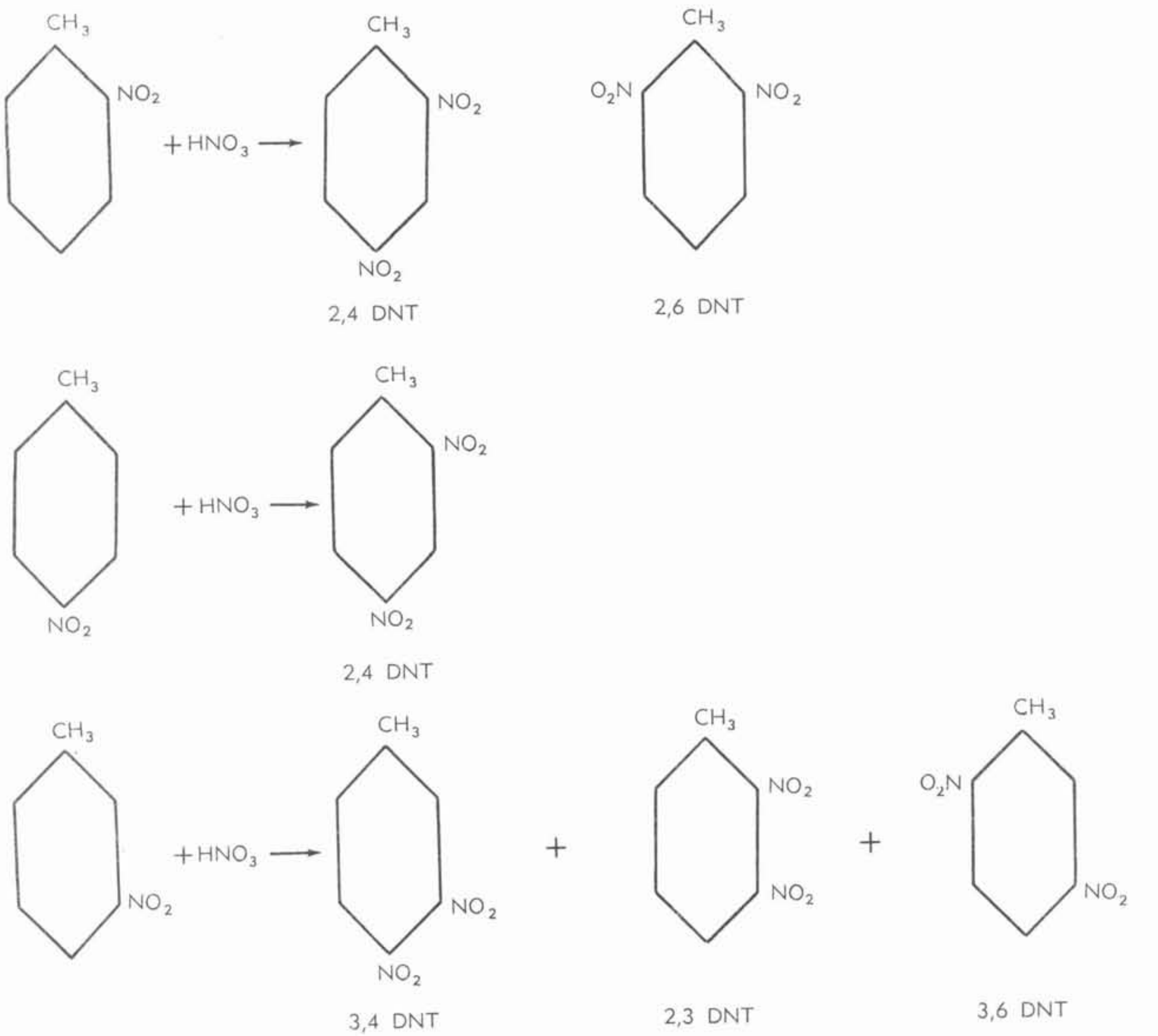


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Figure 1. DNT.

This mixture of dinitrotoluenes contains approximately 74.8 percent of the 2,4 isomer, 20.7 percent of the 2,6 isomer, 2.5 percent of the 3,4 isomer, 1.1 percent of the 2,3 isomer, and 0.9 percent of the 3,6 isomer. The mixture of these products is an oily liquid at the temperatures at which it is separated from the spent acid and subjected to trinitration.

Trinitration involves production chiefly of the following trinitrotoluenes:

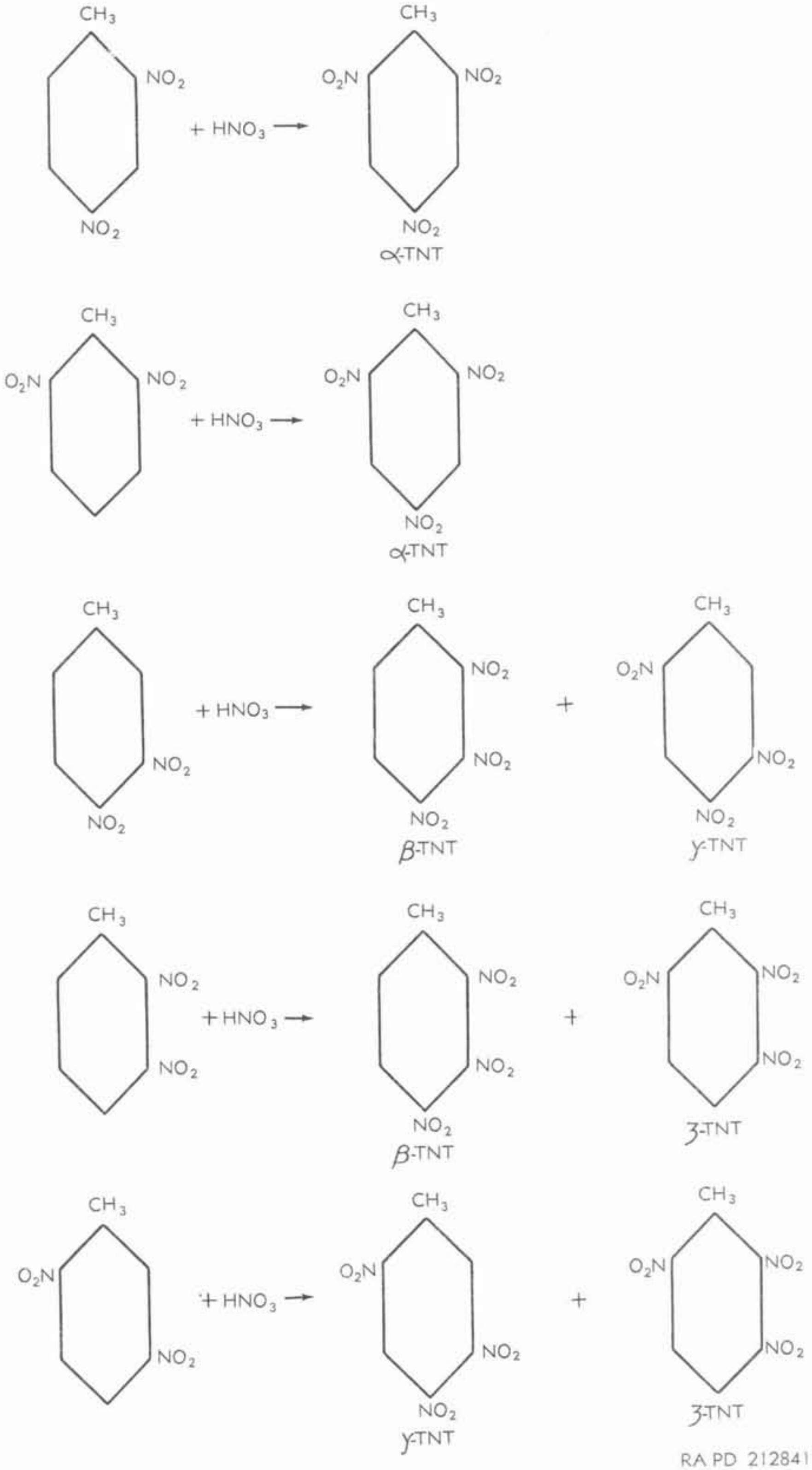

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Figure J. Trinitration products.

The resulting mixture consists of approximately 95.5 percent of α, 1.3 percent of β, 2.9 percent of γ and 0.3 percent of ζ trinitrotoluenes. In addition there may be formed traces of the other two isomeric TNTs, δ or 3,4,5, and ε or 2,3,5. Other impurities in crude TNT include 2,4,6-trinitrobenzoic acid, tetranitromethane, dinitrocresol, phenol, cyanic acid, and ammonium salts; and if trinitration is not complete, some dinitrotoluene is present. When free of acid, crude TNT has a freezing point of only 75° to 76° C.

j. In practice, the production of crude TNT by the direct nitration process is carried out as follows: To a nitrator in the "mono-house," there is transferred approximately 12,500 pounds of "mono-mix" acid prepared by butting up with weak nitric acid, the spent acid from the dinitration operation. The mono-mix acid contains approximately 48 percent sulfuric acid, 14 percent nitric acid, 17 percent nitrosylsulfuric acid, 19 percent water, and 2 percent nitrocompounds. After the acid has been cooled to 36° to 38° C. with agitation, 1,600 pounds of toluene is added, with agitation, under the surface of the acid, at such a rate that the temperature of the mixture increases 1° C. per minute. The temperature is about 55° C., when the addition of toluene is complete, and this is allowed to rise to 57° C., when stirring is decreased and the charge cooled to 38° C. Stirring is discontinued and the spent acid allowed to separate and settle. After drawing off the spent acid, the mono-oil" is transferred to a scale tank in another building. Spent acid from the trinitration operation is transferred to a nitrator in the bi-house, cooled to 77° C., and fortified with weak nitric acid, so that the nitrator contains about 12,500 pounds of a mixed acid containing 54 percent sulfuric acid, 13 percent nitric acid, 17 percent nitrosylsulfuric acid, 8 percent water, and 8 percent nitrocompounds. To this, with stirring, the mono-oil is added, at such a rate that the temperature of the mixture increases to 82° C., and then more slowly to 85° C. The charge is maintained at this temperature for 8 minutes and then cooled to 77° C. Stirring is discontinued and the spent acid is allowed to separate and settle. This is drawn off and the bi-oil is drained into a storage tank before transfer to the tri-house. Here there is placed in a nitrator approximately 8,500 pounds of tri-mix acid containing 82.7 percent total sulfuric acid and 23.3 percent nitric acid and generally at a temperature of approximately 85° C. This is cooled, with agitation, to 80° C., and the bi-oil is added at such a rate that the temperature of the mixture increases about 0.5° C. per minute to a maximum of 90° C. After the charge has been held at that temperature for a few minutes, increase at the rate of 1° C. per minute is permitted until the temperature reaches 110° C. After this temperature has been maintained for 20 minutes, it is decreased to 107° C., with continued agitation of the charge. Stirring then is discontinued and the "tri-oil" is allowed to separate and form a top layer while the temperature decreases to about 93° C. There is obtained approximately 3,500 pounds of tri-oil, which consists of molten crude TNT and is pumped to the "washhouse" for purification.

k. Purification of crude TNT is effected by the sellite process, which consists of washing with a solution of sodium sulfite and sodium hydrogen sulfite. While sodium sulfite reacts to a very slight extent with α-TNT, it is much more reactive with its isomers, in each of which there is present at least one nitro group in the *meta* or 3 position. With the reactive meta groups, the sulfite reacts to form the sodium salt of a sulfonic acid derivative:

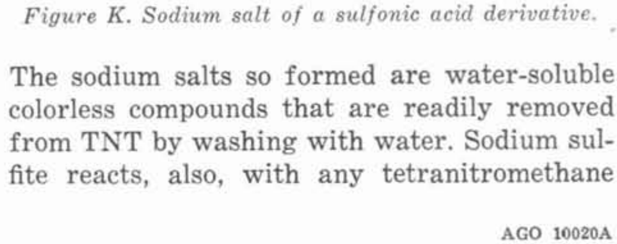


Figure K. Sodium salt of a sulfonic acid derivative.

The sodium salts so formed are water-soluble colorless compounds that are readily removed from TNT by washing with water. Sodium sulfite reacts, also, with any tetranitromethane present in crude TNT to form sodium trinitromethane present in crude TNT to form sodium trinitromethane sulfonate, which also is soluble in water and easily removed:

$$C(NO_2)_4 + Na_2SO_3 \rightarrow C(NO_2)_3.SO_2.ONa + NaNO_2$$

When the process is applied to crude TNT, the resulting solution develops a red color. Sodium sulfite does not react with any dinitrotoluene or trinitrobenzoic acid present. The sodium hydrogen sulfite also used takes no part in the purification reactions. It neutralizes any free acid present and is used for its buffering action.

l. In practice, the sellite purification process is carried out in the tank of the wash-house by agitating the tri-oil with a small amount of hot water for a few minutes to dissolve most of the free acid present. Sufficient cold water then is added to the mixture, with continued agitation, to reduce the temperature to 68° C. and cause crystallization of the TNT. Enough sodium carbonate is added to the slurry to neutralize the free acid present, but care is taken not to add an excess, as this would decompose some TNT. After neutralization, there is added to the agitated slurry a calculated quantity of a solution containing 16 to 17 percent sodium sulfite and 0.1 to 0.3 percent sodium hydrogen sulfite. The sulfite solution is added slowly at first and then more rapidly, the total time being about 15 minutes. During this addition, the color of the liquid in the slurry becomes a deep red. Agitation is continued for 15 minutes more, and the slurry then is transferred to wringers where the red water is separated. The crystalline TNT is washed with cold water to remove any adhering red water and then is transferred to a melt tank where it is melted, washed well with hot water, and then dried by agitation with dry air at 100° C. or more. The dry molten TNT then is cooled and flaked by means of a rotating flaker drum or crystallized in a grainer. The TNT obtained by the sellite purification process generally has a freezing point of 80.3 ±0.1° C. but, by increasing the duration of the sulfite treatment, a product having a freezing point as great as 80.6° C. can be obtained. The yield of purified TNT is approximately 80 percent of that theoretically obtainable from toluene.

m. There are two grades of TNT used for military purposes. Grade I is used for the loading of shell and bombs and the manufacture of demolition blocks and binary explosives. Grade III is used in priming compositions or special compositions that require either high purity or fine crystalline form. The requirements applying to the two grades are as follows:

	Grade I	*Grade III*
Form	Flake or crystalline.	Crystalline
Color	Light yellow through buff.	Light yellow
Solidification point, ° C., minimum.	80.2	80.4
Moisture, percent, maximum.	0.10	0.10
Acidity, percent, maximum.	0.01	0.01
Alkalinity	None	None
Benzene-insoluble, percent, maximum.	0.05	0.05
Sodium, percent, maximum.	0.001	0.001
Thickness of flake, inch, maximum:		
Average	0.025	
Individual	0.04	
Granulation:		
Through No. 14 sieve, percent, minimum.	95	100
Through No. 100 sieve, percent, minimum.		95

The solidification or freezing point of the material indicates the extent to which it has been freed of impurities, such as isomeric TNTs and DNT, while the benzene-insoluble requirement controls the presence of metallic salts, rust, etc. The sodium requirement is intended to prevent the use of excessive sodium carbonate for neutralizing acidity of the tri-oil and incomplete washing of TNT after treatment with sodium sulfite.

n. TNT is one of the least sensitive of the military high explosives, only ammonium picrate, nitroguanidine, and ammonium nitrate being less sensitive. It has high impact test values, is unaffected in the pendulum friction test, explodes in only 2 percent of the trials when subjected to the rifle bullet impact test, has a very high explosion temperature test value (475° C.), and has high minimum detonating charge values for initiation by lead azide and mercury fulminate. It can be detonated by a No. 6 electric blasting cap but the presence of only 7 percent of moisture prevents detonation; whereas 14 and 35 percent moisture is required to cause the desensitization of RDX and PETN, respectively. The fine dust of TNT is sensitive to electric sparks. When ignited in free air, it burns completely without detonation; and the temperature of the liquid phase must be 510° C. if detonation is to result from burning. Even when it is under a gas pressure of 100 psi, the liquid phase temperature must be greater than 285° C., if TNT is to burn with subsequent explosion. In the manufacture of TNT, small amounts of finely divided, condensed TNT may be deposited in ventilating pipes, etc. This is slightly more sensitive to impact than ordinary TNT but is less sensitive than tetryl, having impact test values of 10 inches and 40 centimeters with a 2-kilogram weight. The presence of rust increases the sensitivity of TNT to impact, a mixture of equal weights having an impact test value of only 30 centimeters; but the mixture is no more sensitive to friction than TNT, as judged by the pendulum friction test. Liquid TNT is very much more sensitive to impact than solid TNT. At 80°, 90°, and 105° to 110° C., liquid TNT has impact test values of only 7, 3, and 2 inches, respectively, as compared with 14 inches for solid TNT at 20° C. In rifle bullet impact tests with no air space above the explosive, high order detonations result in 70 percent of the trials with liquid TNT at 105° to 110° C., as compared with partial explosions in 10 percent of the trials with solid TNT at 20° C. While the degree of confinement is quite critical in its effect on the sensitivity of liquid TNT to impact, it is of the same order of sensitivity as mercury fulminate. Liquid TNT is, apparently, no more sensitive to friction than solid TNT, as judged by pendulum friction tests with the base block heated to 130° C., but liquid TNT is considerably more sensitive to explosion by influence than solid TNT. In comparative tests with an air gap of 5 inches, explosion to molten TNT was transmitted in 80 percent of the trials as compared with no explosions of flaked TNT having an apparent density of 0.77. These tests also showed the flaked TNT, crystalline TNT of apparent density 0.91, and cast TNT of density 1.55 to be of decreasing sensitivity in that order. Summarizing, it may be said that solid TNT is a relatively safe material to handle but molten TNT may be an extremely hazardous material under certain conditions; and several accidental explosions in melting equipment have been attributed to the liquid TNT being subjected to shock while under some confinement.

o. TNT has the same brisance as picric acid and guncotton and is superior to explosive D and nitroguanidine in this respect, as judged by the sand test. It is distinctly less brisant than tetryl, haleite, PETN, RDX, or nitroglycerin. Fragmentation tests show TNT to be about equal to picric acid, more brisant than explosive D, and inferior in this respect to tetryl, haleite, PETN, and RDX. As TNT has a lower rate of detonation (6,900 meters per second) than any of the other nine explosives mentioned, it is evident that rate of detonation alone does not determine the brisance of an explosive. As judged by ballistic pendulum and Trauzl lead block tests, TNT is more powerful than explosive D but less so than picric acid, guncotton, tetryl, haleite, PETN, RDX, or nitroglycerin; and TNT has a lower heat of explosion than any of these explosives except explosive D. These comparisons do not indicate TNT to be a weak explosive, as the data indicate TNT to be approximately 80 percent as brisant and 65 percent as powerful as PETN and RDX, the most brisant and powerful of the solid high explosives used for military purposes.

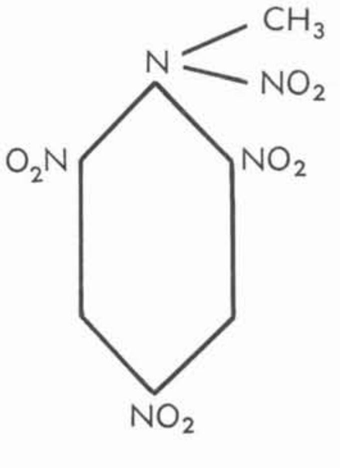
p. The chemical stability of TNT is such that even at 150° C. it undergoes no great decomposition in 40 hours. Molten TNT can be stored at 85° C. for 2 years without any decrease in purity. TNT has been found to withstand storage at magazine temperatures for 20 years without any measurable deterioration. The physical stability of TNT, however, has caused considerable trouble in the past because of the relatively low melting point of α-TNT, the presence of the β and γ isomers and DNT as impurities, and the formation of eutectic mixtures having melting points even lower than the of α-TNT. When loaded in shell and stored at elevated temperatures, the liquified eutectic mixture flows toward the nose of the shell and exudes as a dark oily liquid that is less sensitive than TNT but represents a fire hazard. The exudation of shell causes cavitation of the charge and in some cases desensitization of the booster charge. It has been found that TNT must have a minimum freezing point value of 80.2° C., if exudation is to be avoided in storage at 50° C. (122° F.); but such TNT exudes at 65° C. (150° F.). If exudation at 65° C. is to be prevented, the freezing point of TNT must be greater than 80.4° C. Even TNT having a freezing point of 80.70° undergoes very slight exudation if the storage temperature is 71° C. (160° F.). Binary explosives having TNT as an ingredient also tend to exude at elevated temperatures; but this tendency is less than that of TNT if the other ingredient—such as ammonium nitrate—does not form an eutectic mixture with TNT. When an eutectic mixture is formed—as with PETN—the tendency to exude is increased. While the problem of exudation is not as serious as immediately after World War I, this characteristic of TNT is decidedly disadvantageous.

q. Moisture has no effect on the stability of TNT, which is unaffected by immersion in sea water. However, at 50° C. ethanol reacts with α or γ but not β-TNT. In the case of α-TNT the products are ethyl nitrite and an unidentified nitrocompound that melts at 77.5° to 77.9° C. and is soluble in exudate oil. This was another contributing cause to the exudation of TNT charges in the period following World War I, as ethanol was used to clean the threads in shell noses and an ethanol solution of shellac was used to coat the walls of booster cavities in TNT shell charges. TNT undergoes slight reaction with iron rust as indicated by 120° C. vacuum stability tests of mixtures. When a mixture of TNT, iron, and water is in contact with either hydrochloric acid or trichloroethylene, there is formed a dark brown solid that is nonexplosive but relatively unstable. Potassium nitrite has been shown to be incompatible with TNT, and this effect of nitrite is the basis for the requirement that ammonium nitrate used for manufacturing amatol be free of nitrites. TNT is compatible with nitrates, potassium chlorate, black powder, and coatings such as bakelite, acidproof black paint, and shellac. It causes slight or very slight corrosion of lead, but has no effect on copper, tin, iron, steel, zinc, cadmium, nickel, brass, or bronze.

r. TNT is somewhat toxic, but not extremely so and has no vesicant or lacrimatory action. A maximum concentration in air of 1.5 milligrams per cubic meter has been suggested to avoid undue toxicity.

7-11. Tetryl

a. 2, 4, 6-Trinitrophenylmethylnitramine, tetryl, tetralite, pyronite, or CE is a nitroaryl nitramine having the structure.



RA PD 212843

Figure L. CE.

and a molecular weight of 287.15. As ordinarily manufactured, it has a light yellow or buff color and consists chiefly of aggregates of crystals (figs. 7-19 and 7-20). When freshly prepared and highly purified, it is colorless but rapidly acquires a yellow color when exposed to light. When crystallized from benzene, it forms monoclinic prisms. Tetryl has been used chiefly as a booster explosive, as an ingredient of a binary explosive, and in the loading of detonators and blasting caps. It was first prepared by Mertens in 1877, but was not used as an explosive until World War I. In the early literature, it was designated as tetranitromethylaniline.

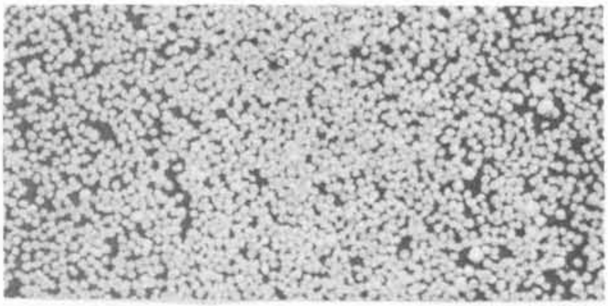

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Figure 7-19. Tetryl crystals, 1½ x.

b. Pure tetryl melts at 129.45° C. with partial decomposition, and repeated solidification and melting rapidly increase the degree of decomposition. Tetryl has a scratch hardness of less than one on the Mohs' scale and therefore the crystals are slightly softer than talc. With a crystal density of 1.73, tetryl has a density of 1.62 when cast. When subjected to pressures of 3,000, 5,000, 10,000, 20,000, and 30,000 psi, it has density values of 1.40, 1.47, 1.57, 1.67, and 1.71, respectively. It has specific heat values of 0.213, 0.217, 0.223, and 0.231 calorie per gram per degree Centigrade at 0°, 20°, 50°, and 100° C., respectively. Tetryl has a heat of fusion value of 20.6 calories per gram. Its heat of combustion at constant pressure is 2,914 calories per gram, and from this is derived a heat of formation value of −5.02 kilogram-calories per mole. The solubility characteristics of tetryl are shown by table 7-18.

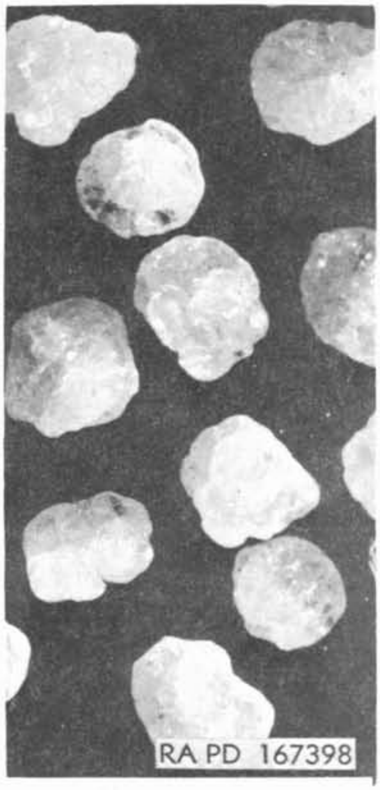
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Figure 7-20. Tetryl crystals, 25 x.

Table 7-18. Solubility Characteristics of Tetryl.

° C.	Grams per 100 grams of solvent at—					
	0	20	50	60	75	80
Water	0.0005	0.0075	0.0195	0.035	0.066	0.081
Ethanol*	0.320	0.563	1.72	2.64	5.33	
Carbon tetrachloride	0.007	0.025	0.095	0.154	0.297	
Chloroform	0.225	0.57	1.78	2.65		
Ethylene chloride	1.5	3.8	12.0	18.8	45	64.5
Carbon disulfide	0.009	0.021				
Ether	0.188	0.418				

*95 percent by volume.

At 100° C., water dissolves tetryl to the extent of 0.184 grams per 100 grams. Reflecting its sparing solubility in water, tetryl has been found to be hygroscopic to the extent of only 0.04 percent when exposed to an atmosphere of 90 percent relative humidity at 30° C. Tetryl is very soluble in acetone. Benzene holds more tetryl in solution if a solution with solid tetryl present is cooled to a given temperature than if a mixture of benzene and excess tetryl is heated to and maintained at that temperature. At 15 , 25°, 35°, 45°, and 55° C., tetryl has respective supersolubility values of 10.2, 12.2, 14.9, 18.25, and 22.5 and subsolubility values of only 3.9, 5.5, 7.4, 9.7, and 13.25 grams per 100 grams of benzene. The spent acid from the production of tetryl contains only 0.3 percent of tetryl.

c. Tetryl reacts with an aqueous alkali to yield the metallic picrate and nitrite and methylamine. Although prolonged boiling with dilute sulfuric acid has no effect, if a solution of tetryl in cold concentrated sulfuric is allowed to stand at ordinary temperature nitric acid and 2,4,6-trinitrophenylmethylamine or *N*-methyl picramide, $C_6H_2(NO_2)_3.NH.CH_3$, are formed. If this reaction is carried out in a nitrometer in the presence of mercury, the nitric acid is reduced to nitric oxide and this can be measured. In benzene solution at ordinary temperatures, tetryl reacts with aniline to form 2,4,6-trinitrodiphenylamine and methylnitramine ($CH_3.NH.NO_2$). Aqueous sodium sulfide decomposes tetryl completely into nonexplosive, water-soluble products. Tetryl is decomposed into nonexplosive products by aqueous sodium sulfite, but the reaction is relatively slow unless the solution is heated to 80–90° C.

d. Two methods have been used extensively for the production of tetryl, the raw materials for nitration being dimethylaniline and dinitrophenylmethylamine. The use of monomethylaniline has not been practical, because of nonavailability of this material in a sufficiently pure form, on a large scale and at low cost. The use of dimethylaniline has the disadvantage of requiring nitric acid for the removal of one methyl group by oxidation:

$$C_6H_5N(CH_3)_2+10HNO_3\rightarrow(NO_2)_3C_6H_2N(NO_2)CH_3+6NO_2+CO_2+8H_2O$$

With the development of methylamine as a cheap commercial bulk chemical, there became practicable the production of dinitrophenylmethylamine from 2,4- or 2,6-dinitrochlorobenzene:

$$C_6H_3(NO_2)_2Cl+CH_3NH_2\rightarrow HCl+C_6H_3(NO_2)_2.NH.CH_3$$

This can be nitrated to tetryl with relative ease and no use of nitric acid for oxidation. A considerable amount of tetryl was prepared by this method in the United States during World War II.

e. In the manufacture of tetryl from dimethylaniline, one part of this is dissolved in 14.4 parts of 96–99 percent sulfuric acid at 20–30° C. Dimethylaniline sulfate is formed. The solution of this is added in a nitrator to 9.2 parts of mixed acid at 68–72° C. which contains 66.7 percent nitric acid and 15.8 percent sulfuric acid. If the water content of the mixed acid is too high, there are formed benzene-insoluble impurities, which are benzidine derivatives. During the nitration there is formed some 2,3,4,6-tetranitrophenylmethylnitramine (*m*-nitrotetryl), which is relatively unstable but can be hydrolyzed to 2,4,6-trinitro-3-hydroxyphenylmethylnitramine. This is soluble in water and readily removed. Tetryl must be purified carefully, so as to remove any tetranitrocompound and occluded acidity, because of the adverse effects of these on the stability of tetryl. Most of the acidity is removed by washing with cold water. Treatment with boiling water decomposes any tetranitrocompound present and removes its product. If benzene is used as a solvent, the crude tetryl is dissolved in this, the solution is washed with water to remove residual acidity, and filtered to remove insoluble matter. The tetryl is deposited on cooling the solution, separated, and dried. If acetone is used as the solvent, the solution is neutralized and filtered and the tetryl is precipitated by mixing the solution with water. The acetone purification process is carried out on a continuous basis, while that with benzene is operated as a batch process.

f. Two grades of material are covered by the specification for military tetryl, grade I being material intended for use in detonators, boosters, projectiles, and the manufacture of tetrytol. Grade I comprises two classes, with respect to granulation. Grade II tetryl is that reclaimed from manufacturing and loading operations and intended for use in the manufacture of tetrytol only. The requirements for the two grades are as follows:

Color: Light yellow.
Moisture and volatiles: Maximum, 0.10 percent.
Insoluble matter: Maximum, 0.10 percent insoluble in benzene.
Grit: None.
Acidity: Maximum, 0.02 percent as sulfuric acid.
Melting point: Minimum, 128.8° C.; maximum, 129.5° C.

	Grade I *Class A*	 *Class B*	*Grade II*
Bulk density: Minimum, g/ml.	------	0.82	
Granulation: Percent through sieve:			
No. 8, minimum	------	------	100
No. 12, minimum	100		
No. 16, minimum	95		
No. 30, minimum	------	100	
No. 40, maximum	------	10	
No. 60, maximum	25	2	
No. 100, maximum	5		

The requirement, with respect to benzene-insoluble matter, is included because of the marked desensitizing effect of such impurities. Freedom from grit is required, because of its effect on sensitivity to impact and friction. The bulk density and granulation requirements for grade I, class *B* tetryl are intended to control the ease with which it pours and consequent action in automatic pelleting machines.

g. Prior to World War II and the standardization of PETN and RDX, tetryl was the most sensitive of the standard noninitiating high explosives. Is greater sensitivity than TNT is reflected by the results of impact and rifle bullet impact tests of the two explosives. That this greater sensitivity does not represent undue hazard in handling is shown by the failure of tetryl to explode in the pendulum friction test. The explosion temperature test value of tetryl (257° C.) also is much lower than that of TNT. The greater sensitivity of tetryl to initiation was one of the main reasons for its substitution for pressed TNT as a booster explosive. This greater sensitivity is shown by minimum detonating charge values of 0.10 gram lead azide and 0.19 gram mercury fulminate as determined by the sand test. It is to be noted that the sensitivity of tetryl to shock and friction has not been found sufficient to necessitate packing in a wet condition, as is required for PETN and RDX.

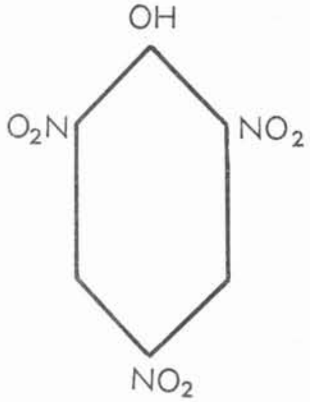
h. Sand test data indicate tetryl to be 113 percent as brisant as TNT, and the rates of detonation of the two explosives bear out this relationship. However, fragmentation tests indicate tetryl to be 121 percent as brisant as TNT. The greater brisance and rate of detonation of tetryl explain its greater boostering action. The standardization of PETN and RDX during World War II has caused some replacement of tetryl by these two even more brisant explosives. Trauzl lead block and ballistic pendulum tests indicate tetryl to be 128 percent as powerful as TNT, and the heat of explosion of tetryl is 121 percent that of TNT. Tetryl is approximately 90 percent as brisant and 82 percent as powerful as PETN or RDX.

i. Vacuum stability tests show tetryl to be less stable than TNT and RDX but to be of excellent stability at temperatures of 120° C. or less. It has been found to withstand storage at 100° C. for 100 hours, at 75° C. for 6 months, at 65° C. for 12 months, and at magazine temperatures for 20 years without significant impairment of its stability. Because its melting point is so much higher than any probable storage temperature and the almost complete absence of impurities that could form eutectic mixtures having low melting points, the physical deterioration of tetryl is not a problem. Tetryl frequently is used with 1 to 2 percent of a binding agent or lubricant, such as graphite, stearic acid, or magnesium stearate; and it has been found to be compatible with such materials as well as black powder. In the presence of 0.5 percent of moisture, tetryl has some corrosion effect on steel and zinc but has little or no effect on copper, tin, lead, nickel, cadmium, aluminum, brass, or bronze. It does not affect coatings of acidproof black paint, baked oil, NRC compound, or shellac, even when stored at 50° C.

j. Tetryl has a strong coloring action on the human skin and can cause a dermatitis. The use of a cold cream containing 10 percent sodium perborate has been found to minimize these effects. Inhalation of tetryl dust has recognized toxic effects; and the suggested permissible maximum concentration of the dust in air is 1.5 milligrams per cubic meter.

7-12. Picric Acid

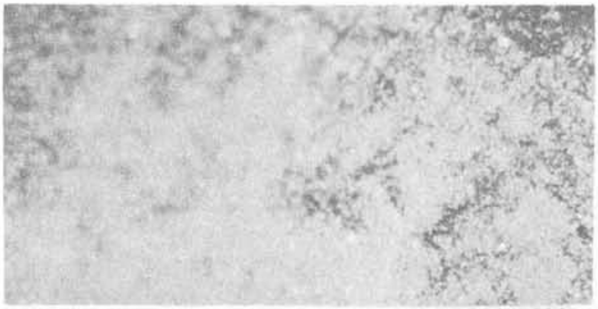
a. 2,4,6-Trinitrophenol, picric acid, melinite, lyddite, pertite, or shimose is a compound having the structure



RA PD 212844

Figure M. Shimose.

and a molecular weight of 229.108. As manufactured, it consists of light to bright yellow crystals, which exist in two polymorphic forms (figs. 7-21 and 7-22). When crystallized from ethanol, it forms yellow, orthorhombic, flattened rods. First prepared in 1771 by Woulff, it was first used as a yellow dye. It was not until 1885 that the French began using it as a military explosive in shell, and other nations soon adopted it. Picric acid was the first high explosive used for melt-loading and its use was nearly general until the beginning of this century, when it was gradually replaced by TNT. The high temperature required for its melt-loading (120° C.) and the occurrence of disastrous explosions attributed to the formation of sensitive metallic compounds rendered is replacement desirable. Picric acid is used in the United States chiefly for the manufacture of explosive "D".



RA PD 167399

Figure 7-21. Picric acid crystals, 1½ x.

b. Pure picric acid melts at 122.5° C. without decomposition and its freezing point can be used for determination of its purity. Material used for military purposes may solidify at as low a temperature as 120.0° C., because of the presence of isomers and dinitrophenol. Picric acid has vapor pressure values of 2 and 50 mm at 195° and 255° C., respectively. At ordinary temperatures, it is of very low volatility but, because of its bitter taste and dustiness, it has the sensory effects of a volatile material. With a crystal density of 1.763 and a density of 1.71 when cast, picric acid can be compressed to density values of 1.49, 1.57, 1.64, and 1.725, when subjected to pressures of 5,000, 10,000, 20,000, and 100,000 psi, respectively. At 0°, 50°, 100°, and 120° C. picric acid has specific heat values of 0.234, 0.274, 0.318, and 0.337 calorie per gram per degree Centigrade, respectively, and its heat of fusion is 20.4 calories per gram. The heat of combustion of picric acid at constant pressure is 2,671.5 calories per gram, and from this is derived a heat of formation value of 56.83 kilogram-calories per mole. Although soluble in water to the extent of 1.4 and 6.8 grams per 100 grams at 20° and 100° C., respectively, picric acid is hygroscopic to the extent of only 0.04 percent, when exposed to an atmosphere of 90 percent relative humidity at 30° C. Picric acid is easily soluble in benzene, sulfuric acid, or nitric acid, but is soluble in ether and ethanol at 20° C. to the extent of only 2.0 and 6.2 grams, respectively, per 100 grams of solvent.

c. Being a phenolic derivative, picric acid is chemically active, although less so than phenol. It decomposes carbonates and reacts with hydroxides to form picrates:

$$C_6H_2(NO_2)_3OH + NaOH \rightarrow H_2O + C_6H_2(NO_2)_3ONa$$

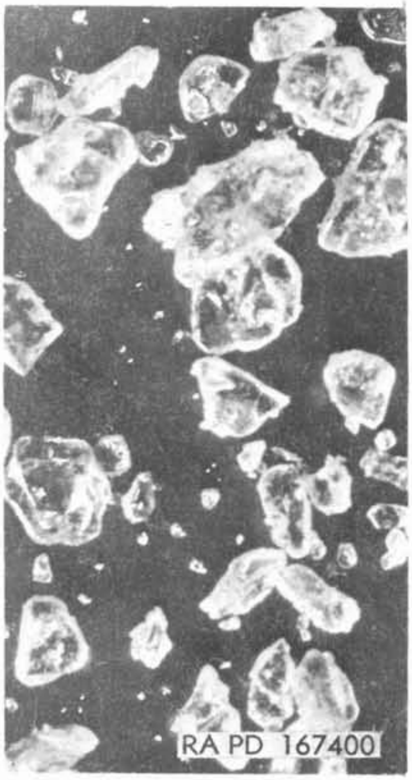
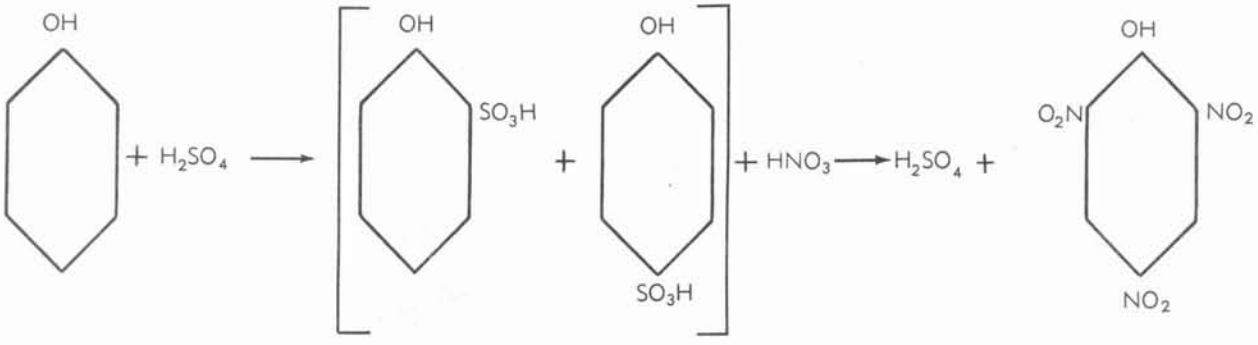


Figure 7-22. Picric acid crystals, 25 x.

It may be titrated with bases by the use of sodium alizarinsulfonate as an indicator. Addition of a solution of copper ammonium sulfate to an aqueous solution containing as little as 0.01 percent of picric acid results in the formation of a green precipitate. Picric acid forms compounds with amines and even aromatic hydrocarbons, such as naphthalene. Solutions of acridine, cinchonine, and nitron in ethanol yield precipitates with picric acid. Aqueous solutions of neutral lead acetate and picric acid yield no precipitate, but if the acetate is basic or ammoniacal, a bright yellow precipitate of lead picrate is formed. This is more sensitive to impact than mercury fulminate although less sensitive to friction. The picrates of lead and zinc can be formed by the contact of molten picric acid with the metal and can initiate detonation of the picric acid. Other metallic picrates are readily formed and generally contain water of crystallization. When dehydrated, these picrates are more sensitive to impact; but they are not as sensitive as lead picrate. The picrates of iron, nickel, zinc, copper, etc., have been considered dangerously sensitive and their formation has been advanced to explain accidental explosions of picric acid. As dry picric acid has little if any effect on the ordinary metals and the hydrated picrates must be heated to 80° to 150° C. to undergo dehydration, their importance—except in the case of lead picrate—probably is not as great as it was previously considered to be. Anhydrous ferric picrate is as sensitive as PETN and nickel picrate is slightly more sensitive, but the anhydrous picrates of cadmium, zinc, and copper are nearly as insensitive as TNT. The anhydrous picrates of sodium and potassium are less sensitive than TNT.

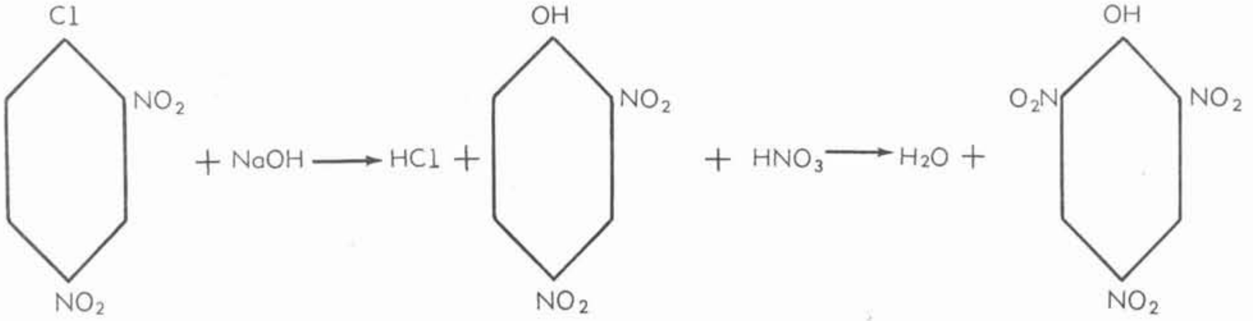
d. Because of the violence of the reaction and consequent low yields, the direct nitration of phenol for the production of picric acid is not practicable. However, phenol and sulfuric acid react to form *ortho-* and *para-*phenol sulfonic acids which can be nitrated to picric acid:



RA PD 212845

Figure N. Nitration of phenol.

The nitration can be carried out in one, two, or three stages and various process have been used. The best yield is claimed to be 220 parts of picric acid from 100 parts of phenol. Another process involves the conversion of dinitrochlorbenzene into dinitrophenol and nitration of this:



RA PD 212846

Figure O. Nitration of dinitrophenol.

A catalytic process for the production of picric acid from benzene also has been used. This involves the refluxing of one part of benzene with four and one-quarter parts of nitric acid of specific gravity 1.42 in which one-twentieth part of mercuric nitrate has been dissolved. On distillation of the refluxed mixture with steam there are removed benzene, nitrobenzene, dinitrobenzene, and dinitrophenol. On filtering and cooling the residue, picric acid crystallizes out with a yield comparable with that obtained from benzene if it is sulfonated and converted to phenol, with sulfonation and nitration of this to picric acid. Purification of crude picric acid can be effected by washing with cold water and recrystallizing from hot water or benzene.

e. But one grade of picric acid is prescribed for military use, this complying with the following requirements:

Color: White to yellow.
Solidification point: Minimum, 120.0° C.
Moisture: Maximum, 0.20 percent.
Acidity: Nitric acid, none. Total sulfuric acid, maximum, 0.1 percent.
Insoluble matter: Maximum, 0.1 percent.
Ash: Maximum, 0.1 percent.
Lead: Maximum, 0.0004 percent.
Granulation: Minimum through No. 14 sieve, 99.5 percent.

The requirement, with respect to lead content, reflects the recognized hazard represented by lead picrate and the necessity for preventing its formation from impurities in raw materials or by the contact of picric acid with lead in equipment during manufacture. The requirement that picric acid be free of nitric acid is necessitated by the adverse effect of nitric acid on the stability of picric acid during storage.

f. Picric acid is only slightly more sensitive than TNT, as indicated by impact tests, but distinctly more so as shown by the rifle bullet impact test. Minimum detonating charges of lead azide and mercury fulminate indicate picric acid to be of the same order of sensitivity to initiation as TNT. The explosion temperature test value of picric acid (322° C.) is lower than that of TNT but somewhat higher than that of tetryl (257° C.). The explosion temperature test values of the three explosives are in the reverse order to their relative sensitivity as shown by the rifle bullet impact test. Picric acid of satisfactory purity, therefore, cannot be considered unduly hazardous with respect to sensitivity; and the accidental explosions during its early history must be attributed to other causes.

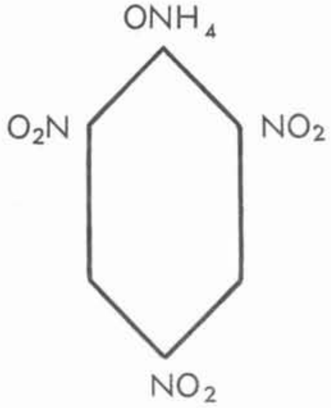
g. From their chemical compositions and structures, picric acid and TNT should be of the same order of brisance and power, with picric acid having any superiority because of its slightly better oxygen balance. Sand test values indicate equal brisance, but the plate dent test shows picric acid to be slightly superior to TNT. The rate of detonation of picric acid (7,350 meters per second) is distinctly greater than that of TNT, but this is reflected more by their relative power values than by brisance values. Both the ballistic pendulum and Trauzl lead block tests show picric acid to be 103 to 109 percent as powerful as TNT. It is to be noted that the heat of explosion of picric acid is 108 percent that of TNT.

h. Picric acid is of a high order of stability, as shown by stability and storage tests, it having approximately the stability of TNT. The values for the two explosives obtained by the 75° C. international, 100° C. heat, and 100° and 120° C. vacuum stability tests are essentially equal. Long term storage tests at magazine temperatures have shown neither to undergo measurable deterioration.

i. Picric acid has a strong staining action on the human skin, but is not as toxic as some of the comparable nitrocompounds. Nevertheless, its dust should not be inhaled, and frequent baths and changes of clothes are prescribed for workers in the production and loading of picric acid.

7-13. Explosive D

a. Ammonium 2,4,6-trinitrophenolate, ammonium picrate, or explosive D is a colored crystalline compound having the structure



RA PD 212847

Figure P. Explosive D.

and a molecular weight of 246.14. It exists as yellow monoclinic crystals (figs. 7-23 and 7-24) representing a stable form and red rhombic crystals representing a metastable form. The two forms are easily interconvertible and do not differ in explosive properties. As manufactured for military use, explosive D is yellow to orange in color. Marchand first prepared the compound in 1841 and in 1869 it was used by Brugere in admixture with potassium nitrate as a military propellant; but it was not until after 1900 that it was used as a detonating high explosive. Its insensitivity to shock makes it suitable for use in armor-piercing projectiles and it is used also as an ingredient of picratol and propellent compositions.

b. Pure explosive D melts with decomposition at 265° to 271° C. It has a crystal density

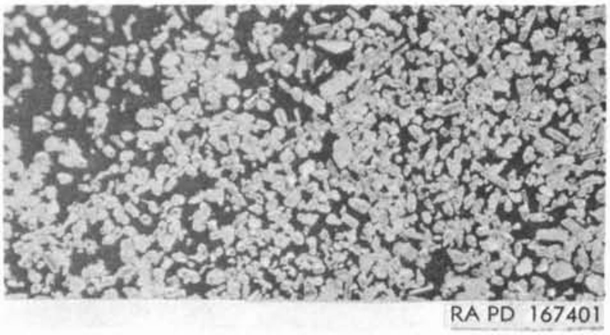


Figure 7-23. Explosive D crystals, 1½ x.

of 1.719. When subjected to pressures of 5,000, 10,000, 20,000, 50,000, and 100,000 psi, it has density values of 1.41, 1.47, 1.53, 1.59, and 1.64 respectively. The heat of combustion of explosive D at constant pressure is 2,745 calories per gram, from which is derived a heat of formation value of 95.82 kilogram-calories per mole. Although 100 grams of water dissolve 1.1 and 74.8 grams of explosive D at 20° and 101° C., respectively, the explosive is nonhygroscopic when exposed to humid air. It is soluble in acetone to the extent of 2.85 grams per 100 grams at 30° C. At 20° and 80° C., explosive D is soluble in ethanol to the extent of 0.86 and 3.75 grams, respectively, per 100 grams and in ethyl acetate to the extent of 0.34 and 0.86 gram per 100 grams, respectively. Its solution in ethanol is very slow and it separates from the solution only slowly on standing. It is practically insoluble in ether. 100 grams of octyl alcohol at 25° C. dissolve less than 0.2 gram of explosive D.

c. Chemically, explosive D is not very reactive. Strong alkalies decompose it into picric acid and ammonia, and this is the basis of one of the methods for determining the purity of the material. When maintained at its melting point, it decomposes into the same products. At 0° C., explosive D absorbs an equimolecular amount of ammonia; but at 26° C., this is lost by volatilization.

d. The manufacture of explosive D is relatively simple. Picric acid is suspended in hot water, in which it is only partially soluble, and neutralized by the addition of gaseous or aqueous ammonia. As the picrate is formed, it goes into solution. The reaction is slightly exothermic. On cooling the solution, the picrate separates out. If a marked excess of ammonia is used, the red form is obtained; so care is taken to avoid this and obtain the yellow form. The yellow form has a higher bulk density than the red and can be pressed better. The separated crystals are washed with cold water to remove any free ammonia present and then dried.

Figure 7-24. Explosive D crystals, 25 x.

e. The specification for explosive D covers one grade of material, representing two classes with respect to granulation. Class *A* material is intended for use in the press-loading of shell, while class *B* is used for the manufacture of picratol and other compositions. The requirements are as follows:

Color: Yellow to orange.
Water-insoluble matter: Maximum, 0.10 percent.
Ash: Maximum, 0.1 percent.
Acidity or alkalinity: Maximum, 0.025 percent (as pic-

ric acid or ammonia).

Ammoniacal nitrogen: Minimum, 5.60 percent; maximum 5.75 percent.

Sulfates: Maximum, 0.1 percent (calculated as sulfuric acid).

Chloroform-soluble matter: Class *A*, maximum, 0.1 percent.

Irritant contaminants: Maximum, 50 parts of chlorine per million.

Moisture: Maximum, 0.10 percent for Class *A* and 0.20 percent for Class *B*.

Visible foreign matter: Class *B*, none.

Granulation:

	Class A	*Class B*
Percent through No. 12 sieve, minimum.	99.9	
Percent through No. 40 sieve, minimum.	------	99.5
Percent retained on No. 70 sieve, minimum.	60	
Percent retained on No. 70 sieve, maximum.	------	20
Percent through No. 200 sieve, maximum.	5	

Bulk density: Class *A*, minimum, 0.82 gram per milliliter.

The color requirement is intended to cover the unavoidable presence of a small amount of the red form of explosive D in admixture with the yellow form. The requirement, with respect to irritant contaminants, represents a control of the purity of picric acid used in manufacture when this is made by the dinitrochlorbenzene process. The chloroform-soluble matter requirement also represents a control of the nature of impurities present in picric acid manufactured by a process other than the nitration of phenol.

f. Explosive D is distinctly less sensitive to impact than TNT, and its slightly greater sensitivity in the rifle bullet impact test is attributable to the much lower temperature required for explosion (318° C.) than that for TNT (475° C.). The low degree of sensitivity of explosive D to impact is paralleled by its relative insensitivity to initiation. In the sand test, it is not detonated by either lead azide or mercury fulminate, a booster charge of 0.06 gram of tetryl being required. In this test, a minimum charge of 0.28 gram of diazodinitrophenol detonates explosive D, but a charge of only 0.15 gram is required for the detonation of TNT. Moisture has a marked effect on the sensitivity of explosive D to initiation. The presence of 0.5, 1.0, and 2.0 percent of moisture causes increase in the minimum detonating charge of tetryl to 0.09, 0.11, and 0.14 gram, respectively. Storage of either the yellow or red form of explosive D at 50° C. for 3 years causes such an increase in sensitivity that it can be initiated by mercury fulminate alone. Subsequent storage of the yellow form at magazine temperatures for 2 years causes desensitization to such an extent that a booster charge to tetryl is required for complete detonation and 4 years of such storage is required for return to the original condition of sensitivity. Storage at 50° C. has the same effect on the sensitivity of red explosive D, but subsequent storage at magazine temperatures for 4 years merely increases the minimum detonating charge of mercury fulminate from 0.23 to 0.29 gram. The changes in sensitivity are not accompanied by any change in brisance or in color.

g. The brisance of explosive D, as measured by the sand test, is only 77 percent of that of TNT, but fragmentation tests indicate it to be 91 and 99 percent as brisant when pressed to density values of 1.50 and 1.55, respectively. Although the heat of explosion of explosive D is but 86 percent that of TNT, the ballistic pendulum test shows it to be 98 percent as powerful as TNT. This combination of brisance and power, almost equal to those of TNT together with relative insensitivity, has made explosive D suitable for use in armor-piercing projectiles. However, the impracticability of melt-loading explosive D has led to its partial replacement by picratol, which was developed during World War II and can be melt-loaded.

h. 150° C. vacuum stability test data show explosive "D" to be at least as stable as TNT at that high temperature. Storage at 65° C. for

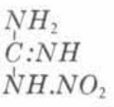
1 year causes no apparent deterioration. While storage at 50° C. for 5½ years causes some increase in sensitivity to initiation, it causes no measurable impairment of stability. Tests during storage at magazine temperatures over a period of 20 years have shown no change in the stability of explosive D. It is, therefore, considered to be of entirely satisfactory stability. At 100° C. it is compatible with TNT or black powder, but undergoes reaction with nitroglycerin, nitrocellulose, PETN, or tetryl.

i. Explosive D is not markedly toxic, but it discolors the skin and may cause a dermatitis in some cases. Inhalation of the dust should be minimized and frequent baths and changes of clothes are desirable for those working with the material in quantity.

j. Potassium picrate has been used to a limited extent in pyrotechnic and priming compositions. It forms rhombic crystals of reddish yellow or green color that have a density of 1.85. The crystals are anhydrous when formed from water, in which the compound is soluble to the extent of 0.5 gram per 100 grams at 15° C. and 25 grams at 100° C. It is soluble to the extent of only 0.2 gram per 100 grams of ethanol at 25° C. Potassium picrate is prepared by neutralizing a hot aqueous solution of potassium carbonate with a hot solution of picric acid and filtering off the crystals that separate when the solution cools. Its impact test value of 21 inches shows it to be less sensitive than explosive D, and it has a higher explosion temperature test value (370° C.) than that of explosive D. It is of the same order of stability as explosive D, but is less brisant. When subjected to contact with a flame, potassium picrate deflagrates with a loud report. Mixtures of potassium picrate with oxidizing agents, such as potassium nitrate, only burn when ignited, but are sensitive to shock.

7–14. Nitroguanidine

a. Nitroguanidine or picrite, a guanyl nitramine, is a colorless crystalline compound having the structure



$$\begin{array}{l} NH_2 \\ | \\ C{:}NH \\ | \\ NH.NO_2 \end{array}$$

and a molecular weight of 104.074. It exists in two forms having the same melting point. That usually produced during manufacture is the α-form, which consists of long, thin, flat needles that are tough and very difficult to pulverize (figs. 7–25 and 7–26). It has a double refraction value of 0.250. The β-form crystallizes from water in clusters of small, thin, elongated plates that have a double refraction value of 0.185. The compound was first prepared by Jousselin in 1877, but it was not until after 1900 that it found limited use in propellent compositions. During World War I, it was used by the Germans as an ingredient of a bursting charge explosive. At present, its chief use is as an ingredient of propellent compositions, because the gases produced by the explosion of nitroguanidine propellants are less erosive than those produced by propellants of comparable force.

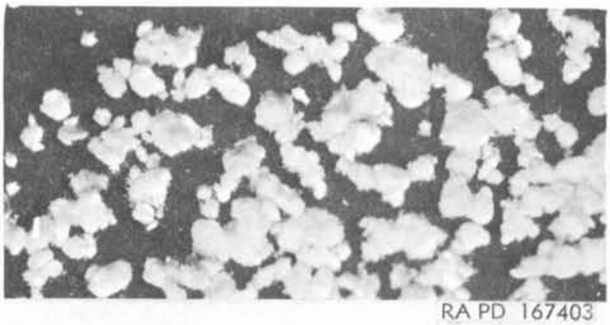


Figure 7–25. Nitroguanidine crystals, 1½ x.

b. The melting point of nitroguanidine varies somewhat with the rate of heating. The pure material melts with decomposition at 232° C. when the rate of heating is moderate, but values from 220° to 250° C. are obtainable with varying rates. Nitroguanidine has a crystal density of 1.715, but because of the toughness and flexibility of its crystals has a density of only 0.95 when subjected to a pressure of 3,000 psi. The heat of combustion of nitroguanidine at constant pressure is 1,995 calories per gram, and from this is derived a heat of formation value of 23.58 kilogram-calories per mole. Both forms of nitroguanidine are soluble in water to the extent of 0.4 gram per 100 grams at 25° C. and 8.25 grams at 100° C.; but the material is nonhygroscopic when exposed to an atmosphere of 90 percent relative humidity at 30° C. Insoluble in ether, nitroguanidine is slightly soluble in ethanol or aqueous potassium hydroxide. At 25° C., sulfuric acid solutions of 15, 30, and 45 percent concentration dissolve nitroguanidine, so as to form solutions containing 0.55, 2.9, and 10.9 grams, respectively, per 100 milliliters.


RA PD 167404

Figure 7–26. Nitroguanidine crystals, 10 x.

c. Neither form of nitroguanidine is converted into the other by recrystallization from water, and the two forms can be separated by fractional crystallization from water. The β-form can be converted into the α-form by solution in concentrated sulfuric acid and precipitation with water or by decomposition of the nitrate or hydrochloride formed by β-nitroguanidine. When α-nitroguanidine is decomposed by heat, some β-nitroguanidine is formed; but the β-compound generally is obtained by the nitration of the mixture of guanidine sulfate and ammonium sulfate, resulting from the hydrolysis of dicyandiamide, $H_2N.C(:NH).NH.CN$, by sulfuric acid. The α-form is obtained by the reaction of guanidine and nitric acid to form the nitrate and dehydration of this to the nitrocompound.

d. Nitroguanidine is chemically reactive because of a free amino group as well as a nitro group and a tendency toward dearrangement. With hot concentrated nitric acid, it forms a nitrate that melts at 147° C. and, with strong hydrochloric acid, it yields a crystalline hydrochloride. On reduction, nitroguanidine yields nitrosoguanidine, $H_2N.C(:NH).NH.NO$, and then aminoguanidine, $H_2N.C(:NH).NH.NH_2$. With a solution of diphenylamine in sulfuric acid, nitroguanidine gives a blue color. When it is tested with ferrous ammonium sulfate and sodium hydroxide, a fuchsine color is developed. When warmed with water and a large excess of ammonium carbonate, nitroguanidine forms guanidine carbonate. With an aqueous solution of hydrazine it reacts to form *N*-amino, *N′*-nitroguanidine, $N_2N.HN.C(:NH).NH.NO_2$, which melts at 182° C. On being heated or undergoing reaction in sulfuric acid solution, nitroguanidine appears to undergo dearrangement according to two modes:

$$H_2N.C.(:NH).NH.NO_2 \rightarrow NC.NH_2 + H_2\ N.NO_2$$

Cyanamide Nitramide

$$H_2N.C(:NH).NH.NO_2 \rightarrow NH_3 + NC.NH.NO_2$$

Ammonia Nitrocyanamide

When decomposed by heating above its melting point, the products obtained from nitroguanidine are water, nitrous oxide, cyanamide, melamine, ammonia, cyanic acid, cyanuric acid, ammeline, and ammelide. These are the products to be anticipated from the dearranged compound. In addition, there are formed carbon dioxide, urea, melam, melem, mellon, nitrogen, hydrocyanic acid, cyanogen, and paracyanogen by the reaction and decomposition of the primary products of decomposition. In aqueous solution, nitroguanidine dearranges in both of the above modes, but this is only to a small degree, unless another compound is present to react with one of the products of dearrangement. As a result of this, nitroguanidine is relatively stable in aqueous solution. A saturated aqueous solution at 25° C. has a pH value of 5.5. After being heated at 60° C. for 2 days, such a solution has a pH value of 8.5. In the absence of ammonia and in the presence of a primary aliphatic amine, nitroguanidine reacts to form an alkylnitroguanidine as if the amine reacted with nitrocyanamide present by dearrangement. A solution of either form of nitroguanidine in concentrated sulfuric acid acts chemically as if the nitroguanidine had dearranged into nitramide and cyanamide, although α-nitroguanidine is precipitated if the solution is poured into water. When such a solution is warmed, nitrous oxide and a little nitrogen are evolved at first from the dehydration of nitramide. Later and more slowly, there is liberated carbon dioxide resulting from the hydrolysis of cyanamide. After standing for some time, a sulfuric acid solution of nitroguanidine no longer gives a precipitate of nitroguanidine when diluted with water. Although a freshly prepared solution contains no nitric acid, this is formed by the hydration of nitramide, if there is present a material that can react with nitric acid. Consequently, nitroguanidine splits off the nitro group quantitatively and the purity of the material can be determined by the nitrometer method.

e. Nitroguanidine is manufactured by reacting guanidine and nitric acid or dicyandiamide and ammonium nitrate to form guanidine nitrate and dehydrating this by means of sulfuric acid.

$$H_2N.C(:NH).NH_2+HNO_3 \rightarrow H_2N.C(:NH).NH_2.HNO_3$$

$$H_2N.C(:NH).NH.CN+NH_4NO_3 \rightarrow H_2N.C(:NH).NH_2HNC_3+NC.NH_2$$

$$H_2N.C(:NH).NH_2.HNO_3 \rightarrow H_2O+H_2N.C(:NH).NH.NO_2$$

As the guanidine or dicyandiamide can be produced from the raw materials coke, limestone, atmospheric nitrogen, and water, the production of nitroguanidine does not involve the use of special natural resources. However, a very large amount of electrical energy is required for the production of dicyandiamide or guanidine. The dehydration of guanidine nitrate to nitroguanidine is effected by adding 1 part of the nitrate to 2.3 parts by weight of sulfuric acid (95 percent), so that the temperature does not rise above 10° C. As soon as all the nitrate has been dissolved, the milky solution is poured into seven and one-half parts of ice and water. The mixture is kept ice-cold until precipitation is complete, when the nitroguanidine is caught on a filter, washed with cold water, and redissolved in 10 parts of boiling water. The nitroguanidine recrystallizes when the solution cools. The yield is approximately 90 percent of the theoretical.

f. The specification for military nitroguanidine covers but one grade of material, comprising two classes with respect to granulation. The requirements with which the explosive must comply are as follows:

Color: White.

Form: Crystalline powder.

Purity: Minimum, 99.5 percent.

Ash content: Maximum, 0.30 percent.

Total volatiles: Maximum, 0.25 percent.

Water-insoluble impurities: Maximum, 0.20 percent.

Water-soluble impurities: Maximum, 1.00 percent.

Acidity: Maximum, 0.06 percent, calculated as sulfuric acid.

Chlorides: Maximum, 0.02 percent, calculated as sodium chloride.

Sulfates: Maximum, 0.20 percent, calculated as sodium sulfate.
pH of water extract: Minimum, 4.5; maximum, 7.0.

Specific surface:
Class *A*. Minimum, 12,000 square centimeters per cubic centimeter.

Maximum, 18,000 square centimeters per cubic centimeter.

Class *B*. Minimum, 18,001 square centimeters per cubic centimeter.

As nitroguanidine is not dissolved when used in the manufacture of propellent compositions, but must be dispersed in the other ingredients in the form of small particles, the specific surface (granulation) requirements assure the degrees of fineness necessary when so used. The other requirements assure control of a variety of impurities that might accumulate during the manufacture of calcium cyanamide, cyanamide, dicyandiamide, or guanidine.

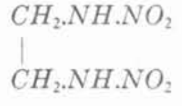
g. Although the two impact tests give inconsistent values for nitroguanidine, the preponderance of the evidence is that this explosive is considerably less sensitive than TNT to impact as well as friction and initiation. Nitroguanidine is not exploded in the rifle bullet impact test, in spite of its relatively low explosion temperature test value (275° C.). In the sand test, it cannot be detonated by lead azide or mercury fulminate, a minimum charge of 0.10±0.05 gram of tetryl being required and the sensitivity to initiation varying with the crystal size. Larger quantities can be detonated by a blasting cap containing 1.5 grams of mercury fulminate. Nitroguanidine, therefore, may be classified as one of the least sensitive of the military explosives.

h. Nitroguanidine is 77 percent as brisant as TNT, as judged by the sand test, although at a density of 1.55, it has a higher rate of detonation (7,650 meters per second) than does TNT at the same density (6,900 meters per second). Although a ballistic pendulum test has indicated nitroguanidine to be 104 percent as powerful as TNT, this probably is incorrect, since the Trauzl lead block test value and the heat of explosion of nitroguanidine are only 78 percent of the corresponding values for TNT. The relatively small heat of explosion of nitroguanidine explains its cooling effect when present in propellent compositions.

i. Nitroguanidine begins to undergo decomposition at a higher temperature (232° C.) than does TNT (180° to 200° C.), and vacuum stability tests at 120° C. show the two to be of the same order of stability. As both are essentially nonhydroscopic and very slightly soluble in water, the impairment of stability by moisture is not a practical problem. Propellent compositions containing nitroguanidine have been found to be of a high order of stability; and it appears that the nitroguanidine acts to some extent as a stabilizer, since such compositions generally do not liberate red fumes when subjected to the 65.5° C. surveillance test.

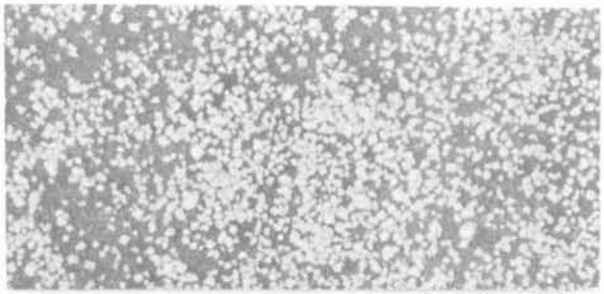
7–15. Haleite

a. Ethylenedinitramine, N,N′-dinitroethylenediamine, haleite, or EDNA is a white crystalline compound (figs. 7–27 and 7–28) having the structure



$$\begin{array}{l} CH_2.NH.NO_2 \\ | \\ CH_2.NH.NO_2 \end{array}$$

and a molecular weight of 150.10. It forms orthorhombic crystals that have refractive index values of $\alpha=1.427$, $\beta=1.686$ and $\gamma=1.730$. The compound was first described in 1887 by Franchimont and Klobbie, but it was not until 1935 that its value as an explosive was recognized. During World War II it was standardized as a military explosive and named haleite, in recognition of its development by the late



RA PD 167405

Figure 7–27. Haleite crystals, 1½ x.

G. C. Hale. It has been found suitable for use as a bursting charge explosive and as an ingredient of the binary explosive ednatol.

b. Haleite melts with decomposition at 177.3° C. It has a crystal density varying from 1.66 to 1.77, depending upon the solvent from which it is crystallized. When subjected to pressures of 5,000, 10,000, 20,000, 30,000, and 40,000 psi, it has density values of 1.30, 1.40, 1.51, 1.55, and 1.58, respectively. The solubility of haleite in water at 10°, 20°, 50°, 75°, and 95° C. is 0.10, 0.25, 1.25, 5.0, and 16.4 grams, respectively, per 100 grams of solvent; but it is hygroscopic to the extent of only 0.01 percent when exposed to an atmosphere of 90 percent relative humidity at 30° C. In 95 percent, ethanol at 10°, 20°, 50°, 75°, and 78.5° C., haleite is soluble to the extent of 0.55, 1.00, 3.50, 10.07, and 11.5 grams, respectively, per 100 grams of solvent. It is soluble also in methanol. In actetone at 20° C., haleite is soluble to the extent of 8.2 grams per 100 grams; but it is insoluble in ether. It is soluble also in nitro-methane, nitrobenzene, and dioxane, which can be used to recrystallize the explosive. The heat of combustion of haleite at constant pressure is 2,490 calories per gram, from which is derived a heat of formation value of 20.13 kilogram-calories per mole.

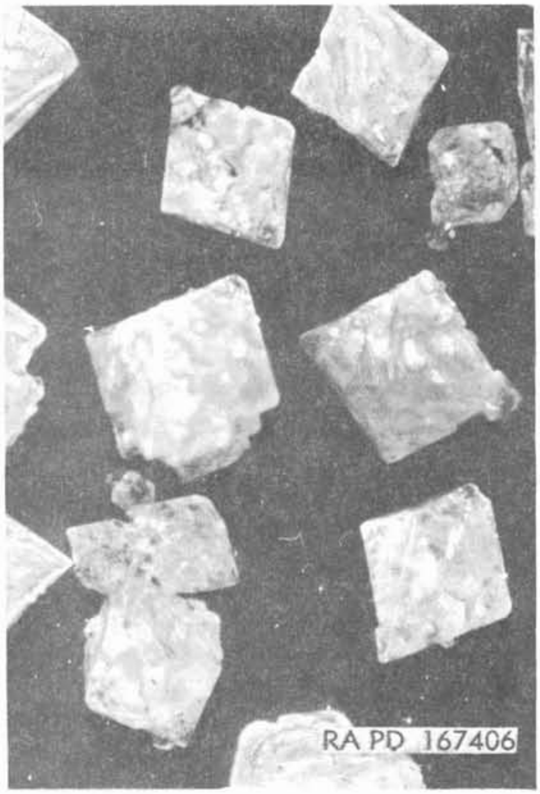
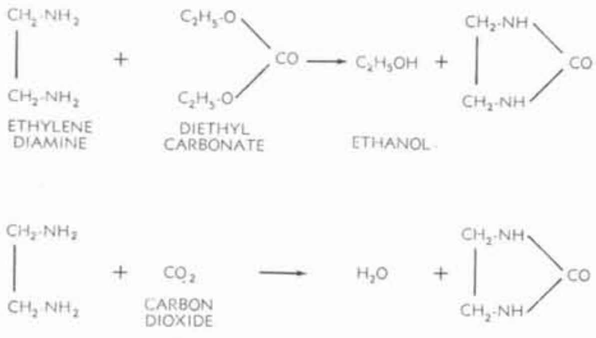


Figure 7-28. Haleite crystals, 25 x.

c. The two amino hydrogen atoms of haleite are chemically active and can be replaced by a metal, when the explosive is dissolved in an aqueous solution of the metallic hydroxide. The potassium compound forms needles, when crystallized from ethanol. The ammonium compound is soluble in water, insoluble in ethanol, and so insensitive to initiation as to require a booster charge of tetryl in the sand test. The lead compound is formed by adding lead nitrate to an aqueous solution of the sodium compound. It is insoluble in water, as sensitive to impact as PETN, and can be initiated to detonation by mercury fulminate. With formaldehyde and ammonia, haleite reacts to form an explosive compound that is almost as brisant as haleite, slightly more sensitive to impact, but somewhat less stable. An aqueous solution of haleite has a slightly acid reaction and prolonged boiling of this causes some decomposition. When haleite is refluxed with dilute sulfuric acid, it is decomposed into nitrous oxide, acetaldehyde, and ethylene glycol. When heated at temperatures greater than 75° C., haleite undergoes decomposition at rates increasing logarithmically with increase in temperature. at 100° and 150° C., heating for 5,800 and 3 hours, respectively, is necessary to cause 1 percent decomposition of the explosive. Dry haleite does not cause the corrosion of aluminum, brass, copper, mild steel, stanless steel, cadmium, nickel, or zinc; but in the presence of 0.5 percent moisture, all of these except aluminum and stainless steel are corroded to a considerable extent by haleite.

d. The manufacture of haleite involves the preparation of ethyleneurea (2-imidazolidone) and nitrating this to the dinitrocompound, which is then hydrolyzed to form haleite. Dinitroethyleneurea can be prepared by each of the following reactions:



RA PD 212848

Figure Q. Dinitroethyleneurea.

The ethyleneurea can be nitrated with either concentrated nitric acid or a mixture of nitric and sulfuric acids. In the latter case, 10 parts of mixed acid containing 74.0 percent sulfuric acid, 15.4 percent nitric acid, and 10.6 percent water is cooled to 10° C. or less and agitated. One part of ethyleneurea is added at such a rate that the temperature does not rise above 10° C. After the last portion of the ethyleneurea has been added, stirring is continued for 5 minutes. A variation of this process is to dissolve 1 part of ethyleneurea in 7.5 parts of 95 percent sulfuric acid, while maintaining the temperature at 20° C. This solution then is added slowly, while maintaining the temperature of the mixture at 0° C. or less, to 3.33 parts of a mixed acid containing 15.8 percent sulfuric acid, 66.7 percent nitric acid, and 17.5 percent water. When addition is complete, the temperature is increased to 10° C. and maintained at that value for 2 hours. The dinitroethyleneurea is caught on a filter and washed with ice water until the washings are almost neutral. Additional dinitroethyleneurea is recovered by drowning the spent acid in ice-water, a total yield of 97 percent of the theoretical yield being obtainable. The dinitroethyleneurea is suspended in 7 times its weight of water and this is boiled until the evolution of carbon dioxide ceases. The reaction is:

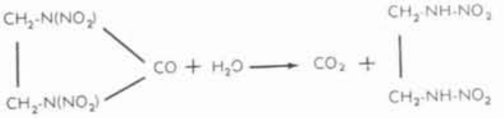


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Figure R. Boiling of dinitroethyleneurea.

Haleite separates as shining crystals when the solution is cooled to room temperature. This is caught on a filter, washed with cold water, and dried. The total yield of haleite from ethyleneurea is approximately 92 percent of the theoretical.

e. One grade of haleite is specified for military purposes. This complies with the following requirements:

Color: White to buff.
Moisture: Maximum, 0.10 percent.
Melting point: Minimum, 174.0° C.
Purity: Minimum, 99.0 percent.
Water—insoluble matter; Maximum, 0.10 percent.
Grit: None.
120° C. vacuum stability test:
  Maximum, 5.0 milliliters of gas in 40 hours.
Granulation:

	Percent
Through No. 10 sieve, minimum	100
Through No. 100 sieve, maximum	20

Purity is determined by reacting a weighed sample of haleite with an excess of standardized sodium hydroxide solution and determining the excess of hydroxide by titration with standardized acid, using phenolphthalein as the indicator.

f. Impact tests show haleite to be less sensitive than tetryl and more sensitive than TNT, but the rifle bullet impact test indicates it to be less sensitive than TNT, in spite of its very low explosion temperature test value (190° C.). It is almost as sensitive to initiation as tetryl, as judged by its minimum detonating charge of lead azide or mercury fulminate. Haleite therefore has an unusual combination of sensitivity characteristics that, in conjunction with other properties, renders it particularly applicable for special purposes.

g. The brisance of haleite as measured by the sand test is almost equal to that of tetryl, but it is about 12 percent more brisant than tetryl, as indicated by fragmentation tests. The rate of detonation of haleite is greater than that of tetryl at the same density (7,750 and 7,340 meters per second at a density of 1.55). The ballistic pendulum test indicates haleite to be 106 percent as powerful as tetryl, but the Trauzl lead block test indicates it to be only 95 percent as powerful as tetryl. The heat of explosion of haleite is 114 percent that of tetryl and it liberates 119 percent as much gas, so the superiority of haleite from this viewpoint is conclusive.

h. As indicated by vacuum stability tests, haleite is very slightly less stable than tetryl at 100° and 120° C. This slight difference is shown also by 100° C. heat and 75° C. international tests, but this may be due in part to the greater volatility of haleite. Tests have shown it to be volatilized at the rate of 0.05 percent per hour when heated at 100° C. in an open dish. Storage tests have shown dry haleite to be of unimpaired stability after storage at 65° C. for 5 months or at 50° C. for 30 months. It therefore is considered to be of satisfactory stability at the temperatures to which it may be subjected during handling and storage.

i. Haleite is not particularly toxic, but inhalation of its dust should be avoided and frequent baths and changes of clothes are recommended for workers handling the explosive in quantity.

7-16. RDX

a. Cyclotrimethylenetrinitramine, hexahydro-1, 3, 5-trinitro-5-triazine, RDX, cyclonite, hexogen, or T4 is a compound having the structure

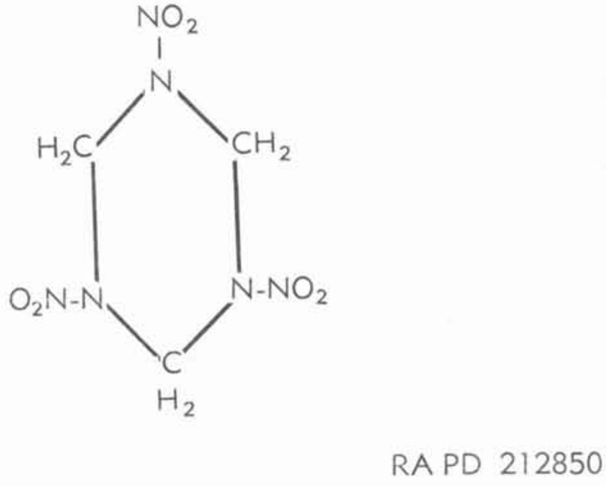
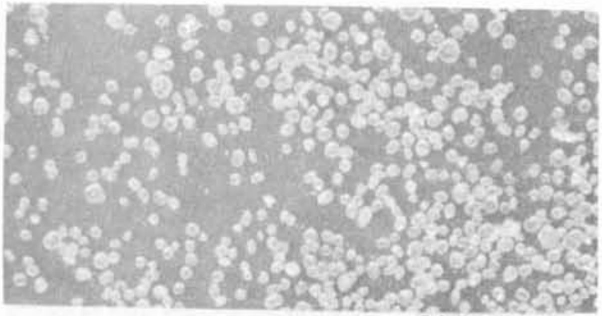


Figure S. T4.

and a molecular weight of 222.126 that forms colorless orthorhombic crystals (figs. 7-29 and 7-30). It was first prepared by Henning in 1899, but it was not until 1920 that von Herz discovered its value as an explosive. Subsequent development of the formaldehyde and ammonia industries was required for the cheap production of RDX, and it was not until World War II that it was used on a large scale. It then was used by all the major participants, the Germans calling it hexogen and the Italians T4. It has found extensive use as a base charge for detonators and, in admixture with other materials, as an explosive for shell and bombs.

b. Pure RDX melts at 204.1° C. and has a crystal density of 1.816. When subjected to pressures of 5,000, 10,000, 20,000, 30,000, and 50,000 psi, it has density values of 1.52, 1.60, 1.68, 1.70, and 1.72, respectively. It has a scratch hardness of 2 to 3 on the Mohs' scale and so is intermediate between talc and gypsum. At 20° C., it has a specific heat of 0.30 calorie per gram per degree Centigrade. The heat of combustion of RDX at constant pressure is 2,307.2 calories per gram, from which is derived a heat of formation value of −24.17 kilogram-calories per mole. RDX is soluble in water to the extent of only 0.0076 gram at 25° C. and 0.196 gram at 96° C. per 100 grams of solvent. The pure compound is nonhygro-



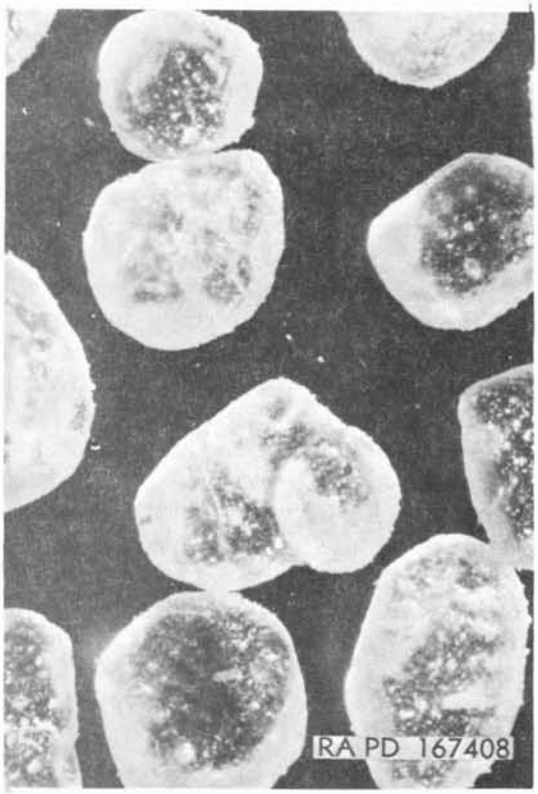
RA PD 167407

Figure 7-29. RDX crystals, 1½ x.

scopic at 90 percent relative humidity and 30° C. The solubility of the explosive in other solvents is given in table 7-19.

Table 7-19. Solubility of RDX

° C.	Solubility in grams per 100 grams of solvent at–				
	20	25	50	70	97
Acetone	7.4	------	12.8		
Cyclohexanone	------	12.7	------	------	25
Nitrobenzene	------	1.5	------	------	12.4
Mesityl oxide	------	3	------	------	12
Methyl acetate	2.95	------	6.0		
Methanol	0.235	------	0.735		
Ethanol	0.105	------	0.370	------	------
Ether	0.055				
Benzene	0.045	------	0.115	0.300	------
Toluene	0.020	------	0.085	0.210	
Carbon tetrachloride.	0.0	------	0.005	0.015	
Carbon disulfide.	0.0				
Chloroform	0.008				
Chlorbenzene	0.33	------	0.7		



RA PD 167408

Figure 7-30. RDX crystals, 25 x.

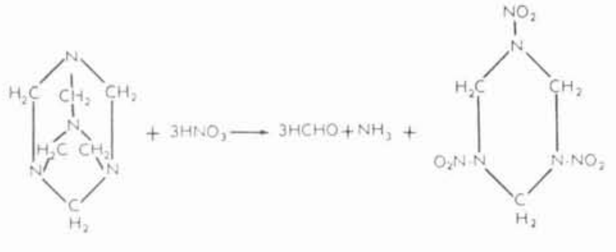
RDX is readily soluble in hot aniline or phenol and dissolves in warm concentrated nitric acid, but separates only partially when the acid solution is cooled. It dissolves very slowly in cold concentrated sulfuric acid.

c. RDX is hydrolyzed slowly when treated with boiling, dilute sulfuric acid or sodium hydroxide:

$$C_3H_6N_6O_6 + 6H_2O \rightarrow 3NH_3 + 3HCHO + 3HNO_3$$

In sulfuric acid solution the nitric acid oxidizes the formaldehyde to formic acid and itself is reduced to nitrous acid. RDX dissolved in cold concentrated sulfuric acid decomposes when the solution is allowed to stand. On reduction of RDX dissolved in hot phenol, it yields methylamine, nitrous acid, and hydrocyanic acid; but if solid RDX is suspended in 80 percent ethanol and reduced, both hydrolysis and reduction take place and the products are methylamine, nitrous acid, ammonia, and formaldehyde. With diphenylamine and sulfuric acid, RDX gives a strong blue color. RDX does not cause the corrosion of aluminum or stainless steel, even in the presence of moisture. Dry RDX causes no corrosion and RDX with 0.5 percent moisture causes only very slight corrosion of nickel or zinc. Dry or damp RDX causes only slight or very slight corrosion of copper, brass, mild steel, or cadmium.

d. RDX is manufactured by the nitration of hexamethylenetetramine, which is obtained by the reaction of formaldehyde and ammonia. The nitration reaction is:



RA PD 212851

Figure T. Nitrolysis reaction.

The formaldehyde liberated by the reaction is oxidized by the nitric acid, if the mixture is allowed to stand or is warmed. It remains in the spent acid after drowning and causes difficulty in recovery of nitric acid from the spent acid. As a result of the rupture and degradation of the hexamethylene-tetramine molecule, there are formed numerous aliphatic and cyclic nitrocompounds that are present in crude RDX. The most important of these is HMX or cyclotetramethylenetetranitramine,

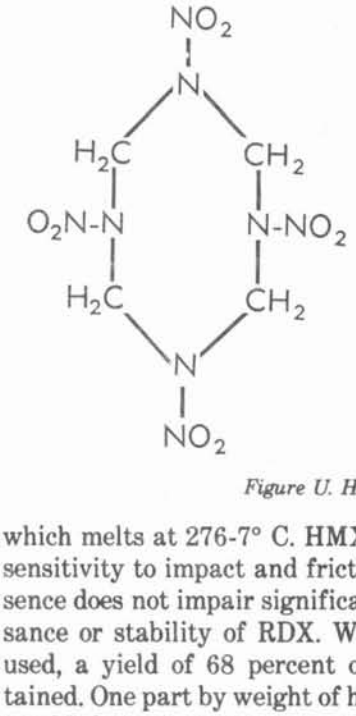


Figure U. HMX RA PD 212852

which melts at 276-7° C. HMX is of the same order of sensitivity to impact and friction as RDX and its presence does not impair significantly the sensitivity, brisance or stability of RDX. When nitric acid alone is used, a yield of 68 percent of the theoretical is obtained. One part by weight of hexamethylenetetramine is added slowly to 11 parts of 100 percent nitric acid, the temperature of which is maintained at 30° C. or less while being stirred vigorously. After being cooled to 0° C., the mixture is stirred for 20 minutes more and then drowned in ice water. The crude RDX is caught on a filter and washed with water to remove most of the acid. Removal of all but a trace of acid is important and purification is accomplished by grinding the wet material and subjecting it to treatments with boiling water or by recrystallization from cyclohexanone or acetone.

e. There are many classes of RDX used for military purposes. One class is distinguished from another class by its granulation. Type A RDX is made by the nitric acid process, while type B material is made by the acetic anhydride process. Classes A and B RDX are used for the manufacture of composition B. The requirements for the two types and classes are as follows:

Color: White.

Total acetone insoluble: Maximum, 0.05 percent.

Inorganic insoluble matter: Maximum, 0.03 percent.

Grit: Retained on No. 40 sieve, none. Retained on No. 60 sieve, maximum, 5 particles per 50 grams.

Melting point: Type A.—Minimum, 200° C.
Type B.—Minimum, 190° C.

Acidity: Type A.—Maximum, 0.05 percent as nitric acid.
Type B.—Maximum, 0.02 percent as acetic acid.

Granulation:

Through sieve No.		Class A, percent	Class B, percent
20	Minimum	100	100
35	Minimum		99
50	Minimum	97	93
100	Minimum	50	65
200	Minimum		30
200	Maximum	40	60

f. Impact tests show RDX to be of the same order of sensitivity as tetryl, but pendulum friction and rifle bullet impact tests indicate RDX to be definitely more sensitive than tetryl. The two explosives have essentially the same explosion temperature test value. They are equally sensitive to initiation by mercury fulminate in the sand test, but RDX is more sensitive than tetryl to initiation by lead azide. Comparative impact tests have shown the two types of RDX to be equally sensitive to impact, friction, heat, and initiation. This is due to the fact that the sensitivity characteristics of HMX, the chief impurity in type B RDX, are very similar to those of RDX. Temperature has a marked effect on the sensitivity of RDX. Impact tests of a sample of type A RDX at 20°, 88°, and 105° C. gave values of 9, 8, and 5 inches, respectively. RDX is much less sensitive to an electric spark than tetryl, TNT, or PETN. In the presence of more than 14 percent moisture, it cannot be detonated by a No. 6 electric blasting cap.

g. Although sand test values indicate RDX to be slightly less brisant than PETN, fragmentation and plate dent tests indicate the reverse. The two explosives have essentially the same rate of detonation. Ballistic pendulum and Trauzl lead block tests show RDX and PETN to have the same explosive power and be 115 to 132 percent as powerful as tetryl. The heat of explosion value of RDX is 116 percent that of PETN, and it may be concluded that RDX is the second most powerful standard military explosive, netroglycerin being the most powerful.

h. At temperatures of 100° to 150° C., the stability of RDX is slightly less than that of TNT, as judged by vacuum stability tests, but 75° C. international and 100° C. heat tests indicate that at these temperatures RDX is as stable as TNT. It has been found to withstand storage at 85° C. for 10 months without impairment of stability and must be considered highly satisfactory in this respect.

i. RDX does not appear to be markedly toxic, and the only precautions prescribed for those working continuously with large quantities of the explosive are daily baths and changes of clothes.

j. PB-RDX. An explosive consisting of RDX coated with polystryrene, plasticized with dioctyl phthalate, was initially developed in 1952. The specific formulation of 90/8.5/1.5RDX/polystyrene/dioctyl phthalate was subsequently standarized. This explosive, originally designated PBX, has been redesignated PB-RDX. Attempts to prepare it by blending the dry ingredients together in a Baker Perkins-type mixer yielded a product which was nonuniform in granulation and very dusty. The final procedure for its preparation was a wet process which incorporated the better features found from a study of such variables as solvents, solvent/plastic ratios, lacquer addition and temperature, agitation, RDX particle size distribution, dispersants and rosin additive. This slurry method gave a product which was uniform, free flowing and dustless, and exhibited satisfactory drying, handling and pressing characteristics.

7-17. HMX

a. Cyclotetramethylene tetranitramine (H-MX), homocyclonite, or 1, 3, 5, 7 tetranitro 1, 3, 5, 7 tetrazacyclooctane is a compound having the structure illustrated in paragraph 7-16*d.* It was discovered as an impurity (by-product) in the nitration of hexamethylene tetramine to form RDX. When nitration is carried out with nitric acid alone, HMX is formed in relatively small amounts, but when nitration is carried out in the presence of acetic anhydride and ammonium nitrate, the product contains approximately 10 percent of HMX. It is a crystallive solid with a molecular weight of 296.17. Ordinarily, the least sensitive β form is produced, though the compound exists in at least 4 polymorphic forms. The most sensitive form is the α, which forms under certain conditions. It is practically insoluble in water and is nonhygroscopic. Its solubility in other solvents is comparable with that of RDX.

b. Pure HMX melts at 276-7° C. and has a crystal density of 1.87. Its scratch hardness is 2.3 on the Mohs scale. At constant pressure it has a heat of combustion value of 2230 cal. per gram and a heat of explosion of 1356 cal. per gram. Its heat of formation has been calculated to be -60.5 cal. per gram. At a density of 1.84 its detonation rate is 9124 meters per second.

c. Preparation.

(1) Glacial acetic acid and acetic anhydride are mixed, the temperature of the mixture being brought to 45° ± 1° C. and held there for the duration of the entire reaction. The reagents (hexamine in glacial acetic acid, acetic anhydride and a solution of 42.3/57.7—ammonium nitrate/98% nitric acid) are then added simultaneously, continuously and equivalently over a 25 minute period. The reaction mixture is aged 15 minutes. This initial operation requires two men to regulate the addition of reagents and control of temperatures. The reagents (42.3/57.7—ammonium nitrate/98% nitric acid and acetic anhydride) are then added simultaneously, continuously and equivalently over a 25 minute period. The mixture is aged 65 minutes, poured into water and simmered on a steam bath for 12 hours. The RDX-HMX precipitate is cooled, filtered, and dried. The RDX is destroyed, leaving HMX by treating the RDX-HMX mixture with a solution of sodium tetra borate decahydrate in water. The RDX-HMX and borate solution are heated to boiling, with agitation. To this solution is added 5N NaOH at the rate of 3cc/min. When about 750 cc have been added the pH increases sharply from a little over 8.7 to over 9.7, which corresponds to complete destruction of the RDX. The HMX is filtered from the hot mixture and has a melting point of 279.5–280.5° C. It is purified by recrystallization from nitromethane and has a melting point of 281–282° C. at this time.

(2) For compositions of RDX-HMX containing 80 percent or more of HMX, the RDX is removed by the following procedure: The RDX-HMX is slurried with acetone and agitated at room temperature. Before complete settling, the RDX-HMX-acetone mixture is decanted. Acetone is added to the residual HMX-RDX in order to slurry it. The slurry is heated to boiling on a steam bath, and while boiling, is agitated for several minutes. The boiling RDX-HMX-acetone solution is decanted. The residual HMX is now washed with cold acetone into a funnel. This HMX is now taken up in 95 percent alcohol, filtered and dried. The yield is 70.78 percent.

(3) All the acetone extracts are combined and evaporated to dryness; yield, 26.5 percent.

Yield balance	*Percent*
Pure HMX obtained	70.78%
Total RDX-HMX mixture recovered.	26.50%
Samples taken during process	0.48%
Loss during process	2.24%
	100.00%

d. HMX is of the same order of sensitivity to impact and friction as RDX, but is less sensitive to initiation, having higher explosion temperature test value (337° C.) than RDX (260° C.). The sand test (60.4) indicates it to be 90 percent as brisant as RDX. The ballistic pendulum test shows the two compounds to have the same explosive power but the Trauzl lead block test shows HMX to be only 90 percent as powerful as RDX. HMX is the more stable, as indicated by the 150° C. vacuum stability test values of .6 and 2.5 ml. of gas, respectively. HMX is more resistant to decomposition by sodium hydroxide than RDX. Its toxicity characteristics are very similar.

e. HMX, though almost as powerful as RDX, is seldom used by itself in military explosive applications; it is usually mixed with a compound, such as TNT, in its uses. Variations of such compositions, their preparation and properties follow:

(1) Octol 70/30

The composition of this material is
HMX 70
TNT 30

(2) Octol 75/25

The composition of this material is
HMX 75
TNT 25

(3) HTA-3

The composition of this material is

HMX 49

TNT 29

Aluminum 22

(4) Preparation—The octols are prepared by mixing water-wet HMX slowly with molten TNT in a steam-jacketed kettle and stirring until all the moisture is evaporated. The composition is then cooled to a satisfactory pouring temperature and cast directly into ammunition components, or prepared in the form of chips to be stored for later use. HTA-3 is similarly prepared with the aluminum added when all the moisture had been evaporated.

(5) *Properties of HMX Compositions*

	Density	*Heat of Combustion*	*(Cal/gm) Explosion*	*Explosion Temp ° C.*	*Sand Test gms*	*Detonation Rate m/sec*
Octol 75/25	1.81	2676	1131	350	62.1	8377 (1.81)
Octol 70/30	1.80	2722	1074	335	58.4	8643 (1.81)
HTA	1.90	3687	1190	370	61.3	7866 (1.90)

7-18. Amatol

a. During World War I, in order to conserve the limited supply of TNT, the British developed two compositions termed amatols. A mixture containing 80 parts of ammonium nitrate and 20 parts of TNT, termed 80-20 amatol, was manufactured by mixing molten TNT at 90° C. with ammonium nitrate heated to the same temperature. The resulting mixture, resembling wet brown sugar, was loaded while hot into shell by means of an extrusion machine. This composition is essentially oxygen-balanced and has a rate of detonation of only 5,300 meters per second. It is less brisant but has more power than TNT. Difficulties in the loading of 80-20 amatol made it preferable to use a mixture of approximately equal weights of ammonium nitrate and TNT termed 50-50 amatol (fig. 7-31). This can be melt-loaded and was used extensively in shell during World War I. During the early part of World War II, 65-35 amatol was used in shell and bombs, but the rapid development of a hugh supply of TNT removed the necessity for using ammonium nitrate insteat of TNT. The availability of more powerful binary ex-

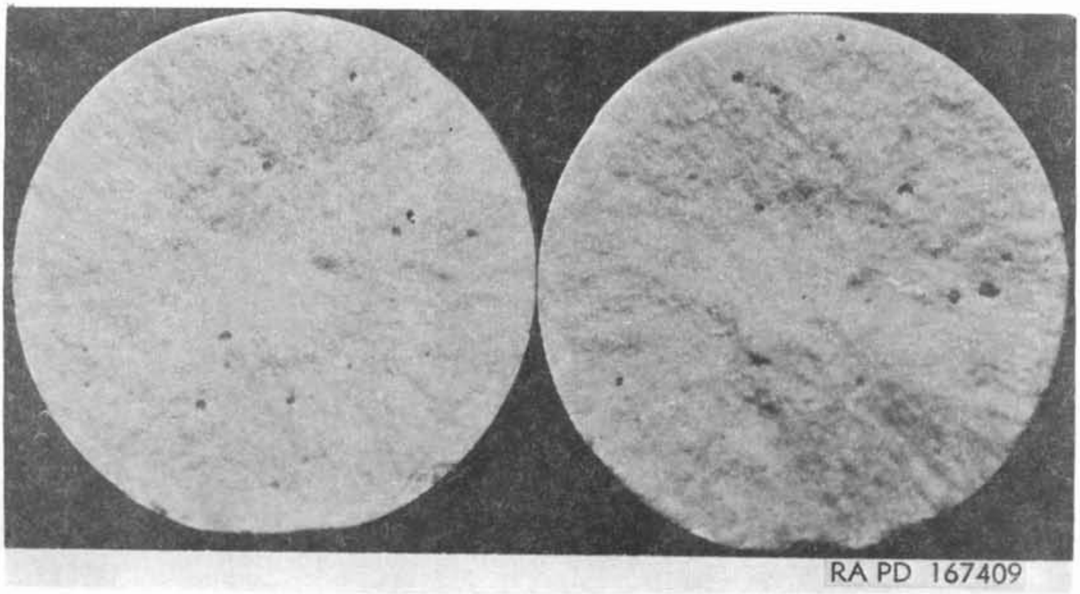


Figure 7-31. Cast 50-50 amatol, 1½ x.

plosives, such as composition B and pentolite, was an additional factor in the disuse of amatol.

b. As ammonium nitrate is insoluble in liquid TNT, melting of 50–50 amatol does not begin until the melting point of TNT is reached. Cast 50–50 amatol has a density of 1.59 to 1.60 and a specific heat value of 0.383 over the range 20° to 80° C. At constant volume, 50–50 amatol has a heat of combustion value of 2,073 as compared with values of 2,402 and 1,254 for 40–60 and 80–20 amatols, respectively. The solubility of amatol is that of its ingredients. The amatols are very hygroscopic, as the TNT does not moistureproof the ammonium nitrate. The chemical reactivity of amatol is the summation of the reactivities of its ingredients. In the presence of moisture, it corrodes copper, brass, and bronze.

c. In the manufacture of 50–50 amatol, care must be taken to insure proper granulation of the ammonium nitrate, if maximum density of the cast amatol is to be assured. The ammonium nitrate is dried so as to contain not more than 0.25 percent moisture and is heated to about 90° C. before being added to an equal weight of molten TNT contained in a melting kettle equipped with an agitator. After addition of the nitrate is complete, mixing is continued for some time to insure uniformity and permit the temperature of the mixture to decrease to 80° to 85° C. It is then loaded by pouring into shell or bombs.

d. Impact, pendulum friction, and rifle bullet impact tests show 50–50 amatol to be of the same order of sensitivity as TNT or slightly more sensitive. The explosion temperature test value of 50–50 amatol (265° C.) is much lower than that of TNT (475° C.). In the sand test, it is less sensitive to initiation than TNT, requiring a minimum charge of 0.05 gram of tetryl. However, 50–50 amatol can be detonated by diazodinitrophenol, and special tests have shown that less of this initiating agent is required to detonate cast or pressed 50–50 amatol than is required for cast or pressed TNT, respectively.

e. As judged by the sand test, 50–50 amatol is only 81 percent as brisant as TNT, and fragmentation tests support this relationship. The rate of detonation of cast 50–50 amatol is 93 percent of that of TNT. Because of its better oxygen balance and larger volume of gaseous products of explosion, 50–50 amatol has greater power than TNT. Ballistic pendulum and Trauzl lead block tests indicate amatol to have 122 to 124 percent the power of TNT, although the heat of explosion of amatol is only 106 percent that of TNT. 50–50 amatol, however, is not as satisfactory as TNT from the viewpoint of blast effect. Its peak pressure and positive impulse values are only 97 and 87 percent, respectively, those of TNT.

f. The stability of 50–50 amatol is a little less than that of TNT at temperatures of 100° and 120° C., as indicated by vacuum stability tests, there evidently being very slight reaction between TNT and ammonium nitrate at those temperatures. At temperatures below th melting point of TNT (80° to 81° C.), there is no evidence of reaction. After storage at 50° C. for 3 months, there is no change in the sensitivity, brisance, or stability of 50–50 amatol; and many additional months of such storage without effect might be anticipated.

g. Determination of the composition of amatol can be made by extracting a weighted sample with benzene, drying the residue, and weighing this. The weight of the residue is calculated to percentage of ammonium nitrate and the difference between this and 100 percent represents the percentage of TNT.

7–19. Ammonal

a. During World War I, the scarcity of TNT led to the development and use by foreign nations of the ammonals—compositions containing aluminum, ammonium nitrate, and TNT led to the development and use by forsant action. A composition containing 22 percent ammonium nitrate, 11 percent flaked aluminum, and 67 percent TNT was 17 percent more brisant than 50–50 amatol and 83 percent as brisant as TNT. It explodes with a bright flash, and is more sensitive than amatol to initiation.

b. During World War II, the plentiful supply of TNT in the United States rendered unnecessary the use of ammonal or amatol; and the development of explosives having great blast effects rather than brisance led to the use of tritonal by the United States and of minol by Great Britain. Minol is really an ammonal containing less TNT and more ammonium nitrate and aluminum than the formula given above.

7-20. Tritonal

a. Tritonal, a composition containing 80 percent TNT and 20 percent flaked aluminum, was developed and standardized in the United States during World War II. It can be melt-loaded and is used in bombs for its blast effect. When cast it has a density of 1.73 and is a silvery solid in appearance (fig. 7-32).

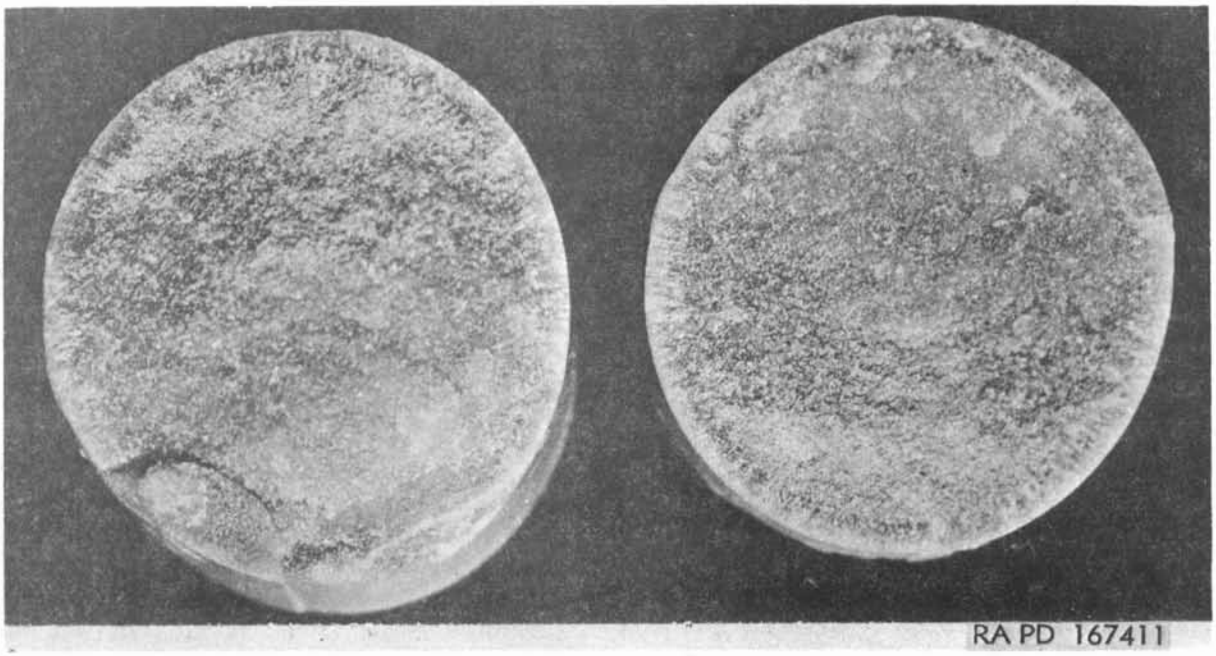

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Figure 7-32. Cast 80-20 tritonal, 1½ x.

b. As metallic aluminum is insoluble in liquid TNT, 80-20 tritonal does not begin to melt until the melting point of TNT is reached. Its calculated specific heat at 20° C. is 0.305 calorie per gram per degree Centrigrade, and it has a heat of combustion value of 4,315 calories per gram at constant pressure, as compared to 3,590 calories per gram for TNT. The thermal conductivity of tritonal at 0° C. is 0.0011 calorie per second per centimeter per degree Centrigrade and therefore twice that of TNT. Like TNT, it undergoes considerable expansion on melting and contraction on freezing. The solubility characteristics of tritonal are those of TNT, and it is essentially nonhygroscopic.

c. The chemical reactivity of tritonal is that of TNT and aluminum. In addition, in the presence of moisture, the TNT and aluminum undergo slight reaction with the evolution of gas. This is not of practical importance, because of the very slight hygroscopicity of TNT and the fact that any moisture present is driven off by heat during the manufacture of tritonal.

d. 80-20 tritonal is manufactured from TNT and grained aluminum. The TNT and aluminum are run slowly through separate chutes into a steam-heated melting kettle equipped with a stirrer, which is kept in motion while the ingredients are being added. Heating and mixing are continued until all the TNT is melted, the temperature is greater than 81° C., and the fluidity of the mixture is considered satisfactory. The tritonal is then loaded by pouring into bombs, with at least the same precautions and techniques used in the melt-loading of TNT.

e. Although the pendulum friction test shows no measurable disference in sensitivity between tritonal and TNT, impact tests indicate tritonal to be somewhat more sensitive to impact than TNT and less so than tetryl. The rifle bullet impact test value for tritonal is more similar to that of tetryl than that of TNT. The explosion temperature test value of tritonal (470° C.) is almost identical with that of TNT. Liquid tritonal at 90° C. is slightly more sensitive than the solid, as judged by rifle bullet impact tests. The minimum detonating charge of lead azide (0.30 gram) for tritonal, obtained by the sand test, is slightly greater than that for TNT (0.26 gram); but nonstandard tests with diazodinitrophenol as the initiator show tritonal and TNT to be of essentially the same sensitivity to initiation.

f. The brisance of tritonal is 97 percent of that of TNT, as judged by sand test values, but plate dent tests indicate it to be only 93 percent and fragmentation tests of shell charges show it to be but 91 percent as brisant as TNT. The rate of detonation of cast tritonal is approximately 97 percnet that of cast TNT. The heat of explosion of tritonal is 59 percent greater than that of TNT, but tritonal is only 124 percent as powerful as TNT, as measured by the ballistic pendulum test. When tested for blast effect, tritonal has relative peak pressure and positive impulse values greater than those of any other binary explosive except torpex, these values being 113 and 118 percent, respectively, of those for TNT.

g. As tritonal has the same 150° C. vacuum stability test value as TNT, aluminum and TNT do not react at that or lower temperatures. Tritonal, therefore, has the same stability as TNT, if free from moisture. Deterioration in the presence of moisture is not serious, probably being limited by the formation of a layer of oxide on the surface of the particles of aluminum that prevents or retards further reaction. Tritonal, like TNT, can undergo exudation if stored at elevated temperatures, but this tendency is less than that of TNT because of the presence of 20 percent by weight of metallic particles that tend to hold oily exudate by surface tension and prevent it from coalescing and exuding.

h. The composition of a sample of tritonal is determined by extracting a weighed sample with benzene, drying the residue, and weighing this. The loss in weight represents TNT and the weight of residue represents aluminum. Each is calculated to a percentage basis.

7-21. Pentolite

a. With the standardization of PETN during World War II, there were developed PETN-TNT mixtures termed pentolites (fig. 7-33). Of these, the one composed of equal weights of the two explosives is the most important and has been used as a bursting charge for grenades and a booster-surround charge. Such use of 50-50 pentolite is based cheifly on its sensitivity to initiation, brisance, and suitability for melt-loading.

b. PETN and TNT form an eutectic mixture that freezes at 77° C. and contains 86.5 percent TNT. Cast 50-50 pentolite, therefore, consist of 42.2 percent PETN and 57.8 percent of the eutectic mixture. It has a density of 1.64 to 1.66 and offers the advantage of 6 percent more charge weight than cast TNT when loaded in the same volume. The solubility characteristics of pentolite are those of its ingredients and it is nonhygroscopic when exposed to air of 90 percent relative humidity at 30° C. Dry pentolite has no corrosive action on mild steel, stainless steel, copper, brass, aluminum, cadmium, nickel, or zinc; but in the presence of 0.5 percent moisture, it has very slight action on copper, brass, and zinc.

c. Pentolite is manufactured by either of two methods. In the slurry method, the PETN is suspended by agitation in water heated above 80° C. When TNT is added, it melts and coats the particles of PETN; and on cooling the slurry with rapid stirring, the TNT solidifies. The granules then are separated on a filter or in a centrifugal wringer and dried at a temperature below 75° C. In the coprecipitation method, the PETN and TNT are dissolved

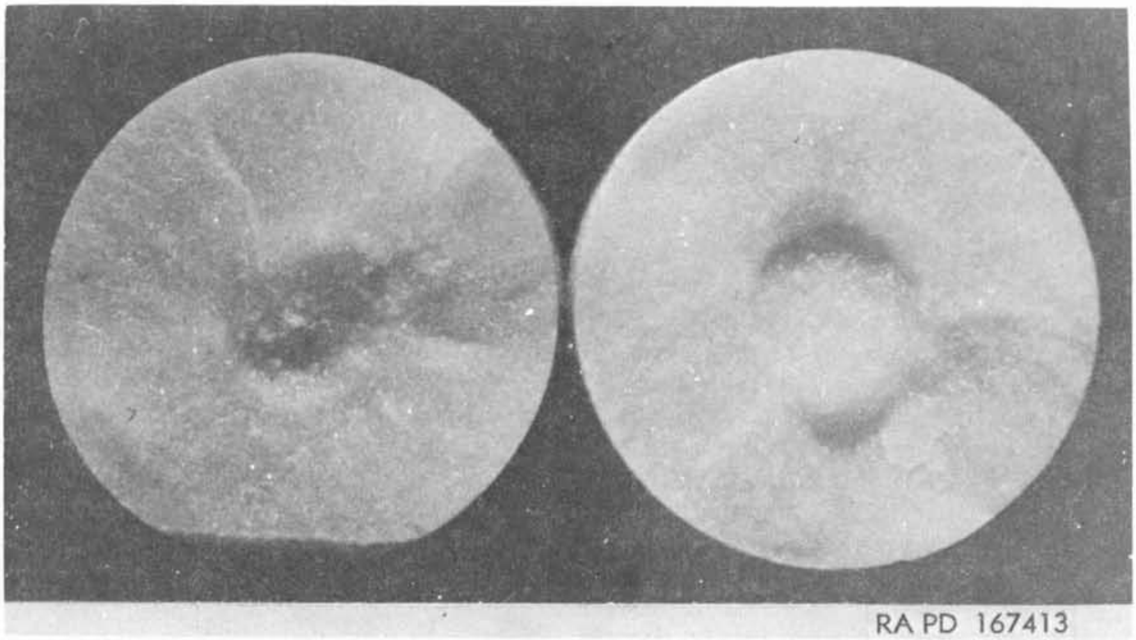

Figure 7–33. Cast 50–50 pentolite 1½ x.

separately in acetone, the solutions are mixed and filtered, and the two explosives are precipitated simultaneously by pouring the solution into water, with vigorous agitation. The precipitated solid is then separated and dried.

d. Two grades of pentolite are used for military purposes. These differ only in that grade II material complies with a bulk density requirement. Grade I is used for melt-loading, while grade II is used in the manufacture of pellets and the press-loading of ammunition. The requirements are—

Volatile matter: Maximum, 0.5 percent.

Composition: PETN, 50 ±2 percent. TNT, 50 ±2 percent.

Total acetone insoluble: Maximum, 0.04 percent.

Inorganic acetone insoluble: Maximum, 0.02 percent.

Grit: None.

Acidity or alkalinity: Maximum, 0.005 percent.

PETN granulation:*

Through sieve number	30	200
Percent, minimum	95	0
Percent, maximum		30

100°C. vacuum stability test: Maximum, 5 milliliters of gas.

Bulk density: Grade II, 0.70—0.10 gram per milliliter.

The composition of pentolite is determined by extracting a weighed sample with chloroform saturated with PETN, drying the residue, and determining its weight. It and the loss in weight are calculated to percentage of PETN and TNT respectively. The grit requirement is included, because of the recognized effect of grit in increasing the sensitivity of PETN to friction and impact.

e. The two impact tests yield contradictory data with respect to the sensitivity of pentolite to impact, one indicating it to be nearly as insensitive as TNT and the other indicating to it be as sensitive as tetryl. The results of rifle bullet impact tests confirm the latter, and the explosion temperature test value of pentolite is essentially that of PETN. The minimum detonating charges of lead azide and mercury fulminate required for pentolite are intermediate between those for PETN and TNT and

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*Applicable to pentolite manufactured by the coprecipitation method.

are very close to those for tetryl. On the whole, the sensitivity of pentolite may be considered to approximate that of tetryl rather than that of PETN. The presence of rust or grit increases the sensitivity of pentolite. A mixture of equal weights of pentolite and dry rust has an impact test value of 21 centimeters, as compared with a value of 32 centimeters for the pentolite used in making the mixture.

f. Sand tests show 50–50 pentolite to be as brisant as tetryl and 114 percent as brisant as TNT. Fragmentation tests of shell charges, however, show pentolite to be 131 percent as brisant as TNT and plate dent tests indicate the ratio to be 126 percent. The rate of detonation of cast pentolite is 108 percent that of TNT. Pentolite is 122 percent as powerful as TNT as measured by the Trauzl lead block test and 126 percent as powerful as measured by ballistic pendulum test, and the heat of explosion of pentolite is 132 percent that of TNT. In spite of this and its high rate of detonation, pentolite has much less blast effect than might be expected, its relative peak pressure and impulse values being 105 and 107 percent, respectively, those for TNT. Pentolite, therefore, may be considered an excellent fragmentation explosive that is not too suitable for use in producing blast effect.

g. Although 50–50 pentolite is slightly less stable than PETN, as judged by vacuum stability test values, it has been found to be of satisfactory stability and contain no excessive acidity after storage at 65° C. for 2 years. It withstands 10 remelting operations at approximately 98° C. without becoming unstable or unduly acid, but subsequent storage at 65° C. for 6 months renders it unduly acid. While storage at 65° C. has only limited effect on the chemical stability of pentolite, it undergoes some exudation if stored at temperatures greater than 50° C. This may be due to the formation of an eutectic mixture of TNT and impurities in PETN. One of the impurities present in PETN is dipentaerythritol hexanitrate, which melts at 73.6° C. and forms with TNT an eutectic mixture that freezes at only 57.9° C.

h. Because of the somewhat lower stability and greater sensitivity of PETN as compared with RDX, the corresponding relationship of pentolite to composition B, and the tendency

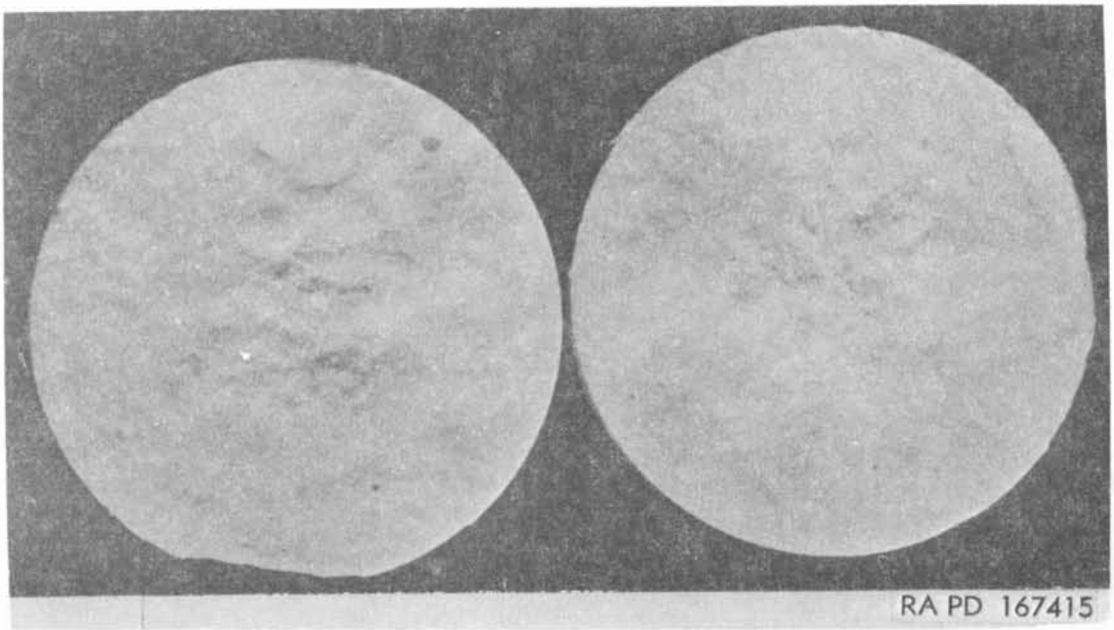

Figure 7–34. Cast 70–30 tetrytol, 1½ x.

of pentolite to undergo some exudation above 50° C., the present trend is toward the replacement of pentolite by composition B as well as that of PETN by RDX.

7-22. Tetrytol

a. During World War II, there was developed a binary bursting charge explosive consisting of 65 to 75 percent tetryl with TNT termed tetrytol. The 70-30 tetrytol (fig. 7-34) has been used in burster tubes of chemical shell; 75-25 tetrytol as a demolition explosive; and 65-35 tetrytol as a bursting charge for land mines.

b. TNT and tetryl form an eutectic mixture that freezes at 67.5° C., and tetrytol begins to melt at 68° C. When cast, it has a density of 1.60 to 1.61 and so permits the use of a slightly greater weight of charge in a fixed volume than TNT. It is only 0.02 percent hygroscopic when exposed to air of 90 percent relative humidity at 30° C. The solubility characteristics of tetrytol are essentially those of its ingredients and this is true also of its chemical reactivity.

c. Tetrytol is manufactured by heating TNT in a melting kettle equipped with an agitator until all the TNT is melted and the temperature of the liquid slightly exceeds 100° C. The proper amount of tetryl then is added, with continued stirring. Part of the tetryl dissolves in the liquid TNT. The temperature then is allowed to decrease until the mixture has thickened somewhat and is considered to be of maximum viscosity suitable for pouring in the melt-loading operation. It is then poured into the ammunition component or a mold. As cast, tetrytol is a mixture of tetryl and the TNT-tetryl eutectic mixture, which contains approximately 55 percent of tetryl.

d. As might be expected, tetrytol is intermediate between TNT and tetryl, with respect to sensitivity to impact, rifle bullet, heat, and initiation.

e. As judged by the sand test, 70-30 tetrytol is 97.5 percent and 111 percent as brisant as tetryl and TNT, respectively. Plate dent tests show it to be 117 percent as brisant as TNT and fragmentation tests of shell charges indicate the same relationship. The rate of detonation of tetrytol is only 106 percent that of TNT. As judged by the ballistic pendulum test, tetrytol is 120 percent as powerful as TNT.

f. 70-30 tetrytol is slightly less stable than tetryl at 100° C. and higher temperatures, as shown by vacuum stability tests. However, it is of satisfactory stability at 65° C. and lower temperatures, as indicated by storage at 65° C. for 2 years without change in stability, acid content, sensitivity, or brisance. Although tetryl undergoes partial decomposition on melting, the melting of tetrytol does not have the same effect. Tetrytol that had been melted and solidified 12 times showed no change in freezing point, sensitivity to impact, or 100° C. vacuum stability test value, although the temperature was each time raised to 107° C. and maintained at that value for half an hour prior to solidification by cooling. Storage at 65° C. causes some separation of an oily exudate and distortion of blocks of tetrytol, so the maximum temperature at which it should be stored is less than 65° C.

g. Determination of the composition of a sample of tetrytol can be made by treating a weighed sample with sufficient boiling carbon tetrachloride to dissolve the TNT present, cooling to 0° C., evaporating a weighed portion of the solution to dryness, and correcting for the amount of tetryl dissolved by the carbon tetrachloride at 0° C.

7-23. Picratol

a. Prior to World War II, armor-piercing projectiles were press-loaded with explosive D. This required the application of pressures of the order of 10,000 to 12,000 psi to successive increments of the charge and was a slow and expensive operation. The development of armor-piercing bombs necessitated the use of an insensitive explosive that could be melt-loaded; and for this reason, there was developed a composition containing 52 percent of explosive D and 48 percent of TNT and designated as picratol (fig. 7-35).

b. Molten TNT has little or no solvent action on explosive D, and consequently, cast picratol consists essentially of a physical mixture of

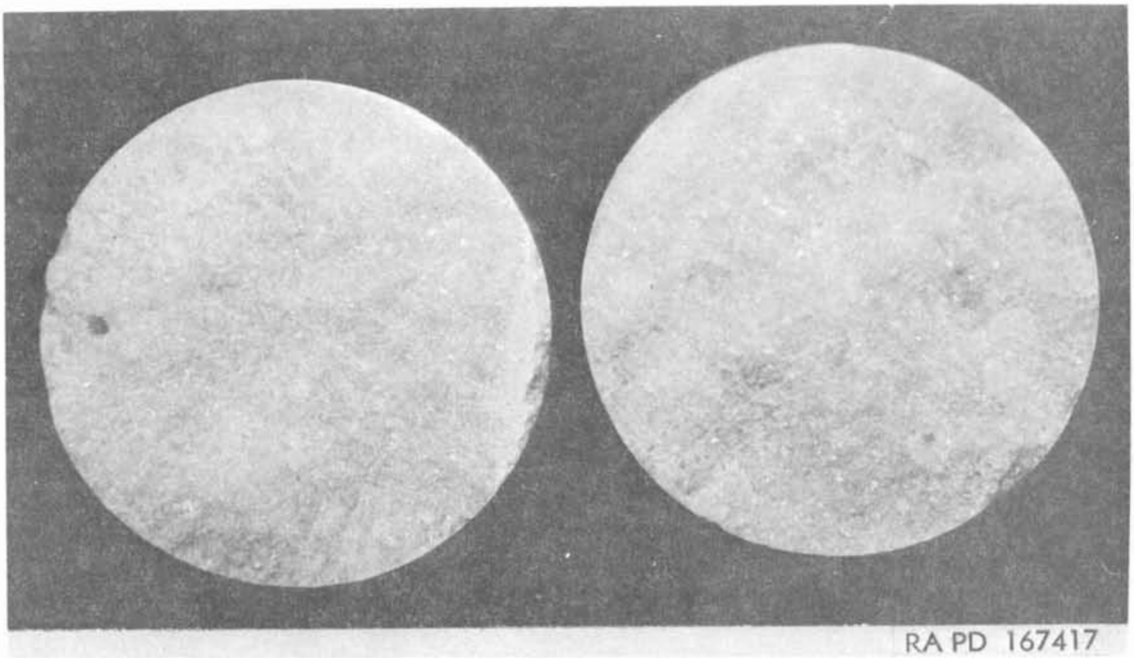

Figure 7–35. Cast 52–48 picratol, 1½ x.

crystals of the two explosives. Its density is 1.61 to 1.63 and this permits a weight of charge almost equal to that of explosive D pressed under 10,000 to 12,000 psi. Picratol has the solubility and reactivity characteristics of its ingredients. It is hygroscopic to the extent of only 0.02 percent when exposed to an atmosphere of 90 percent relative humidity at 30° C.

c. Picratol is manufactured by heating TNT to above 90° C. in a steam-jacketed melting kettle equipped with a stirrer. The explosive D is added slowly, without preheating, with continuous agitation; and this is continued for a short time after all the explosive D has been added. The thick slurry is allowed to cool to about 85° C. before being loaded into ammunition components. Like molten TNT, picratol undergoes marked contraction in volume on solidification; but this is much less than in the case of TNT.

d. The standard small scale impact tests indicate picratol to be as sensitive to impact as TNT, rather than explosive D, and large scale impact tests of cast charges confirm this. Pendulum friction tests show no difference between picratol, TNT, and explosive D, with respect to sensitivity to friction. When subjected to the rifle bullet impact test, picratol burns in about 40 percent of the trials but undergoes no detonation. Explosive D burns in about 30 percent of the trials. The explosive temperature test value of picratol (285° C.) is less than that of explosive D (318° C.) and much less than that of TNT (475° C.). Picratol is as insensitive to initiation as explosive D, having a minimum detonating charge value of 0.06 gram of tetryl. The overall sensitivity of picratol, therefore, is not greater than that of TNT.

e. 52–48 picratol is only 94 percent as brisant as TNT, as judged by the sand test, but the plate dent test indicates the two to be equally brisant, and the fragmentation of shell charges show picratol to be 102 percent as brisant as TNT. The rate of detonation of cast picratol is 101 percent that of cast TNT. Picratol and TNT are of equal power, as measured by the ballistic pendulum test, and have equal blast effects.

f. The stability of picratol is very slightly less than that of either TNT or explosive D, as measured by vacuum stability tests at 100°, 120°. and 150° C. These test results may be considered to indicate slight reaction between the explosive D and the molten TNT ingredients at the elevated temperatures of the tests. At ordinary temperatures, there is no evidence of such reaction and picratol appears to be of the same high order of stability as its ingredients.

g. The composition of picratol can be determined by extracting a weighed sample with cold ether that has been saturated with explosive D. The residue is dried and weighed and this and the loss in weight are calculated to percentage of explosive D and TNT, respectively.

7-24. Ednatol

a. Because of the excellent brisance of haleite and the impracticability of melt-loading this explosive, there was developed during World War II a composition containing 55 percent haleite and 45 percent TNT suitable for melt-loading and designated as ednatol (fig. 7-36). Although standardized, no ednatol has been loaded in ammunition by the end of the war. It was considered suitable for use in shell, bombs, and special ammunition components.

b. Cast 55-45 ednatol has a density of 1.62 or 1.63, which is 4 percent greater than that of cast TNT or haleite pressed under 30,000 psi. It is essentially nonhygroscopic when exposed to humid air.

c. Ednatol is manufactured by heating TNT to about 100° C. in a steam-jacketed melting kettle equipped with a stirrer. To the molten and stirred TNT, there is added slowly wet haleite. When addition of this is complete, heating and stirring are continued until all moisture has been driven off. The mixture then is cooled to about 85° C., with continued stirring, and loaded by pouring. Like TNT, ednatol undergoes some contraction in volume upon solidification.

d. The sensitivity of ednatol to impact is greater than that of TNT and less than that of haleite. The pendulum friction test indicates no greater sensitivity to friction than that of its ingredients. When subjected to the rifle bullet impact test, it does not detonate and undergoes only occasional ignition. Ednatol and haleite have the same explosion temperature test value (190° C.), which is lower than that of any other standard noninitiating high explosive. As might be expected, ednatol is more sensitive than TNT and less sensitive than haleite to initiation.

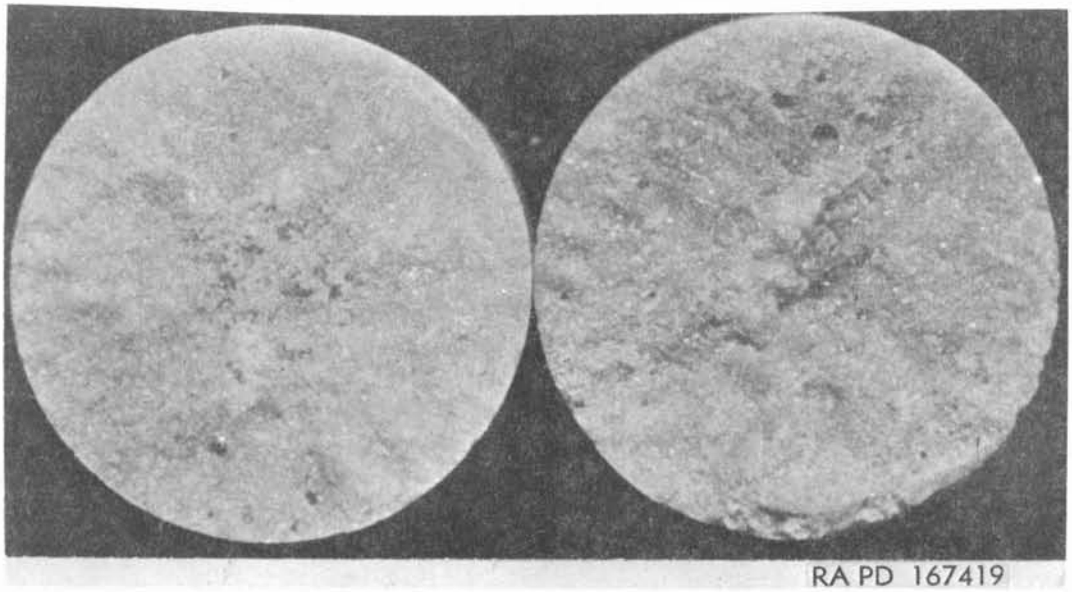

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Figure 7-36. Cast 55-45 ednatol, 1½ x.

e. Ednatol is 94 percent as brisant as haleite, as measured by the sand test, and 103 percent as brisant as TNT. The plate dent test indicates it to be more than 105 percent as brisant as TNT. However, fragmentation tests with shell charges are much more favorable, since they show ednatol to be 124 percent as effective as TNT. The rate of detonation of cast ednatol (7,340 meters per second) is 106 percent that of cast TNT. As measured by the ballistic pendulum and Trauzl lead block tests, ednatol is 119 and 120 percent, respectively, as powerful as TNT, but the blast effect of ednatol is only 108 to 110 percent as great as that of TNT. As an explosive for producing blast effect, it is, however, superior to amatol, pentolite, and composition C-3 and nearly equal to composition B.

f. At 100° C., ednatol is of the same order of stability as haleite, but it is much less stable than haleite at 120° C. Ednatol has been found to withstand storage at 65° C. for 2 years without change in stability, sensitivity to initiation, or brisance; and it does not undergo exudation at that temperature. Dry ednatol has no corrosive action on aluminum, stainless steel, cadmium, or nickel but causes very slight corrosion of copper, brass, mild steel, and zinc. In the presence of 0.5 percent of moisture, corrosion is much more pronounced and cadmium and nickel also are affected.

g. The composition of ednatol can be determined by extracting a weighed sample with cold ether saturated with haleite and drying and weighing the residue. The weight of residue and loss in weight are calculated to percent of haleite and TNT, respectively.

7-25. Composition B

a. Because of the availability of RDX in quantity and its superiority over TNT, with respect to brisance and power, the British developed composition B during the period between World Wars I and II and it was standardized by the United States early in World War II. Compositions consisting of RDX and TNT are designated as cyclotols, and composition B consists of 60-40 cyclotol, to which has been added a wax that has a desensitizing effect. Composition B, therefore, contains 60.0 percent RDX, 39.0 percent TNT, 1.0 percent desensitizer. It can be melt-loaded and is used extensively in artillery shells, in bombs and in land mines. In appearance, composition B is a brownish yellow solid when cast (fig. 7-37).

b. The density of cast composition B is 1.65.

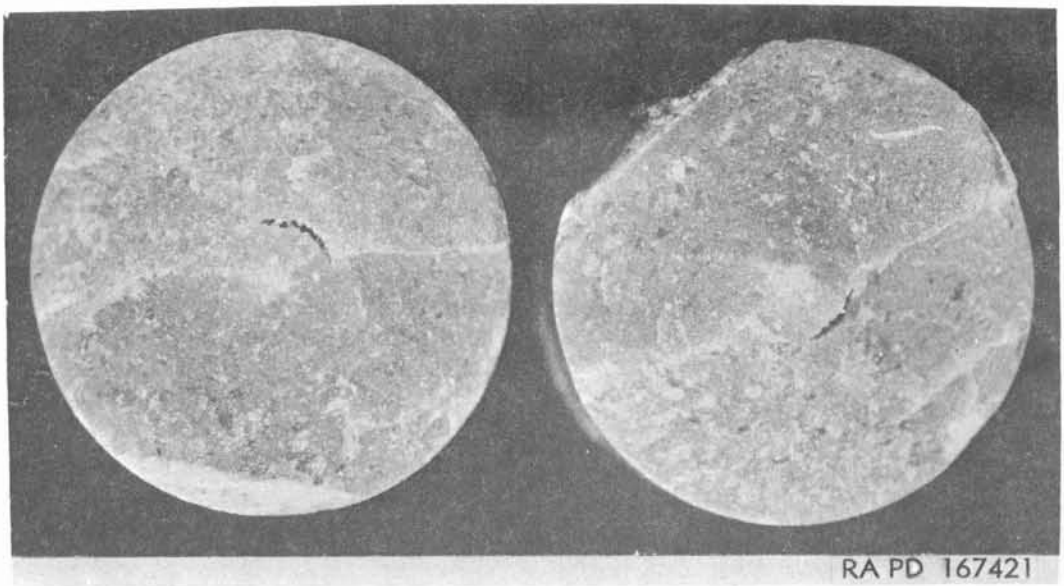


Figure 7-37. Cast composition B, 1½ x.

RDX is slightly soluble in molten TNT and they form an eutectic mixture that freezes at 79.0° C. and contains 95.84 percent TNT and 4.16 percent RDX. When heated, RDX undergoes some softening at a relatively low temperature, because of melting of the wax present and further melting at the eutectic temperature. As the temperature is increased, some of the RDX is dissolved, and at 100° C. there are in equilibrium 42.8 parts of molten solution and 57.2 parts of solid RDX. The viscosity of composition B as cast is an important characteristic that is affected markedly by the granulation of the RDX and the nature of the wax used, the wax causing increase in the viscosity. Representative viscosity values for composition B at 83° and 95° C. are 3.1 and 2.7 poises, respectively. The hygroscopicity of composition B, when exposed to air of 90 percent relative humidity at 30° C., is 0.02 percent.

c. Composition B is manufactured from TNT and water-wet RDX. The TNT is melted in a steam-jacketed melting kettle equipped with a stirrer and the temperature is brought to approximately 100° C. The damp RDX is added slowly with stirring, and after addition of the RDX is completed, most of the water is poured off and heating and agitation are continued until all moisture has been driven off. The wax is then added and mixed thoroughly with the other ingredients by stirring. The nature of the wax is important, since only certain waxes mix with the RDX and TNT and do not tend to segregate during cooling. The thoroughly mixed composition B then is cooled with continued agitation until it is of satisfactory fluidity for casting. It is then cast directly into bombs or so as to form chips approximately 0.75 inch square and 0.25 inch thick. The chips are produced when the composition B is to be stored or shipped for use elsewhere than the plant at which it is manufactured.

d. Solid composition B is slightly more sensitive than TNT but much less sensitive than RDX, as shown by impact tests. Rifle bullet impact tests show the same relationship. Cast composition B is distinctly more sensitive to impact than the powdered material. As judged by the pendulum friction test, powdered composition B is no more sensitive than TNT, and at 100° C., the two explosives show no increase in sensitivity to friction. Like TNT, cast composition B can be drilled without undue hazard. The explosion temperature test value of composition B (270° C.) is only slightly greater than that of RDX, but the sensitivity of composition B to electrical sparks is more nearly that of TNT than that of RDX. Composition B is intermediate between TNT and RDX, with respect to sensitivity to initiation. Cast composition B is somewhat less sensitive to initiation than the pressed material.

e. The brisance of composition B is 90 percent that of RDX, as measured by the sand test. The plate dent test shows composition B to be 129 to 132 percent as brisant as TNT, but the fragmentation of shell charges shows composition B to be 139 percent as brisant on an equal volume basis. This corresponds to 131 percent on an equal weight basis. The rate of detonation of cast composition B is only 113 percent that of cast TNT. Their ballistic pendulum, Trauzl lead block, and heat of explosion values indicate composition B to be 131 to 134 percent as powerful as TNT, but its blast effect is indicated to be only 110 percent that of TNT. Nevertheless, composition B is inferior only to tritonal and torpex, with respect to blast effect.

f. Vacuum stability tests indicate composition B to be as stable as RDX and only slightly less stable than TNT at 100° and 120° C. Storage at 75° C. for 1 month causes no decrease in stability, and it has been stored at 65° C. for over a year without change in acidity, sensitivity to impact, or brisance. However, 5 months of such storage causes slight exudation; and when composition B is made from TNT having a freezing point of 80.71° C., it undergoes very slight exudation when stored at 71° C. Composition B, therefore, is of a high order of chemical stability but should not be stored at too elevated temperatures because of physical instability at such temperatures. Composition B reacts slightly with rust at 100° C. At ordinary temperature, dry composition B causes very slight corrosion of copper and brass, but does not affect aluminum, mild steel, stainless steel, nickel, cadmium, or zinc. In the presence of 0.5 percent of moisture, composition B causes some corrosion of cadmium and zinc also.

g. Two grades of composition B are used for military purposes. Grade A is manufactured from class A RDX and grade B from class B RDX. The specification requirements for composition B are as follows:

Composition:

	Percent
RDX	60.0 ±2.0
TNT	39.0 ±2.3
Desensitizer	1.0 ±0.3

Moisture: Maximum, 0.25 percent.

Acidity: Grade A, maximum, 0.03 percent (as nitric acid).

Grade B, maximum, 0.02 percent (as acetic acid).

Viscosity: Grade A, efflux time, 5.0 ±2.0 seconds.

Grade B, efflux time, 12.0 ±5.0 seconds.

Insoluble particles: Retained on No. 60 sieve, maximum, 5 particles per 50 grams.

h. The RDX content of composition B can be determined by extracting a weighed sample with benzene saturated with RDX, drying the residue, and calculating the weight of this to percentage of RDX. The desensitizer content is determined by extracting another weighed sample with acetone cooled to 5° C., drying the residue, and calculating the weight of this to percentage of desensitizer. The percentage of TNT in the composition is obtained by subtracting from 100 the sum of the percentages of RDX, desensitizer, and moisture found present.

i. The cyclotol containing 60 percent RDX and 40 percent TNT is known also as composition B-2, but is not a standard explosive. Differing from composition B only in the absence of wax, it has a greater density when cast (1.70) and is slightly more brisant, as indicated by a sand test value of 55.0 grams as compared with 53.0 grams for composition B. The rate of detonation of cast composition B-2 is 7,900 meters per second, as compared with 7,800 meters per second for composition B. Composition B-2 is distinctly more sensitive than composition B, as judged by large scale impact and rifle bullet impact tests, and is slightly more sensitive to initiation. The two compositions are of the same stability and hygroscopicity. Because of its greater sensitivity to impact, composition B-2 is not as suitable as composition B for use in bombs.

j. A cyclotol with slightly varied percentage composition but no desensitizer has been designated Comp B3 as distinct from B-2. The newer composition B4 has the following ingredients:

RDX	60 ±2.0
TNT	39.5±2.0
Calcium Silicate	.5± .15

7-26. Torpex

a. Developed during World War II, a composition designated as torpex was standardized for use in bombs. It contains 41 percent RDX, 41 percent TNT, and 18 percent aluminum and is notable for its great blast effect. It is suitable for melt-loading and is a silvery-white solid when cast (fig. 7-38).

b. Torpex has a density of 1.81 when cast. At 15° C. it has a specific heat of 0.24 calorie per gram per degree Centigrade and thermal conductivity of 0.00094 calorie per second per centimeter per degree Centigrade. Its coefficient of thermal expansion at ordinary temperature is 0.00047 inch per inch per degree Centigrade. When the explosion undergoes solidification, there is a decrease in volume of 6 percent. It is nonhygroscopic when exposed to air of 90 percent relative humidity at 30° C. When heated, it undergoes partial melting near the melting point of TNT. The partially liquid explosive has viscosity values of 4.5 and 2.3 poises at 83° and 95° C., respectively. This is approximately the viscosity of glycerin at room temperature.

c. Torpex is manufactured by melting TNT in a steam-jacketed kettle equipped with a stirrer and heating the molten TNT to approximately 100° C. RDX slightly wet with water is added slowly and mixing and heating are continued until all water has been driven off. Grained aluminum is added and the mixture is stirred until uniformity is obtained. The mixture then is cooled, with continued stirring,

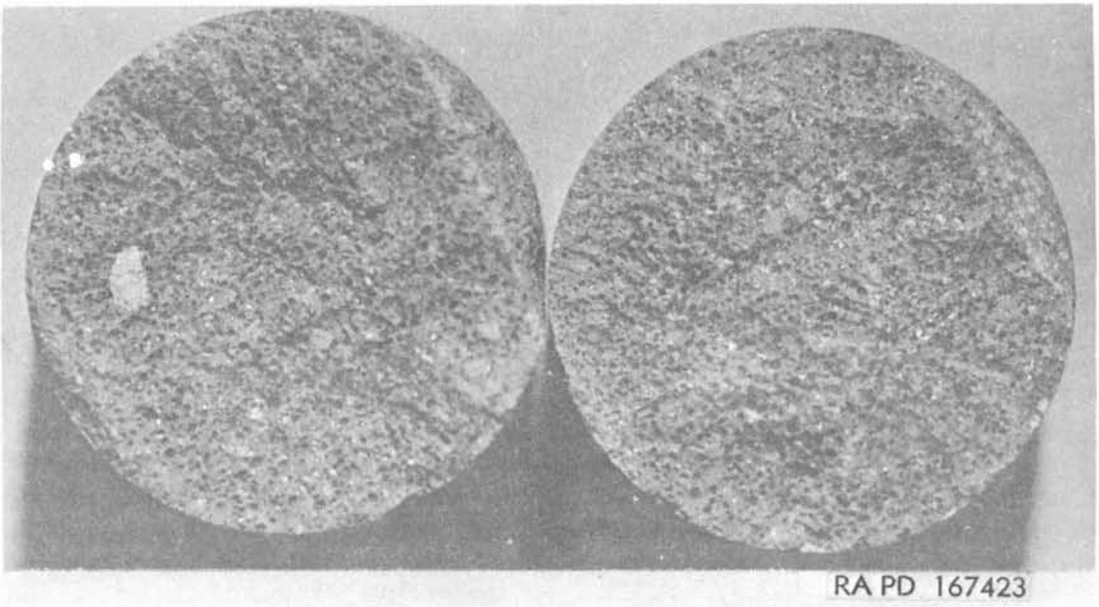


Figure 7-38. Cast torpex, 1½ x.

until its viscosity is such as to render it suitable for loading by pouring. Torpex of slightly different composition can be manufactured by melting 12.5 parts of TNT, adding 69.5 parts of composition B and, after further heating and stirring, adding 18 parts of aluminum. From this it will be recognized that torpex is essentially aluminized 50–50 cyclotol.

d. Torpex is considerably more sensitive to impact than composition B and undergoes partial or complete explosion in all trials in the rifle bullet impact test. Its explosion temperature test value (260° C.) is the same as that of RDX. Cast torpex is much more sensitive to initiation than is cast TNT, and pressed torpex is as sensitive as RDX to initiation by mercury fulminate.

e. Sand tests indicate torpex to be 98 to 99 percent as brisant as RDX and 122 percent as brisant as TNT. As judged by plate dent test values, cast torpex is 120 percent as brisant as TNT. Fragmentation tests of shell charges indicate torpex to be 126 percent as brisant as TNT and, therefore, inferior to composition B in this respect. The rate of detonation of cast torpex is 110 percent that of TNT. Trauzl lead block tests and their heats of explosion indicate torpex to be 161 to 162 percent as powerful as TNT, but the ballistic pendulum test gives torpex a superiority of only 34 percent. Torpex produces more blast effect than any other standard bursting charge explosive.

f. Vacuum stability tests show torpex to be of the same order of stability as composition B. Storage at 75° C. for 1 month has no effect on its stability, nor has storage at 65° C. for 13 months. It therefore is of a high order of stability. However, if all traces of moisture are not removed during manufacture, reaction between this and the ingredients of the composition results in the liberation of gas, which may cause rupture of the ammunition component or increase in the sensitivity of the torpex to shock.

g. The composition of torpex can be determined by extracting a weighed sample with cold benzene or toluene that has been saturated with RDX. The residue is dried and weighed and the loss in weight is calculated to percentage of TNT. The dried residue is extracted with hot acetone, dried, and weighed. The loss in weight is calculated to percentage of RDX. The weight of the final residue is calculated to percentage of aluminum.

h. Another modification of torpex is a composition designated as HBX–1. It can be manufactured from 70 parts composition B, 12 parts TNT, 18 parts aluminum, and 5 parts of a desensitizer known as composition D2. Composition D2 consists of 84 percent paraffin and other waxes, 14 percent nitrocellulose, and 2 percent lecithin. The composition of HBX–1 therefore is—

	Percent
RDX	39.6
TNT	37.8
Aluminum	17.1
Desensitizer	5.0
Calcium Chloride	0.5
Total	100.0

While slightly more viscous than torpex, molten HBX–1 is much less so than composition B and so can be loaded more easily. When cast, HBX–1 has a density intermediate between the density of composition B and that of torpex. It is less sensitive to impact and initiation than both torpex and composition B as shown by large scale impact, rifle bullet impact, and sand tests. HBX–1 is less brisant than torpex or composition B as judged by shell fragmentation and plate dent tests, and its rate of detonation is approximately 7,400 meters per second. The ballistic pendulum test shows HBX–1 to be as powerful as torpex and composition B, but it is intermediate between these, with respect to air blast and underwater shock values. As it contains no ammonium nitrate, HBX–1 does not have the objectionable hygroscopicity of DBX.

i. Recent modifications of torpex are the compositions HBX–3 and H6, developed essentially for high blast explosive applications. The compositions follow:

	HBX–3	*H–6*
RDX	31	45
TNT	29	30
Aluminum	35	20
D–2 Wax	5	5
Calcium Chloride	.5	.5

These compositions compare favorably with torpex and exhibit lower sensitivity to impact and much higher explosion test temperatures.

7–27. Composition A–3

a. During World War II, the British introduced the use of an explosive composition containing 91 percent RDX and 9 percent beeswax, which was highly brisant and suitable for press loading. This was designated as composition A. When standardized by the United States, the beeswax was replaced with wax derived from petroleum. Subsequent changes in the method of adding the desensitizer led to designation of the explosive as composition A–2. More recently, the composition has been designated as A–3, because of later changes in the granulation of RDX and the method of manufacture. The wax is so applied as to coat the particles of RDX and act as a binding agent when the composition is pressed. The color of the composition depends upon the particular desensitizing wax used in manufacture. Figures 7–39 and 7–40 show photographs of composition A–3.

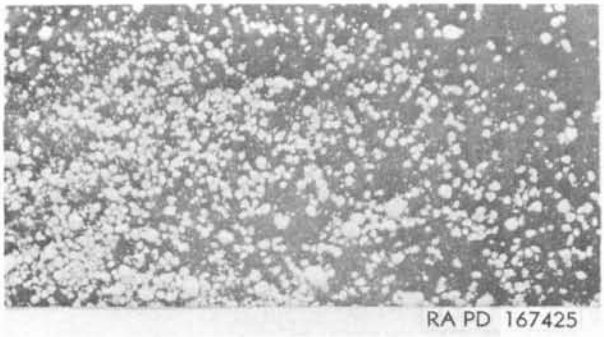


Figure 7–39. Composition A–3 granules, 1½ x.

b. Composition A–3 has density values of 1.46 and 1.63 when pressed at 3,000 and 12,000 psi, respectively. It is completely soluble in benzene and acetone when these are used successively.

c. The manufacture of composition A–3 is carried out by heating a water slurry of RDX to nearly 100° C. with agitation. The wax, which contains a wetting agent, is added with continued agitation and this is continued while the mixture is allowed to cool to a temperature less than the melting point of the wax. After being caught on a filter, the composition is air-dried at 77° C. (170° F.).

d. Composition A–3, of which there is but

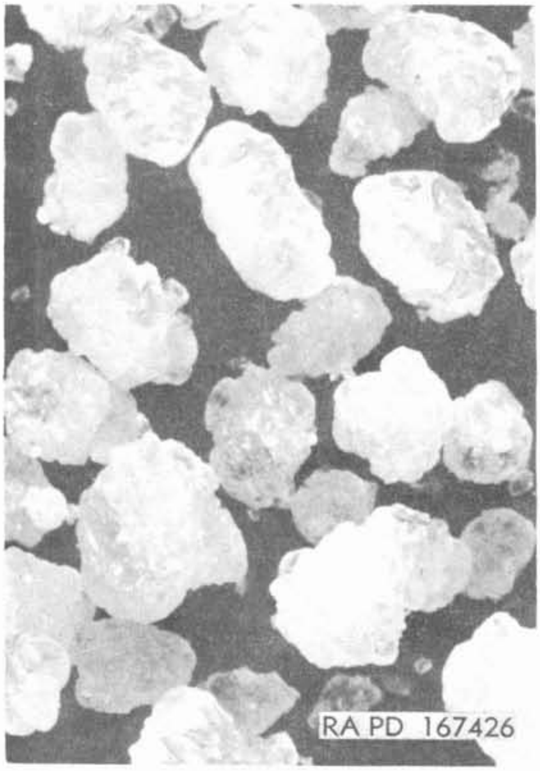


Figure 7-40. Composition A-3 granules, 25 x.

one grade, complies with the following requirements:

Composition:
RDX, 91.0±0.7 percent. Desensitizer, 9.0±0.7 percent.
Moisture: Maximum, 0.1 percent.
Acidity: Maximum, 0.045 percent.
Benzene-acetone insoluble:
Maximum, 0.15 percent.
Inorganic insoluble:
Maximum, 0.05 percent.
Grit:
Maximum particles per 50-gram sample
Retained on No. 40 sieve ____________ None.
Retained on No. 60 sieve ____________ 5.
Bulk density:
Minimum, 0.83 gram per milliliter.
Granulation:
Minimum through No. 6 sieve, 100 percent.
Maximum through No. 100 sieve, percent.
Consistency at 70° to 100° C.:
Soft and self-binding under light hand pressure.

e. Although composition A-3 has an explosion temperature test value of only 250° C., it is quite insensitive to shock and friction, being less sensitive than TNT. It is slightly more sensitive to initiation than TNT. This combination of sensitivity characteristics renders it suitable for use under conditions where it is subjected to high set-back forces.

f. As judged by sand test values, composition A-3 is 86 percent as brisant as RDX and 108 percent as brisant as TNT. Plate dent tests, however, indicate it to be 126 percent as brisant as TNT; and fragmentation tests of shell charges show it to be 50 percent more effective than TNT. Ballistic pendulum tests indicate composition A-3 to be 132 percent as powerful as TNT.

g. When subjected to vacuum stability tests at 100° and 120° C., composition A-3 liberates less gas than does RDX and only slightly more than that liberated by TNT. 100° C. heat test data for composition A-3 indicate it to undergo chiefly volatilization at that temperature. As it is nonhygroscopic, it is not affected by the absorption of moisture during storage. It should not be stored at temperatures above 75° C., because of the softening effect on the wax coating.

h. Compositions A4 and A5 have been developed consisting of 97.0 and 98.5 percent RDX respectively with desensitizer added but these explosives are not widely used in the field.

7-28. Compositions C-3 and C-4

a. During World War II, the British used a plastic demolition explosive that could be shaped by hand and had great shattering power. As standardized by the United States, it was designated as composition C and contained 88.3 percent RDX and 11.7 percent of a nonexplosive oily plasticizer. Included in the plasticizer was 0.6 percent lecithin, which helped to prevent the formation of large crystals of RDX which would increase the sensitivity of the composition. While plastic from 0° to 40° C., composition C became brittle and less sensitive below 0° C. and tended to become gummy and exude oil at temperatures above 40° C. It was replaced by composition C-2, which contained 80 percent RDX and 20 percent explosive plasticizer. This explosive plasticizer was composed of mononitrotoluene, a liquid mixture of dinitrotoluenes, TNT, nitrocellulose, and dimethylformamide. Composition C–2 was plastic from –30° to 52° C., but became less plastic in hot storage, because of evaporation of volatile matter. It has been replaced by composition C–3, which contains 77±2 percent RDX and 23±2 percent explosive plasticizer. The plasticizer contains mononitrotoluene, a liquid mixture of dinitrotoluenes, TNT, tetryl, and nitrocellulose. It is a yellowish puttylike solid that has a density of 1.60 and is soluble in acetone. Photographs of composition C–3 are shown in figures 7–41 and 7–42.

b. In the manufacture of composition C–3, the mixed plasticizing agent is placed in a steam-jacketed melting kettle equipped with a stirrer and heated to nearly 100° C. Water-wet RDX is added slowly, and heating and stirring are continued until a uniform mixture has been obtained and all the water has been driven off. The mixture then is cooled with continued agitation.

c. But one grade of composition C–3 is manufactured, this comprising two classes that differ with respect to acidity only. Class *A* material is used for the manufacture of demolition blocks, while class *B* explosive is used in demolition blocks or the loading of ammunition in which lower acidity is desirable. The requirements applicable to composition C–3 are—

Composition:

RDX plus nitrocellulose, 78.0±2.0 percent.
Plasticizer minus nitrocellulose, 22.0±2.0 percent.

Moisture: Maximum, 0.25 percent.

Acetone insoluble material:
Maximum, 0.15 percent.

Inorganic acetone insoluble material:
Maximum, 0.05 percent.

Grit:

Maximum particles per 50-gram sample

Retained on No. 40 sieve	None.
Retained on No. 60 sieve	5.

Plasticity:
Minimum extension in length at 25° to 30° C., 90 percent.

Acidity:

	Percent
Class *A*.—Maximum	0.064
Class *B*.—Maximum	0.05

d. Composition C–3 is of the same sensi-

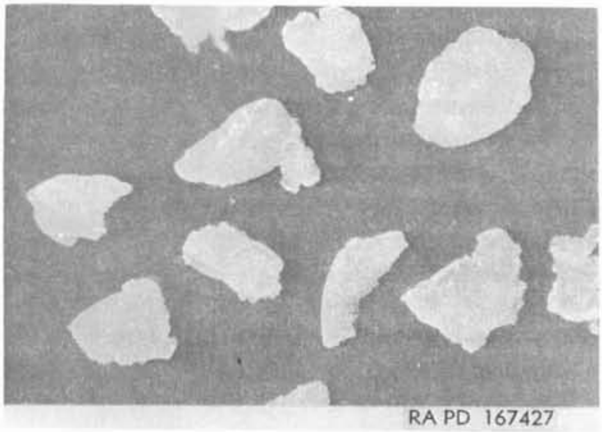


Figure 7–41. Composition C–3 granules, 1½ x.

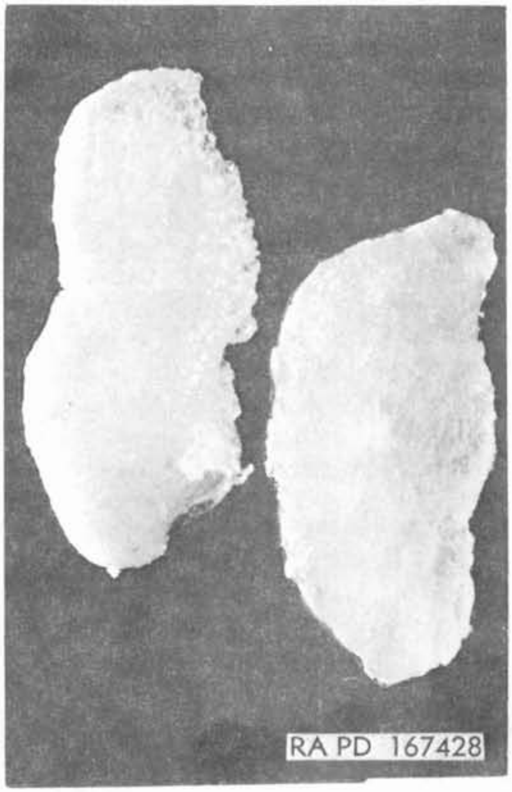


Figure 7–42. Composition C–3 granules, 6 x.

tivity to impact as TNT and is not exploded in the pendulum friction test. In the rifle bullet impact test, it undergoes partial explosion in 40 percent of the trials, which indicates greater sensitivity than that of TNT but much less sensitivity than that of RDX. It is less sensitive to initiation than TNT, requiring a minimum detonating charge of 0.08 gram of tetryl in the sand test. Storage for 4 months at 65° C. in an atmosphere of 95 percent relative humidity does not impair its sensitivity to initiation. The explosion temperature test value of composition C–3 (280° C.) is only slightly greater than that of RDX (260° C.).

e. As judged by sand test values, composition C–3 is 90 percent as brisant as RDX and 112 percent as brisant as TNT. The plate dent test indicates it to be 114 percent as brisant as TNT, but fragmentation of shell charges shows composition C–3 to be 133 percent as effective as TNT. The rate of detonation of composition C–3 (7,625 meters per second) is 111 percent that of cast TNT. The Trauzl lead block and ballistic pendulum tests indicate composition C–3 to be 115 and 126 percent, respectively, as powerful as TNT, but it is slightly inferior to composition B as an explosive for producing blast effect.

f. The chemical stability of composition C–3 is acceptable at least, but its physical stability is not entirely satisfactory. Its volatility is such that it loses 1.2 percent by weight when exposed to air at 25° C. for 5 days. It is hygroscopic to the extent of 2.4 percent when exposed to air of 90 percent relative humidity at 30° C., but tests have shown composition C–3 to be of unimpaired brisance after immersion in water for 24 hours. When stored at 77° C., it undergoes considerable exudation. It becomes hard at −29° C. (−20° F.).

g. Composition C–3 is not unduly toxic, but since it contains aryl nitrocompounds, it should be handled with the same precautions as tetryl and similar compounds.

h. Because of the hardening of composition C–3 at −29° C., its volatility, and its hygroscopicity, there has recently been developed and standardized an improved plastic explosive designated as composition C–4. This contains the following ingredients:

	Percent
RDX	91.00
Polyisobutylene	2.10
Motor oil	1.60
Di-(2-ethylhexyl) sebacate	5.30
Total	100.00

It is a puttylike material of dirty white to light brown color that has found application in demolition blocks and specialized uses.

i. Only one grade of composition C–4 is used for military purposes, although di-(2-ethylhexyl) adipate may be substituted for di-(2-ethylhexyl) sebacate in the composition. The method of manufacture is classified as confidential.

j. Composition C–4 has no odor and has practically the same density (1.59) as composition C–3. It does not harden at −57° C. (−70° F.) and does not undergo exudation when stored at 77° C. (170° F.).

k. Composition C–4 is less sensitive to impact than composition C–3, as indicated by impact and rifle bullet impact tests, but they are of the same order of sensitivity to initiation and composition C–4 can be detonated by a No. 6 blasting cap. It is not affected in the pendulum friction test.

l. Composition C–4 is slightly more brisant than composition C–3, as shown by sand and plate dent tests and indicated by its higher rate of detonation (8,040 meters per second). The ballistic pendulum test shows composition C–4 to be slightly more powerful than composition C–3.

m. The stability of composition C–4 is greater than that of composition C–3, as indicated by 100° C. vacuum stability tests, and 100° C. heat tests show composition C–4 to be of a high order of stability and much less volatile than composition C–3.

n. Composition C–4 is essentially nonhygroscopic.

Warning: **Although C–4 contains no aryl nitrocompounds and lacks the toxicity characteristics of C–3, composition C–4 is poisonous and dangerous if chewed or ingested.**

o. Composition C–4 has not replaced composition C–3 entirely; both are standard explosives at the present time.

7-29. Dynamites

a. Military operations frequently necessitate excavation, demolition, and cratering operations, for which the standard high explosives are unsuited, and recourse is made to commercial and special compositions. Commercial blasting explosives, with the exception of black powder, are referred to as dynamites, although in some cases they contain no nitroglycerin.

b. Nobel gave the name dynamite to mixtures of nitroglycerin and kieselguhr, the strength of the dynamite being indicated by the percentage of nitroglycerin in the mixture. The porous kieselguhr was chemically inert and absorbed the nitroglycerin, rendering it much safer to handle. Later, it was found that even stronger dynamites could be made by substituting sodium nitrate and a combustible absorbent, such as wood pulp, for the kieselguhr. The resulting composition was called an active dope dynamite. Subsequently, the replacement of part of the nitroglycerin and sodium nitrate by ammonium nitrate brought into existence the less costly *ammonia dynamites* that are notable for their great heaving rather than shattering effects. The replacement of nitroglycerin in dynamite by nitrostarch resulted in the development of a class of dynamites free from some of the objectionable characteristics of nitroglycerin dynamites. While the active dope and ammonia dynamites are hygroscopic and desensitized by water, necessitating a moistureproof wrapper, a waterproof composition termed *blasting gelatin* is obtained by colloiding nitrocellulose with nitroglycerin. When mixtures are made of 88 to 92 percent of nitroglycerin and 8 to 12 percent of nitrocellulose having a nitrogen content of about 12 percent, a relatively tough material is formed. Blasting gelatins are particularly adapted to deep-water blasting. By including some nitrocellulose in the compositions of active dope dynamites, the so-called *gelatin dynamites* were formulated. These possess greater density and water resistance than straight active dope dynamites and so are more suitable for use under wet conditions.

c. The cost of glycerin and the tendency of nitroglycerin to freeze at some atmospheric temperatures prompted the partial replacement of nitroglycerin by antifreeze materials, such as nitrated diglycerin, sugars, and glycols. Antiacid materials, such as calcium carbonate or zinc oxide, have been added to most dynamite compositions to neutralize any acidity developed during storage. The inclusion of special purpose ingredients, such as sulfur, ferrosilicon, alum, nitrotoluenes, sodium chloride, copper sulfate, etc., and the substitution of starch, ground peanut hulls, vegetable ivory, etc., for wood pulp have further increased the complexity of dynamite compositions. Usually, they are formulated so as to have certain rate of detonation and heaving force values that render them suitable for various types of blasting operations. At the same time care must be taken that the compositions are oxygen balanced when the paraffined paper wrapper is taken into consideration. Compositions overbalanced with respect to oxygen produce nitrogen oxides on detonation; while underbalanced compositions produce some carbon monoxide instead of carbon dioxide. While many of the commercial dynamites are given strength designations on a percentage basis, this no longer means that they contain that percentage of nitroglycerin or have the same characteristics as a mixture of kieselguhr and nitroglycerin containing that percentage of nitroglycerin. Representative compositions and characteristics of the various types of dynamite are given in table 7-20.

Table 7-20. Compositions[1] *and Characteristics of Dynamites*

Strength of dynamite, percent	20	30	40	50	60	100
Straight dynamites:						
Nitroglycerin	20.2	29.0	39.0	49.0	56.8	
Sodium nitrate	59.3	53.3	45.5	34.4	22.6	
Carbonaceous fuel	15.4	13.7	13.8	14.6	18.2	
Sulfur	2.9	2.0				

Strength of dynamite, percent	20	30	40	50	60	100
Antacid	1.3	1.0	0.8	1.1	1.2	
Moisture	0.9	1.0	0.9	0.9	1.2	
Rate of detonation, m/sec	------	------	4,400	5,200	5,900	
Ballistic pendulum, percent TNT	------	------	94.5	102.5	114	
Ammonia dynamites:						
Nitroglycerin	12.0	12.6	16.5	16.7	22.5	
Sodium nitrate	57.3	46.2	37.5	25.1	15.2	
Ammonium nitrate	11.8	25.1	31.4	43.1	50.3	
Carbonaceous fuel	10.2	8.8	9.2	10.0	8.6	
Sulfur	6.7	5.4	3.6	3.4	1.6	
Antacid	1.2	1.1	1.1	0.8	1.1	
Moisture	0.8	0.8	0.7	0.9	0.7	
Rate of detonation, m/sec	2,700	------	3,300	3,900	4,600	
Ballistic pendulum, percent TNT	81	------	91	99	109	
Gelatin dynamites:						
Nitroglycerin	20.2	25.4	32.0	40.1	49.6	91.0[2]
Sodium nitrate	60.3	56.4	51.8	45.6	38.9	
Nitrocellulose	0.4	0.5	0.7	0.8	1.2	7.9
Carbonaceous fuel	8.5	9.4	11.2	10.0	8.3	
Sulfur	8.2	6.1	2.2	1.3		
Antacid	1.5	1.2	1.2	1.2	1.1	0.9
Moisture	0.9	1.0	0.9	1.0	0.9	0.2
Rate of detonation, m/sec	4,000	4,600	5,200	5,600	6,200	
Ballistic pendulum, percent TNT	74	79	84.5	90.5	99	
Ammonia gelatin dynamites:						
Nitroglycerin	------	22.9	26.2	29.9	35.3	
Sodium nitrate	------	54.9	49.6	32.0	33.5	
Ammonium nitrate	------	4.2	8.0	13.0	20.1	
Nitrocellulose	------	0.3	0.4	0.4	0.7	
Carbonaceous fuel	------	8.3	8.0	8.0	7.9	
Sulfur	------	7.2	5.6	3.4		
Antacid	------	0.7	0.8	0.7	0.8	
Moisture	------	1.5	1.4	1.6	1.7	
Rate of detonation, m/sec	------	4,400	4,900	5,300	5,700	
Ballistic pendulum, percent TNT	------	83	88	92	97.5	

[1] Percent by weight.

[2] Blasting gelatin.

d. In addition to military dynamite (*h* below), dynamites of the types described may be used for military operations, and are procured as commercial items. A composition having great heaving force and relatively low rate of detonation is preferable for blasting soft rock or earth, while a gelatin dynamite of low heaving force and high rate of detonation is used for hard tough rock.

e. A special demolition explosive standardized shortly before World War II is based on nitrostarch instead of nitroglycerin. It is pressed into ¼-pound pellets, which are covered with paper, and 1-pound packages of the pellets are wrapped in paper with markings indicating the location of holes for blasting caps in the pellets. The composition used is—

	Percent
Nitrostarch	34.5 ± 2.5
Barium nitrate	43.5 ± 1.5
TNT	15.0 ± 2.0
Aluminum	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 barium nitrate is coated with the paraffin before the ingredients are mixed together and pressed into pellets. The composition is required to undergo no ignition or explosion when heated at 100° C. for 48 hours and to have a density of 1.75±0.10 when pressed.

f. Nitrostarch demolition explosive is sensitive with respect to crumbling or breaking action. It does not explode in the pendulum friction test, but does so in the rifle bullet impact test. It is more sensitive to initiation than TNT. The sand test indicates it to be 90 percent as brisant as TNT and the ballistic pendulum test shows it to be 96 percent as powerful as TNT. Although it is hygroscopic to the extent of 2.1 percent when exposed to air of 90 percent relative humidity at 30° C., nitrostarch demolition explosive is of satisfactory stability as packed, as shown by long-term storage tests. Its 100° C. heat test values indicate it to undergo some volatilization, rather than decomposition, in 96 hours at that temperature. In the 75° C. international test, it loses 1 percent of its weight but shows no signs of deterioration.

g. During World War II, there was used a cratering explosive having the composition

	Percent
Ammonium nitrate	86.6
Dinitrotoluene	7.6
Ferrosilicon	5.7
Red dye	0.1

This inexpensive and easily manufactured explosive is very insensitive to shock and initiation, has a low rate of detonation but high heaving force value, and is very stable. As it is somewhat hygroscopic in spite of the moistureproofing action of the dinitrotoluene, the explosive is packed in hermetically sealed containers.

h. Military dynamite M1, M2 and M3 is a medium velocity (20,000 feet per second) blasting explosive in three cartridge sizes. Low velocity (24,000 feet per second) military dynamites are under development. Military dynamite M1, M2, and M3 has been standardized for use in military construction, quarrying, and service demolition work. The explosive composition is packaged in standard dynamite cartridge waxed-paper wrappers. The models differ only in the cartridge size. Cartridges are 1¼ inches in diameter by 8 inches long for the M1, 1½ inches in diameter by 8 inches long for the M2, and 1½ inches in diameter by 12 inches long for the M3. The composition used is—

	Percent
RDX	75 ± 1.0
TNT	15 ± 0.5
Grade SAE No. 10 engine oil plus polyisobutylene.	5 ± 0.5
Cornstarch	5 ± 0.5

Desensitized RDX, which is coated with engine oil before mixing with the other ingredients, and grained TNT are used in the manufacture of the dynamite. Military dynamite M1, M2, and M3 is equivalent in strength to 60 percent commercial dynamite. The military dynamite is safer to transport, store, and handle than 60 percent straight nitroglycerine commercial dynamite and is relatively insensitive to friction, drop impact, and rifle bullet impact. The composition remains plastic at minus 70° F. after 24 hours. It is odorless, free from nitroglycerine toxicity, nonhygroscopic and chemically stable at 160° F. and 80 percent relative humidity for more than 1 month. It will not freeze in cold storage nor exude in hot storage. Turning of shipping containers in storage is not necessary.

CHAPTER 8

FOREIGN HIGH EXPLOSIVES

8-1. General

a. Broadly, the high explosives used by other nations have been the same as, or very similar to, those used by the United States. During World War II, problems of logistics necessitated some use of substitutes by the Germans and Italians, in order to achieve maximum production of explosives, and the use of a disproportionately large amount of the less desirable explosives by the Japanese. Examples are the use of hexanitrodiphenylamine by the Germans and picric acid instead of TNT by the Japanese.

b. The earlier development in Germany of explosives, such as lead styphnate and tetracene, lead to the use of priming and detonating compositions containing these materials, as well as lead azide and mercury fulminate. As in the United States, it was not found practicable to eliminate mercury fulminate entirely.

c. World War II saw the use by the United States and other nations of binary explosives in which TNT was used as a dispersing agent for more powerful, less fusible explosives, rather than compositions in which TNT was the more powerful ingredient of an explosive mixture. Again, problems of logistics sometimes prevented full realization of this policy and dictated the use of binary explosives such as amatol and hexanite. The use of aluminized explosives similar to tritonal and torpex was general in other countries as well as the United States, and in some cases, the trend abroad in this direction was even more pronounced.

8-2. Initial Detonating Agents

a. Although lead azide was the most important initiating agent used by other nations, its high ignition temperature and insensitivity to initiation by percussion necessitated the use of more sensitive materials, as cover charges, in mixtures, and as ingredients of priming compositions.

b. The Germans used lead styphnate as a cover charge for lead azide and, in some cases, mixed it with lead azide. Such mixtures contained 20 to 35 percent of lead styphnate. A mixture containing 95 percent lead azide and 5 percent tetracene was used to a limited extent. A PETN-lead azide mixture, containing 30 percent PETN and a PETN-lead styphnate-lead azide mixture containing 10 percent PETN and 30 percent lead styphnate, were also used. While PETN was the usual base charge for detonators, waxed RDX, a TNT-tetryl mixture, and pentolite also were used for this purpose.

c. Japanese initiating agents were similar to those used by the Germans, except that tetracene was not used and fewer mixtures were employed. Mercury fulminate was used in a few cases. While PETN was used extensively as a base charge for detonators, tetryl also was used largely for this purpose.

d. The Italians used lead styphnate as a cover charge for lead azide and admixture of the two. Mercury fulminate was used alone to some extent. Silver azide was used as an initiator to a slight extent. RDX was used more extensively than PETN as the base charge for detonators.

8-3. Priming Compositions

a. The Germans did not use lead azide in priming compositions. The majority of the compositions used were of the mercury fulminate-potassium chlorate-antimony sulfide-abrasive type. Their most stable compositions contained lead styphnate as the explosive ingredient and calcium silicide as the abrasive material. Basic lead picrate, coated with lacquer, was used instead of lead styphnate to a certain extent as an electrically ignited priming composition. Compositions containing no explosive ingredient also were used in German ammunition. Representative German priming compositions of the several types are shown by table 8-1.

Table 8-1. German Priming Compositions

	Percent					
Lead styphnate	88	25	35	35		
Potassium chlorate	----	45	----	----	55	55
Barium nitrate	----	----	45	45		
Calcium silicide	----	30	20	15	14	
Antimony sulfide	----	----	----	5	30	28
Nitrocellulose lacquer	12					
Carbon	----	----	----	----	----	10.5
Abrasive	----	----	----	----	----	5
Binder	----	----	----	----	1	1.5
Total	100	100	100	100	100	100

b. Like the Germans, the Japanese and Italians used chiefly priming compositions that contained mercury fulminate. They do not appear to have used compositions similar to the German lead styphnate and nonexplosive ingredient compositions.

8-4. Noninitiating High Explosives

a. Until production difficulties during the last part of World War II required the use of inferior substitutes, such as dinitrobenzene and ethylenediamine dinitrate, the Germans used the following high explosives:

TNT
RDX (Hexogen)
PETN (Penta)
Picric acid
Hexite (hexanitrodiphenylamine)
Textryl
Ammonium nitrate (in amatols)

TNT and amatol were used in the largest amounts in artillery and mortar shell, bombs, grenades, mines, and demolition charges. In armor-piercing or concrete-piercing shell, the TNT in some cases was desensitized by the addition of 3 to 10 percent of wax. The amatols used in shell and bombs contained from 30 to 60 percent, while that used in grenades contained 80 percent of ammonium nitrate. A mixture of equal weights of TNT and RDX, desensitized with 1 to 3 percent wax and corresponding to composition B, was used in shell. RDX, desensitized with 5 to 10 percent wax, was used in shell and rockets. A pentolite, containing 30 percent PETN and PETN desensitized with 3 to 20 percent wax, was used to a lesser extent in shell. Mixtures of powdered aluminum with TNT, amatol, and TNT-RDX were used for loading bombs and torpedo warheads. Trialen No. 105 was a mixture containing 70 percent TNT, 15 percent RDX, and 15 percent aluminum. In a few cases, powdered zinc was used instead of aluminum. A mixture of hexanitrodiphenylamine (hexite), TNT, and aluminum was used extensively as a charge for torpedo warheads; while hexanite, a mixture containing 40 percent hexite and 60 percent TNT, was used in bombs. Less wide application was made of trinitrochlorbenzene in shell and a bomb charge consisting of 90 percent ammonium nitrate, 6 percent carbon, and 4 percent mineral matter. Waxed PETN and waxed RDX were the booster charges chiefly used, but picric acid, tetryl, TNT, and 40/60 tetrytol also were used as booster charges. The whole pattern of the use of high explosives by the Germans during World War II reveals their limitations in the production of individual explosives and the necessity for producing as many different explosives and applying each as widely as practicable.

b. During World War II, the Japanese suffered from the same difficulties in production as the Germans and used a correspondingly wide range of high explosives. Their basic standard explosives were—

TNT	Chakatsuyaku or Type 92
Tetryl	Meiayaku
Picric acid	Oshokayaku or Shimose bakuyaku
RDX	Tan-o-yaku or Shouyaku
PETN	Shoeiyaku
Hexanitrodiphenylamine	
Trinitroanisole	Type 91
Trinitrophenetole	Keineyaku
Ammonium nitrate	

These were used as widely as practicable, in spite of the vesicant effect of hexanitrodiphenylamine and the toxicity of trinitroanisole. Examples of compositions used by the Japanese are given in table 8-2.

Table 8–2. Japanese Explosives

Name	*Composition*	*Used in—*
Chanayaku	70 percent TNT 30 percent Dinitronaphthalene	Projectiles
Chaoyaku	25 percent TNT 75 percent Picric acid	Bombs
Nigotanyaku	50–70 percent TNT 30–50 percent RDX	Bombs, projectiles, land mines
Shotoyaku	50 percent TNT 50 percent Ammonium nitrate	Bombs, projectiles
Seigatta or Type 97	60 percent TNT 40 percent Hexanitrodiphenylamine	Torpedo warheads, depth charges
Otsu-B	60 percent TNT 24 percent Hexanitrodiphenylamine 16 percent Aluminum powder	Torpedo warheads, mines, depth charges.
Pentoriru	50 percent TNT 50 percent PETN	Machinegun bullets
Type 92	66 percent TNT 34 percent Aluminum powder	Machinegun bullets
Onayaku	50 percent Picric acid 50 percent Dinitronaphthalene	Projectiles
Oshiyaku	90 percent Picric acid 10 percent Wax	Projectiles
Angayaku	25 percent RDX 75 percent Ammonium nitrate	Bombs
	85 percent RDX 15 percent Wax	Armor-piercing projectiles
	42 percent RDX 50 percent PETN 8 percent Wax	Machinegun bullets
Haishokuyaku	17 percent RDX 77 percent Ammonium perchlorate 1.5 percent Silicon carbide 4.5 percent Paraffin	Demolition blocks
Oshitsuyaku	80 percent RDX 20 percent Vegetable oil	Demolition rolls
Type 94	40 percent RDX 60 percent Trinitroanisole	Torpedo warheads
H2Kongo or Type 98	70 percent Trinitroanisole 30 percent Hexanitrodiphenylamine	Bomb auxiliary boosters, bombs, sea mines, depth charges.
Shonoyaku	90 percent Ammonium nitrate 10 percent Dinitronaphthalene	Projectiles
Shoan bakuyaku	79 percent Ammonium nitrate 10 percent Dinitronaphthalene 10 percent Sodium chloride 1 percent Sawdust	Demolition charges
Ammon yaku	Ammonium nitrate Charcoal	
Type 1	81 percent Ammonium picrate 16 percent Aluminum powder 2 percent Wood pulp 1 percent Petroleum	Depth charges
Karitto	66 percent Ammonium perchlorate 16 percent Silicon carbide 12 percent Wood pulp 6 percent Petroleum	Demolition charges, mines, depth charges.

Table 8-2. Japanese Explosives—Continued

Name	*Composition*	*Used in—*
Haensoson-bakuyaku or Type 88	75 percent Ammonium perchlorate 16 percent Ferrosilicon 6 percent Wood meal 3 percent Petroleum	
Entoyaku	80 percent Potassium chlorate 16 percent Dinitrotoluene 4 percent Castor beal oil	Demolition charges, grenades, mortar projectiles.
Ennayaku	80 percent Potassium chlorate 15 percent Mononitrotoluene 5 percent Castor bean oil	

The number of markedly different compositions used for a single purpose indicates the necessity under which the Japanese labored to utilize everything available having explosive value. Technical investigation after World War II indicated that the Japanese explosives industry made no significant advances during the war period.

c. The military economy of Italy, because of the lack of coal deposits, was built around its chemical rather than its coal tar industry. In the period prior to World War II, PETN and RDX (designated at T-4) were developed on this basis as major military explosives. While TNT was used as extensively as possible, its limited availability necessitated the maximum use of other explosives. DEGN (diethyleneglycol dinitrate) was used in high explosives as well as propellants. An example of this is the Italian plastic explosive consisting of—

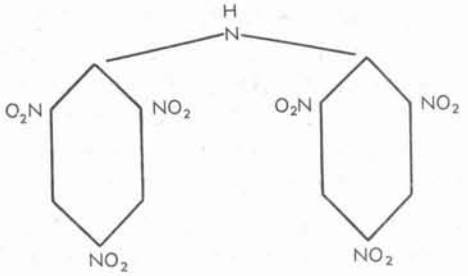
	Percent
RDX	78.5
DEGN	*17.5
Petroleum jelly	4
Total	100

A mixture containing 60 percent picric acid and 40 percent TNT designated as MAT, a modified tridite composition designated as MBT, and mixtures containing trinitronaphthalene were used for loading artillery projectiles.

8-5. Explosives of Special Interest

a. Hexanitrodiphenylamine (*Hexite*).

(1) This compound, can be obtained by the nitration of diphenylamine, but a more practical process involves the condensation of aniline with 2,4-dinitrochlorbenzene to form 2,4-dinitrodiphenylamine and nitration of the condensation product to the hexanitro compound. The availability of this explosive from benzene derivatives has made it of interest when limitation of the toluene supply results in a shortage of TNT.



RA PD 212853

Figure V. Hexite.

(2) The pure compoound forms yellow needles, which melt with decomposition at 243.5° to 244.5°C., are readily soluble in acetone and warm acetic acid, fairly soluble in ethanol, sparingly soluble in ether, and insoluble in water or chloroform. As ordinarily manufactured, it is brownish yellow and melts at 240° to 245°C. The crystal density of the compound is 1.653 and its heat of combustion at constant pressure is 3,001 calories per gram. Chemically the compound acts as a pseudo acid. It is soluble in

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*Contains 0.3 to 0.4 percent of collodion nitrocellulose.

aqueous alkalies, except potassium hydroxide, to form metallic salts. The potassium salt is insoluble in water. Hexite is a powerful vesicant and injurious to the mucous membranes.

(3) Hexite (hexanitrodiphenylamine) is more like tetryl than TNT, with respect to sensitivity and brisance, intermediate between these with respect to power, and more like TNT than tetryl, with respect to stability.

b. Hexanite. Hexanite, called Novit by the Germans, is a mixture containing 60 percent TNT and 40 percent of hexite. It is slightly superior to TNT with respect to brisance and power, and comparable with TNT with respect to sensitivity and stability. The only significant advantage offered by hexanite is the lessened demand for TNT resulting from its use.

c. Schiesswolle 18 (*TSMV 1–101*). Aluminized hexanite, called Schiesswolle 18 and TSMV 1–101 by the Germans, is a mixture containing 60 percent TNT, 24 percent hexite, and 16 percent powdered aluminum. As it has a blast effect greater than that of tritonal, it was used extensively in loading torpedo warheads.

d. Minol. Minol is a castable mixture containing 40 percent TNT, 40 percent ammonium nitrate, and 20 percent powdered aluminum. It may be considered to 50–50 amatol, to which has been added 25 percent of aluminum; and it therefore offers the advantage of ready availability with relatively small demand on the supply of TNT. It is as sensitive to initiation as TNT or tritonal, but more sensitive to shock. With a rate of detonation of only 6,000 meters per second at a density of 1.70, it is distinctly less brisant than tritonal, or TNT. Ballistic mortar tests show minol to be considerably more powerful than tritonal or TNT, and this is evident also from its greater blast and shock effects. While the thermal stability of minol is comparable with that of tritonal and amatol, the presence of moisture causes reaction between its ammonium nitrate and aluminum, with the liberation of gaseous products of reaction.

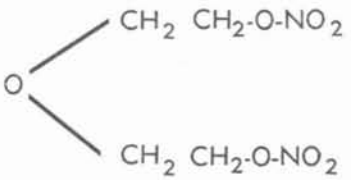
e. Ammonal. Ammonal is a castable composition, containing 67 percent TNT, 22 percent ammonium nitrate, and 11 percent powdered aluminum, developed in the United States during World War I. During that war, the Germans used a press-loaded composition containing 30 percent TNT, 54 percent ammonium nitrate, and 16 percent aluminum. The ammonals, forerunners of minol, were used in shell for their fragmentation effects. As they are inferior to minol with respect to blast and shock effects and less brisant than composition B or pentolite, the ammonals offer no advantages for use in connection with modern techniques of warfare.

f. Tridite. Tridite is a mixture containing 80 percent picric acid and 20 percent dinitrophenol. During World War II, the British used a mixture containing 70 percent picric acid and 30 percent dinitrophenol, termed nellite, in shell and armor-piercing bombs. A 60/40 mixture, designated MBT, was used by the Italians for loading shell; and the same mixture, designated DD, was used by the French for loading shell and bombs. All three mixtures are suitable for melt-loading at temperatures below 100°C. and therefore represent an improvement over the melt-loading of picric acid. Tridite is slightly inferior to picric acid as an explosive, and dinitrophenol is somewhat objectionable because of toxicity. With the modern trend away from the use of picric acid, tridite has been used with less frequency.

g. Trimonite. Trimonite, a castable mixture consisting of 88 percent picric acid and 12 percent α-mononitronaphthalene, was developed as an improvement on tridite, to avoid the undesirable characteristics of dinitrophenol. Because of the low eutectic temperature of the mixture (46.7°C.), it is subject to exudation when stored at elevated temperatures. It is less sensitive to initiation and less brisant than picric acid and is of interest only as an emergency substitute for TNT compositions. The French used a mixture containing only 70 percent picric acid during World War II.

h. Diethyleneglycol Dinitrate, Dinitrodiglycol or DEGN.

(1) This is a liquid aliphatic nitrate having the structure and a



RA PD 212854

Figure W. DEGN.

molecular weight of 196.12 that was used in propellent compositions by the Germans during World War II. It was first described in 1927 by Rinkenbach. Propellants based on it and nitrocellulose develop relatively low temperatures and cause relatively little erosion of guns, but are unduly volatile.

(2) DEGN boils at 160°C. when heated rapidly. When cooled, it solidifies to a stable form that melts at 2°C. or to a labile form having a melting point of −10.9°C. It has a specific gravity of $1.385 \frac{15}{20}$ and a refractive index of 1.4517 at 20° C. with sodium light. At 20° and 60° C., its vapor pressure is 0.0036 and 0.130 mm of mercury respectively. At 60° C. DEGN has a volatility of 0.19 milligram per square centimeter per hour, as compared with 0.11 milligram for nitroglycerin. It is much less viscous than nitroglycerin, having a viscosity of 8.1 centipoises at 20° C. At constant pressure, DEGN has a heat of combustion of 2,792 calories per gram, from which is calculated a heat of formation value of 103.52 kilogram-calories per mole. DEGN is miscible with ether, acetone, chloroform, benzene, nitrobenzene, glacial acetic acid, and nitroglycerin. It is immiscible with or slightly soluble in ethanol, carbon tetrachloride, and carbon disulfide. Its solubility in water at 25° and 60°C. is 0.40 and 0.46 gram, respectively, per 100 grams. It is colloiding agent for nitrocellulose. DEGN resembles nitroglycerin, with respect to chemical reactivity, but is less subject to hydrolysis and is not readily saponified by alcoholic sodium hydroxide.

(3) DEGN can be manufactured with a yield of approximately 85 percent of the theoretical by adding diethylene glycol to mixed acid containing approximately 50.3 percent nitric acid, 44.7 percent sulfuric acid, and 5.0 percent water. The temperature of the mixture is kept at 30°C. or less. The spent acid is very unstable and fumes off if heated or allowed to stand for a few hours. The separated DEGN is purified by washing with successive portions of water, dilute sodium carbonate solution, and water until neutral. Its purity is dependent chiefly on the purity of the diethyleneglycol used in its manufacture.

(4) DEGN is much less sensitive to impact than nitroglycerin, but explodes in the pendulum friction test. Its explosion temperature test value of 237° C. is slightly higher than that of nitroglycerin (222° C.). The brisance of DEGN, as judged by the modified sand test, is equal to that of TNT and 81 percent that of nitroglycerin. Its rate of detonation (6,800 meters per second) is very similar to that of TNT. Trauzl lead block and ballistic pendulum tests indicate DEGN to be 144 and 127 percent, respectively, as powerful as TNT and 77 and 90 percent, respectively, as powerful as nitroglycerin. Vacuum stability tests show DEGN to be much more stable than nitroglycerin at 100° C., and it withstands long-term storage at ordinary temperatures when free from acidity.

(5) The toxicity effects of DEGN are comparable with those of nitroglycerin, but there is some evidence that fewer individuals are affected than in the case of nitroglycerin. Inhalation

of the vapor or absorption of the liquid through the skin can result in a violent headache.

i. Ethylenediamine Dinitrate (EDD).

(1) This is a molecular compound, $(CH_2.NH_3.HNO_3)_2$, readily formed by neutralizing ethylenediamine with nitric acid, that was used by the Germans during World War II. It was used to a limited extent as a bursting charge. It forms colorless crystals that are readily soluble in water and are somewhat hygroscopic. An aqueous solution of EDD is distinctly acid. The compound has a molecular weight of 186.132 and a heat of combustion of 1,999.8 calories per gram at constant pressure. It can be pressed to a density of at least 1,525.

(2) EDD is more sensitive to shock than TNT, having impact test values of 9 inches and 75 centimeters, respectively, with the Picatinny and Bureau of Mines apparatuses; but it is less sensitive to initiation than TNT, since it requires a minimum detonating charge of 0.10 gram of tetryl in the sand test. The explosion temperature test value of EDD is 445°C., as compared with 475°C. for TNT. The sand test indicates EDD to be 96 percent as brisant as TNT, and their plate dent test values are practically the same. EDD is indicated to be 112 and 125 percent as powerful as TNT by the ballistic pendulum and Trauzl lead block tests, respectively. As judged by the 120°C. vacuum stability test, EDD is less stable than tetryl, but its value of 5 milliliters of gas does not indicate poor stability.

(3) Because of its solubility, hygroscopicity, and acidity characteristics, EDD is not very satisfactory for use as a military explosive and is an inferior substitute for TNT.

j. Tetranitrocarbazole (TNC).

(1) Tetranitrocarbazole is an explosive material produced by the nitration of carbazole:

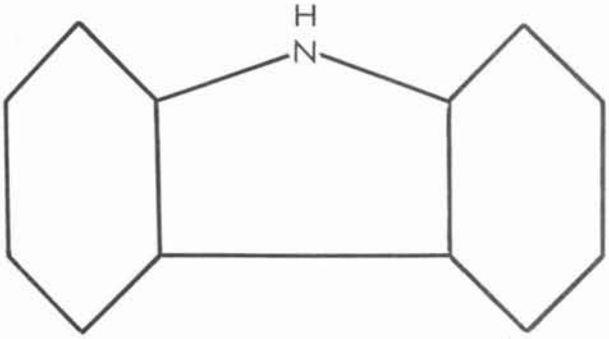


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Figure X. TNC.

The main isomer formed is the 1,3,6,8 derivative, together with probably some of the 2,3,6,8 compound. It has a molecular weight of 283.20. The Germans used it in pyrotechnic compositions during World War II.

(2) As produced, TNC is a crystalline material that melts at 300°C. and is insoluble in water, ethanol, ether, benzene, and carbon tetrachloride. It is only slightly soluble in acetone and fairly soluble in nitrobenzene. It is nonhygroscopic.

(3) TNC is slightly less sensitive to impact than explosive D, having an impact test value of 18 inches with the Picatinny apparatus. Its explosion temperature test value (470°C.) is almost identical with that of TNT, but it does not deflagrate in the test as does TNT. The sand test indicates TNC to be 87 percent as brisant as TNT. It is as stable as TNT and explosive D, as indicated by vacuum stability tests at 100° and 120°C.

(4) Since TNC is not markedly superior to TNT or explosive D, it is suitable for only highly specialized uses.

CHAPTER 9

PROPERTIES AND TESTS OF PROPELLANTS

9-1. Colloidal Structure

a. The use of black powder as a propellant in the form of a dust or fine powder permitted only the most rudimentary control of its ballistic effects. Granulation of the powder as a coarse powder and later as cubical or lozenge-shaped grains resulted in greater velocity and range of the projectile. Rodman's discovery that the rate of release of energy by black powder can be controlled by the form and density of the powder grain led to pressing of the powder into larger grains of definite geometrical form. This permitted the development in a gun of a relatively low maximum pressure over a longer period of time than when fine powder was used.

b. Similarly, the first substitution of nitrocellulose for black powder as a propellant involved its use in a fibrous form, similar to that of the cotton from which it was made. Since nitrocellulose has a greater heat of explosion than black powder and can undergo detonation as well as burning, such use led to damaging explosions in guns. In 1884, Vielle found that nitrocellulose could be added to a mixture of ether and ethanol and kneaded to form a plastic mass. This could be rolled into thin sheets, cut into small squares, and dried, so as to be essentially free from the solvent. The use of such colloided nitrocellulose permitted the same control of burning rate that the pressing of black powder into grains had accomplished. The Schultze and E. C. propellants developed in 1870 and 1882, respectively, had involved only partial gelatinization of the nitrocellulose present in the compositions and the burning of these could not be controlled sufficiently to permit their use in rifled guns. In 1888, Nobel invented ballistite, a propellant in which the soluble nitrocellulose was gelatinized by nitroglycerin instead of a volatile solvent that later had to be removed. In 1889, Kellner and Abel invented cordite, a second double-base propellant, the insoluble nitrocellulose of which was gelatinized by nitroglycerin and acetone. Since then, there have been introduced a number of nonexplosive and nonvolatile gelatinizing agents which serve also as stabilizers, moistureproofing agents, or burning rate deterrents.

c. Nitrocellulose is not soluble in the various gelatinizing agents used in propellants, but is dispersed in the form of colloidal particles, which do not consolidate into macroparticles when essentially all of any volatile solvent present is removed. If nitrocellulose, which has a nitrogen content of 12.6 percent (pyrocellulose), is treated with 2:1 ether-ethanol mixture, complete dispersion takes place and evaporation of the solvent leaves a clear, tough film or grain that is free of visible solid particles when examined under the most powerful microscope. Nitrocellulose, which has a nitrogen content of 13.3 percent, cannot be dispersed completely by means of the 2:1 ether-ethanol mixture, a small portion of the material being only softened and swollen by the solvent. Acetone, nitroglycerin, dibutylphthalate, and other materials disperse to the colloidal state the portion of the nitrocellulose undispersed by the 2:1 ether-ethanol mixture; and the use of such agents results in the production of a structure similar to that obtained with pyrocellulose and the 2:1 ether-ethanol mixture. If a relatively dilute solution of nitrocellulose in a gelatinizing agent is added gradually to water, the nitrocellulose is precipitated as a fine powder, of fibrous structure. Each particle of powder represents the aggregation of many of the colloidal particles previously dispersed in the liquid.

d. Camphor is an excellent gelatanizing agent for nitrocellulose, it being used for this purpose in the manufacture of celluloid. It is, however, too volatile for use in military propellants. Other solids, such as dinitrotoluene and the stabilizers ethylphenylurethane and centralite, are efficient gelatinizing agents when dissolved in liquids, which may have no gelatinizing action. In addition to volatile solvents and nitroglycerin, some nonvolatile liquid gelatinizing agents used in the United States are—

Dibutylphthalate	$C_6H_4(CO.O.C_4H_9)_2$
Diethylphthalate	$C_6H_4(CO.O.C_2H_5)_2$
Triacetin	$C_3H_5(O.OC.CH_3)_3$

In Germany and Italy additional gelatinizers used included—

Diethyleneglycol dinitrate (DEGN)	$O(CH_2.CH_2NO_3)_2$
Metriol trinitrate	$CH_3.C(CH_2.NO_3)_3$

Many other liquid gelatinizing agents are available but less desirable for economic or logistic reasons or the possession of unsuitable physical characteristics. Among these are the esters of sebacic, succinic, citric, and tartaric acids.

e. Numerous methods have been used for determining the completeness of dispersion of the nitrocellulose in a colloid and the relative effectiveness of gelatinizing agents; but none has been standardized. Examination of a thin slice of many propellants under a high-power microscope, particularly with the aid of polarized light, generally suffices to reveal any undispersed material. Comparisons of the relative gelatinizing action of materials can be made by dissolving the material in a nongelatinizing liquid, such as benzene, toluene, or ethanol, immersing a few fibers of nitrocellulose, and observing under a microscope the rapidity and completeness of dispersion of the nitrocellulose. A more quantitative method is to use varying amounts of the material in a fixed volume of liquid that has no gelatinizing action and determine the minimum quantity of added gelatinizing agent that will yield a clear colloid with 1 gram of nitrocellulose when the volatile solvent has been removed by evaporation.

9–2. Physical Forms

a. A propellant undergoes autocombustion only on the surface, the burning progressing as if the propellant consisted of very thin layers, each of which burns and ignites the next layer. If burning takes place under conditions of constant pressure, this progresses through successive layers at a linear rate peculiar to the composition. The total surface available for burning and the time required for burning are determined by linear rate, initial burning surface, and any change in area of burning surface as burning progresses. It is by controlling the mass-rate of burning of a propellent charge, through control of physical form, that the ballistic effect of a propellent charge is determined and kept within certain limits. A powder grain is any individual particle or mass of the propellent composition, and the propellent charge is the total weight of the grains used in a weapon.

b. Numerous forms of powder grains have been utilized, the most important of which are shown by figure 9–1. The most important dimension of these grains is the web thickness or minimum distance between any two adjacent surfaces of the grain. In a cord, the diameter is the web thickness and a single perforated grain has but one web thickness; but a multiperforated grain has inner and outer web thicknesses (fig. 7–2), which may be equal or different. The development of propellants for rockets, the burning of which must progress under different conditions than those of a propellant in a gun, has led to the use of additional forms of grains shown by figure 9–3.

c. The best form of powder grain from a ballistic viewpoint is that which, with the smallest weight of charge, will impart the prescribed velocity to a projectile or rocket without developing a pressure greater than the maximum permitted. The uniformity of the ballistic effect must also be taken into account in this connection. As strips and cords burn, the burning surface decreases continuously until the grain is consumed. Such grains are said to burn *degressively* (fig. 9–4). A single perforated grain burns in two opposing directions and the initial diameter of the perforation can be controlled so that the total burning surface changes but little during burning. Such a grain

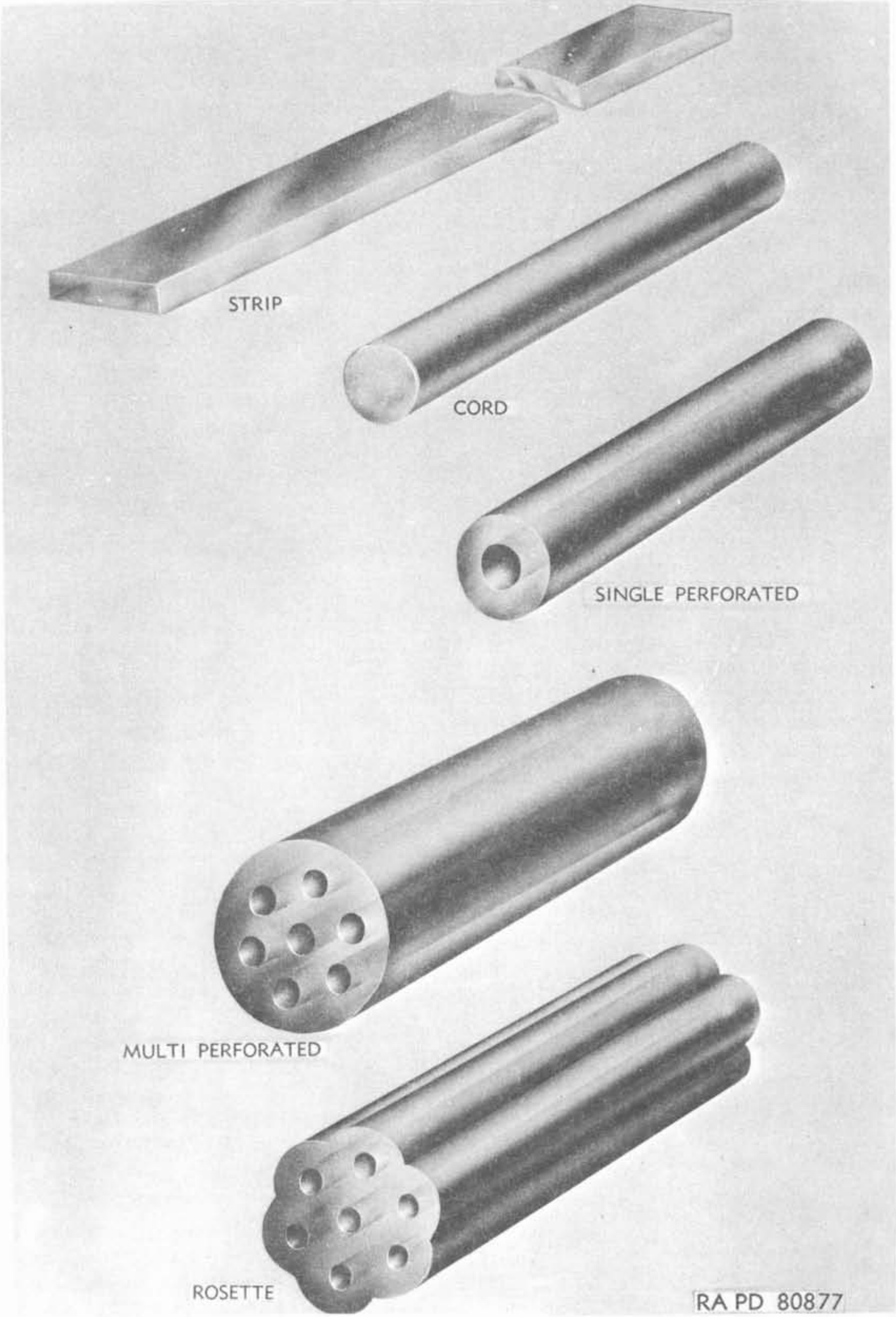
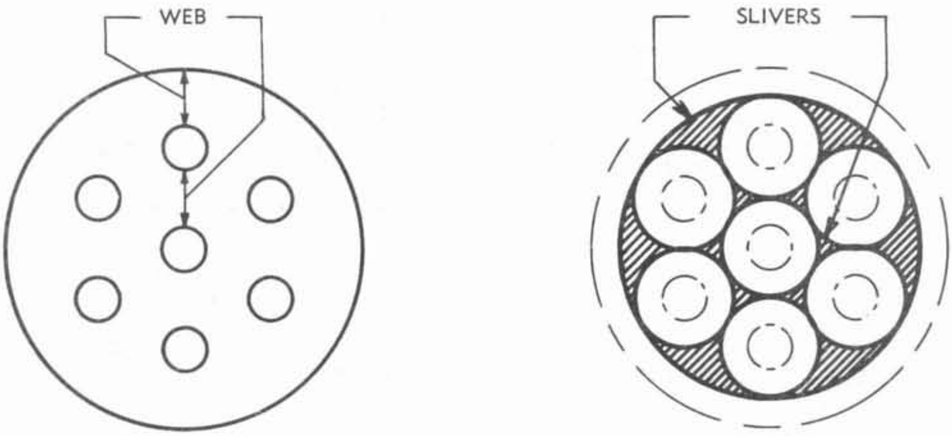


Figure 9-1. Forms of propellent grains.



A—UNBURNED GRAIN

B—BURNING GRAIN

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Figure 9–2. Webs and slivers of propellent grains.

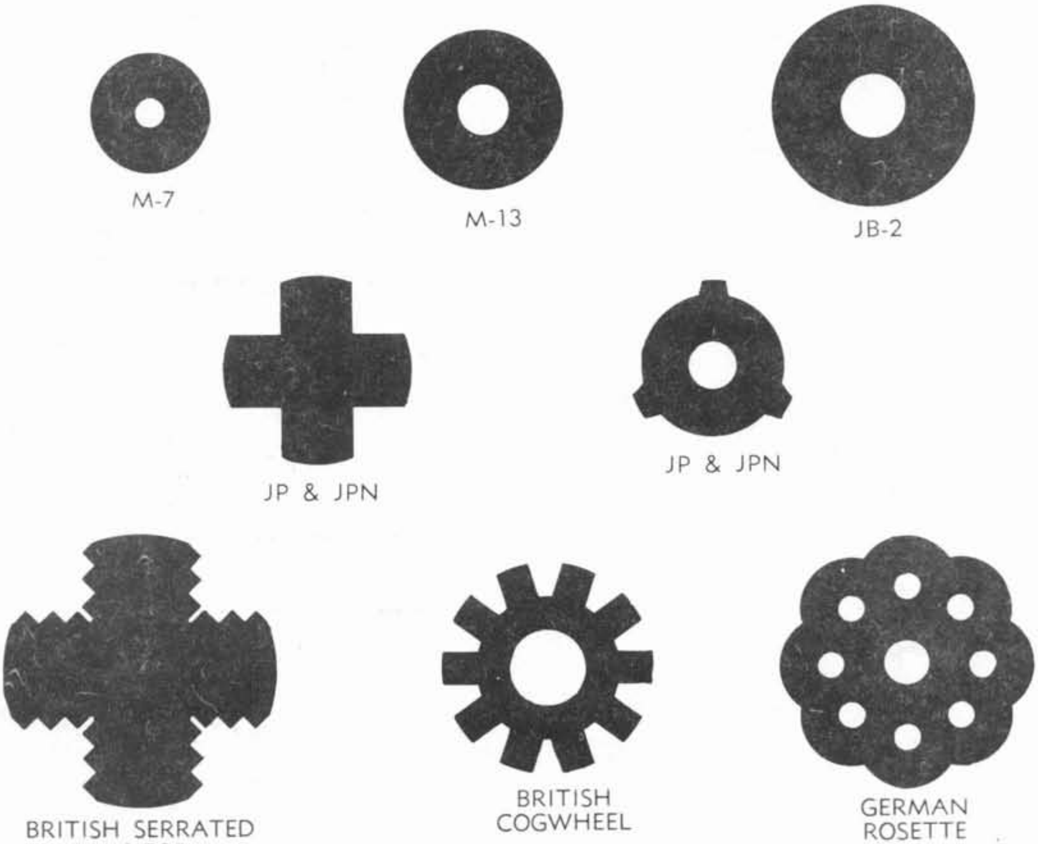

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Figure 9–3. Forms of rocket propellants.

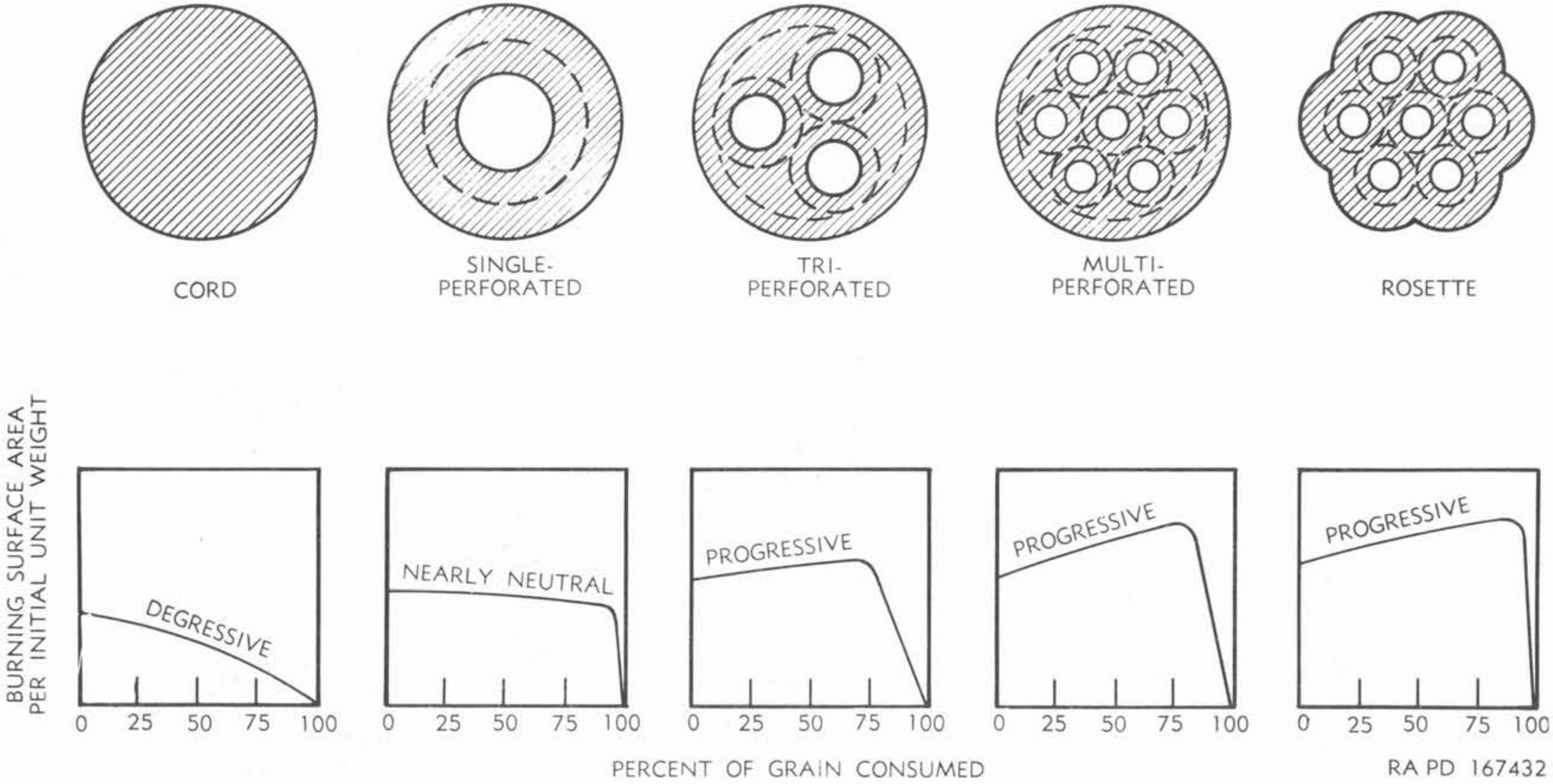

Figure 9-4. Burning of grains of propellant.

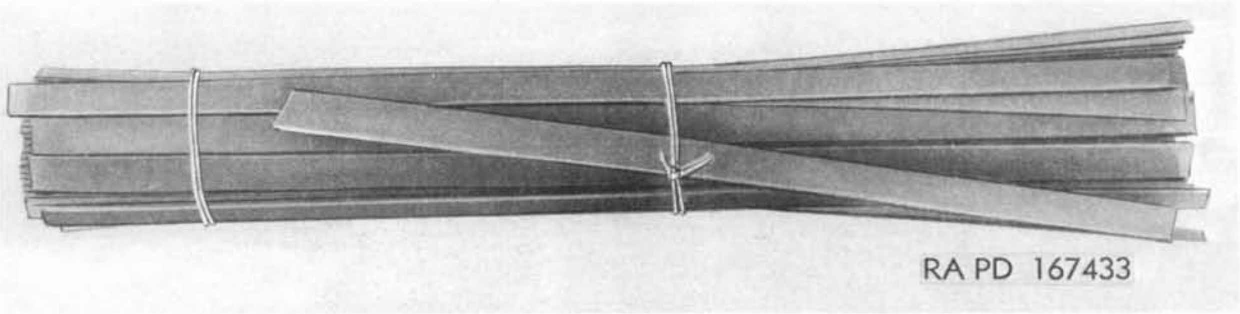

Figure 9-5. German strip propellant for artillery.

is said to have nearly *neutral* burning characteristics. A triperforated grain can be designed so that the burning surface actually increases until burning is nearly completed and *slivers* are formed. Such a grain is said to burn *progressively,* and this characteristic can be made more pronounced if the grain is multiperforated.

d. European nations have favored the use of single perforation, strip, and cord propellants (figs. 9-5 and 9-6). The United States Army and Navy have favored the use of the multiperforated grain form for use in weapons, since its small initial burning surface per unit weight of powder results in a slower increase in pressure during the initial stage of its burning. This permits maximum pressure to be developed at a point farther from the breech of a gun than when a degressively burning powder is used. Single perforated powders are used for United States small arms, minor caliber cannon, and certain howitzers, because of the difficulty in manufacturing by extrusion multiperforated powders having the exceptionally thin webs that would be required if the powders were to meet ballistic requirements. In rockets, there is generally used a grain that is neutral or slightly degressive, in order to obtain a nearly constant pressure during burning. This has led to the use of single perforated grains and special designs (fig. 9-3), in some of which certain surfaces are inhibited from initial burning by means of a coating of less combustible material.

e. When a powder grain having seven perforations burns, there are produced 12 unburned slivers or pieces of triangular cross section that represent approximately 15 percent of the total weight of the grain. These slivers burn degressively and usually are burned in the bore of the weapon; but if the tube is relatively short or a reduced charge of propellant is fired, incompletely burned slivers may be expelled from the muzzle. The rosette or Walsh grain (fig. 9-1), with a scalloped outer periphery, reduces the amount of slivers produced by a multiperforated grain and therefore reduces or eliminates the amount of unburned powder ejected from the seacoast mortars in which such powders have been used.

f. Other factors being equal, the greater the powder chamber capacity of an artillery weapon, the greater is the size of the propellent grain to be used. This size of a multiperforated grain refers to its average web, the mean of its outer web and inner web dimensions. These are the minimum distances, respectively, between the outer periphery of the grain and the wall of an outer perforation, and an outer perforation and the wall of the central perforation (fig 9-2). As an example, the powder chamber capacities of two mortars using the same weight projectile are 2,674 and 3,770 cubic inches and the required average webs of powders for these are 0.060 and 0.076 inch, respectively. Relatively slight changes in the average web of a powder result in appreciable changes in the required weight of propellent charge and pressure produced, although the velocity of the projectile remains constant. In the 3-inch antiaircraft gun, charges of 4.88 and 5.66 pounds of propellant having web

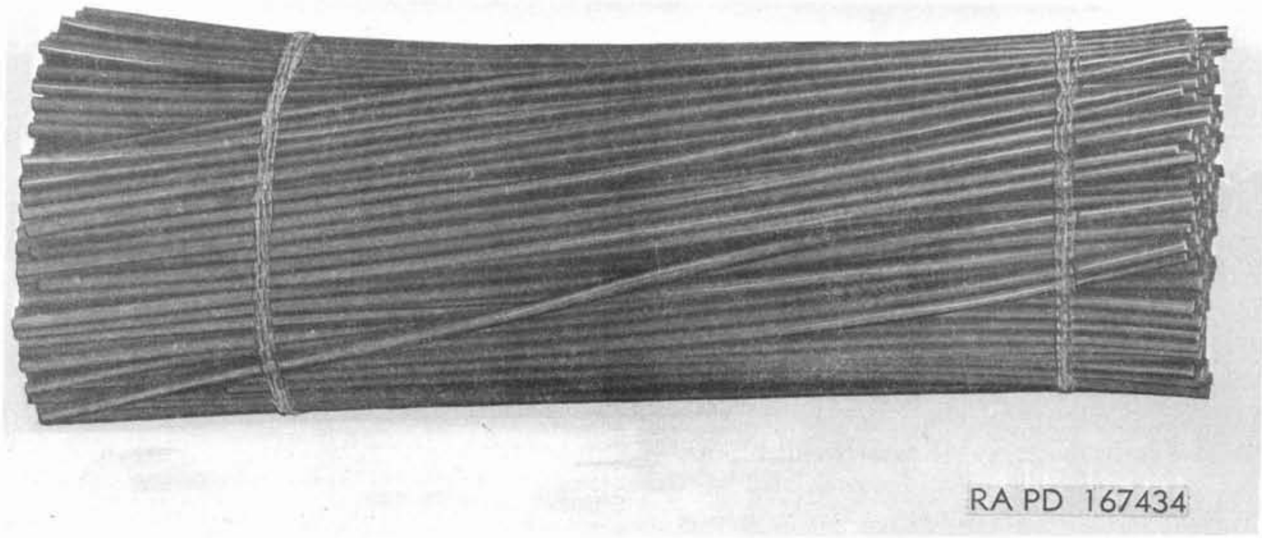
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Figure 9-6. German cord propellant for artillery.

values of 0.0339 and 0.0444 inch imparted the prescribed velocity to the projectile and developed maximum pressures of 34,000 and 28,700 psi, respectively. For economy, it is desirable to use as low a weight of charge as practicable, and therefore the web of the propellant should be as thin as is permitted by maximum pressure requirements. Since better velocity uniformity commonly is obtained at the higher pressures, it is desirable to use propellant with as thin a web as practicable in zone charges, so that higher pressures will be obtained in firing the inner zones.

g. The heavier the projectile or the greater the prescribed velocity, the larger will be the powder grains and the web of these required for the propellent charge, if the maximum pressure is to be maintained below a certain limit.

h. Propellants for artillery, in particular, contain small amounts of residual solvent consisting of water and alcohol. In designing powder grains, this must be taken into account, since the residual solvent content significantly affects the ballistic potential and burning rate of the composition.

i. In practice, there are certain general relationships between the dimensions of powder grains that remain within fairly narrow limits. Specification requirements for cannon powders include the relative dimensions in (1) through (3) below.

(1) The ratio of average grain length (L) to average grain diameter (D) is 2.10 to 2.50 for multiperforated and 3 to 6 for single perforated grains.

(2) The ratio of average grain diameter (D) to the average diameter of perforations (d) is 5 to 15 for multiperforated and approximately 3 for single perforated grains.

(3) The difference between the average outer web thickness (W_o) and the average inner web thickness (W_i) of multiperforated powder is not more than 15 percent of the average web thickness (W_a).

j. Average grain dimensions are determined with great care when a lot of powder is offered for acceptance. The average length of each of 30 grains is determined by means of a micrometer accurate to 0.001 inch and the values for all the grains are averaged. Grain diameter, perforation diameter, and web thickness values for 30 grains are determined by means of a measuring microscope having a magnification of approximately 25 diameters and capable of giving measurements accurate to 0.001 inch.

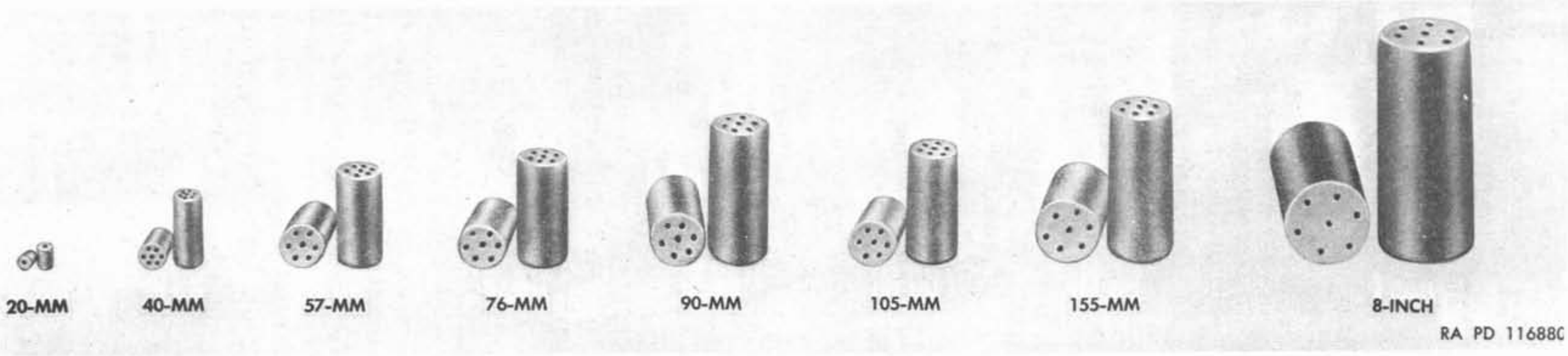


Figure 9-7. Relative grain sizes of propellants.

The average values are calculated from the values for each grain and the above ratios are calculated from the average values.

k. The wide variety of weapons in which propellants are used requires that powder grains be used with a correspondingly wide variation in size as well as form. Figure 9-7 shows the relative grain sizes of powder grains used in artillery weapons. Powder grains used in rockets are considerably larger than those for artillery.

9-3. Ignitibility

a. A charge of propellant in a powder chamber or rocket motor is ignited by suddenly filling the voids between and the perforations in the powder grains of the charge with flaming gas and hot, solid particles. This accomplishes the ignition of all the exposed surfaces of the grains and insures uniform burning of all the grains comprising the charge. In small arms, a primer mixture that is sensitive to percussion is used for ignition. Mortar charges are ignited by a small quantity of double-base propellant loaded in cartridges and ignited, in turn, in the same manner as small arms and shotgun charges. Black powder is used as the ignition material for artillery and rocket propellants. In artillery rounds, the black powder is loaded in metal tubes having many perforations of the walls or in cloth bags sewed to the base of the bag holding the propellant charge. Rocket propellants are ignited by black powder charges, frequently loaded in flat, plastic cases and placed at the front end of the rocket motor.

b. Black powder has been found by experiment to be a more efficient ignition material for nitrocellulose propellants than the finely divided propellants themselves. This is ascribed to the large amount of radiant heat energy in black powder gases emitted by the incandescent, solid particles of potassium compounds that are present.

c. The ignitibility of a propellant is determined by its composition and energy content. In general, the greater the energy content of a composition, the greater is its ignitibilty. Experiments have shown the following:

(1) Increase in the nitrogen content of the nitrocellulose present causes slight increase in ignitibility.

(2) The substitution of nitroglycerin for part of the nitrocellulose in a composition increases ignitibility to a marked extent.

(3) The inclusion of an inert gelatinizing agent, such as dibutylphthalate or triacetin, in a propellent composition results in decrease in ignitibility.

(4) The inclusion of TNT or dinitrotoluene in a composition causes measurable decrease in ignitibility.

(5) The inclusion of 5 percent or less of potassium nitrate in a composition causes significant increase in ignitibility.

(6) The presence of a coating of graphite on a propellant causes retardation of ignition.

d. In some cases, it is desirable that the initial part of the burning of a propellant take place with a burning rate less than that of the composition to be used. This can be accomplished by the application to the powder grain of a coating of a deterrent, such as dinitrotoluene or centralite. The deterrent is chosen so as to have a moistureproofiing effect and a melting point less than 100°C. The coating then is applied, without heating to excessive temperature, by tumbling the dried powder with the molten deterrent in a steam-heated sweetie-barrel. The coating so applied is less ignitible than the propellent composition and burns more slowly.

e. No test for determining the relative ignitibility of propellent compositions has been standardized; but comparative values have been obtained by a variety of methods. One consists of determining the minimum pressure of a gas mixture that will ignite a sample of powder of a certain granulation when the gas is ignited by an electric spark. The gas mixture used has a composition equivalent to two molecules of hydrogen, one molecule of oxygen, and one molecule of nitrogen. The greater the pressure required, the less ignitible is the composition. Another method consists of suddenly exposing a sample of propellant to a fixed volume of gas at a temperature of approximately 1,800°C. and, by means of a pressure gage and recorder, determining the time interval between such exposure and beginning of burning of the propellant. The greater the time interval, the less ignitable is the composition. Comparative values for standard compositions, obtained by testing by the first method, are given in table 9-1. A value for black powder is included for comparison. A test for determining the relative ignitibility of propellant grains was standardized at Picatinny Arsenal.

f. In the practical use of propellants, complete ignition of the propellant charge must be assured; and this requires careful design of the igniting charge. A charge of priming composition or black powder that is entirely adequate for the uniform ignition of a standard powder charge may be inadequate if the composition of the propellant is changed. An example of this was observed when the less ignitible M1 powder replaced pyrocellulose powder in artillery ammunition.

Table 9-1. Minimum Pressures of $2\ H_2 + O_2 + N_2$ *Required for Ignition of Propellents*

Propellent composition	*Minimum pressure, inches*
Pyrocellulose	25.0
M1	27.5
M2	13.5
M3	31.0
M9A	7.0
Black powder	<5.0

The use of larger charges of black powder in longer primers resulted in uniform ignition of the M1 powder and correspondingly uniform maximum pressure and velocity effects.

g. The initial temperature of a propellant composition has considerable effect on its ignitibility, the ignitibilty decreasing with decrease in temperature. As the effect on the burning rate index of the composition is similar, firing of a propellant charge at a lower than standard temperature results in low velocity and pressure effects. When the charge is fired with a higher than standard temperature, high velocity and pressure effects are obtained.

9-4. Burning Rates

a. Propellant compositions burn at characteristic linear rates, which are affected by the initial temperature and the pressure under which burning takes place. Increase in pressure or initial temperature generally causes increase in the characteristic burning rate of a composition. If a powder is burned under confinement, the resulting increase in pressure causes rapid acceleration of the rate of burning.

b. When compositions used in artillery are granulated, the form of the grain, as well as the characteristic burning rate, determines the time required for complete burning, or relative quickness values, of the propellants as such.

c. The pressure effects of burning a typical artillery propellant in a bomb are shown by figure 9-8. After ignition, acceleration of the burning rate takes place, due to the production of pressure. At first, this acceleration per unit increase in pressure is relatively small and is represented by a in the equation:

$$R = a + bP$$

where R is the rate of burning at a given initial temperature, b is the acceleration in burnng rate per unit increase in pressure at

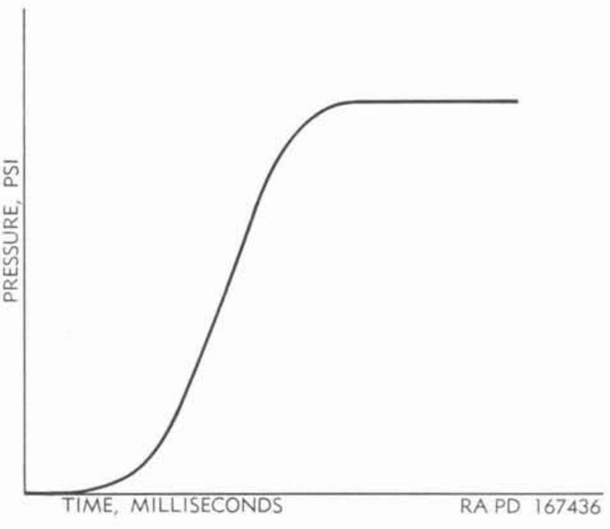


Figure 9-8. Pressure produced by propellant burned in a bomb.

the higher pressures produced, and P is pressure in pounds per square inch. After a certain pressure has been produced, the rate of acceleration of the burning velocity increases rapidly to the constant value b, which is maintained until all of the powder has been consumed. This is shown by the straight or nearly straight portion of the curve. The value b, termed the burning velocity index, is characteristic of the composition. It is of major importance in the burning of artillery powders, while the value a is of negligible importance. Tests of such powders in a bomb are so representative of artillery firings that bomb tests can be used with experimental compositions and in partial substitution for proof tests of propellants. On the other hand, since rockets operate with relatively low maximum pressures, the characteristic a of rocket powders is of major importance. The burning rate of a propellant composition can be expressed also by the equation:

$$R = cP^n$$

where c and n are empirical constants characteristic of the composition.

d. Solid rocket propellants, being required to function under steady-state pressure conditions much lower than the maximum pressures under which cannon powders burn, have maximum burning rates in use that are correspondingly lower than those of cannon powders. The range of burning rates of solid rocket propellants under a pressure of 2,000 psi is about 1 to 2 inches per second. When the propellent charge of a rocket is ignited, the subsequent increase in pressure within the chamber is quite rapid, generally requiring less than 0.05 second to reach its maximum. Thereafter, the rate of burning of the propellant remains quite constant, because the pressure is nearly constant until the charge is nearly consumed. The rates of burning of propellent compositions for rockets are comparable with those of propellent compositions used in artillery; and in some cases the compositions themselves are not dissimilar.

e. Increase in the initial temperature of a propellent composition causes increase in the burning rate of the composition as shown by table 9–2.

Table 9–2. Effect of Temperature on Burning Rate

Composition	*Temperature, ° C.*	*Burning rate, in/sec/psi*
M2	0	26 x 10^{-5}
	25	29 x 10^{-5}
	58.5	34 x 10^{-5}
M9	0	39 x 10^{-5}
	25	43 x 10^{-5}
	58.5	47 x 10^{-5}

Decrease in burning rate because of decrease in initial temperature results in decreased pressures and, because of difficulty in achieving satisfactory ignition, frequently results in increase in the variation of projectile velocity. The effects of changes in the initial temperature of rocket propellants are more pronounced than in the case of cannon propellants. One reason for this is that the performance of a weapon that operates at a constant pressure, such as a rocket, is much more affected by slight changes in the rate of burning than is that of a weapon operating under conditions of increasing pressure, as does a cannon.

9–5. Ballistic Effects

a. General. The autocombustion of a solid propellent charge in a weapon represents the conversion of a solid into a gas, the liberation of energy, and the development of much gas pressure. The considerations that deal with the rate of energy release, the total quantity released, the temperature and pressure developed, and the velocity imparted to the projectile or rocket constitute the field of *interior ballistics*. As the use of propellants in rockets differs somewhat from that of propellants in cannon, mortars, and small arms, the interior ballistic aspects of rocket propellants will be considered separately.

b. Weapons.

(1) When fired in a cannon or similar weapon, the potential thermal energy of a propellant is partially converted into kinetic energy of the projectile. The proportion of the total available energy that can be communicated to the projectile is limited by the length of travel of the projectile in the gun

tube and energy losses through friction, heat conductance, barrel expansion, etc. Figure 9-9 shows a representative pressure-travel curve for a projectile in a cannon. The maximum pressure is reached at point *A*, the distance of this from the powder chamber being determined partly by the form and consequent burning characteristics of the powder grain. The more progressively the powder burns, the further *A* will be from the powder chamber. With maximum pressure at point *A*, a higher velocity is obtainable at a lower pressure than would be the case if the same weight of powder was consumed in the powder chamber. This is apparent also from figure 9-10, which shows pressure-travel curves for three types of grains.

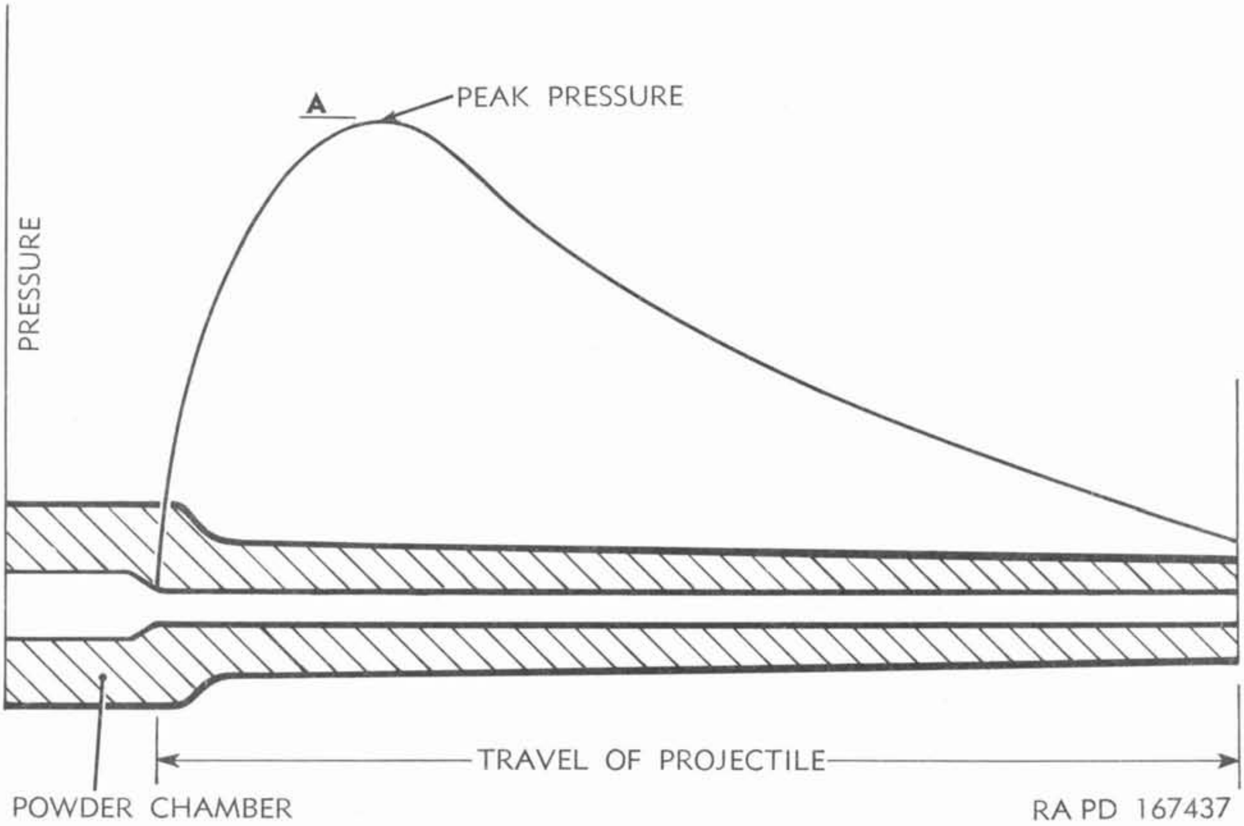


Figure 9-9. Pressure travel curve for a projectile in a gun.

(2) Both the pressure in the gun tube and the velocity of the projectile increase directly, with increase in weight of powder of a given granulation used, until a critical point is reached. Beyond this point, a further increase in weight of powder charge causes a very sharp increase in maximum pressure but no corresponding increase in velocity of the projectile. This is known as the point of pressure break. For satisfactorily uniform ballistic effects, the web of the powder grain must be such as to give the desired projectile velocity well below the point of pressure break.

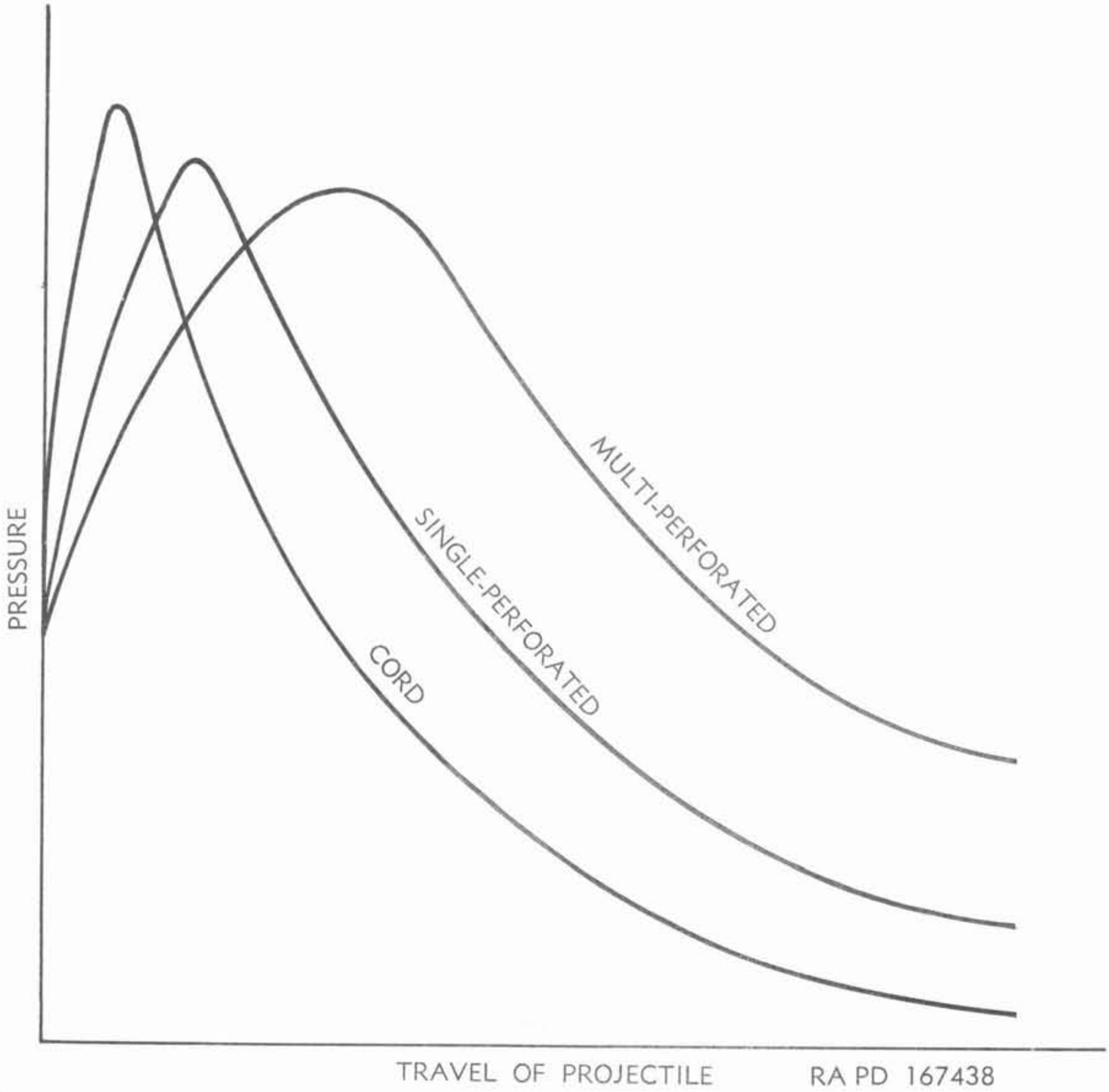


Figure 9-10. Pressure-travel curves for three types of propellent grains.

(3) Since the energy of a propellant is almost completely converted to heat during burning, the heat of explosion is a good indication of the ballistic potential of a propellant. The work the propellant can do can be expressed as—

$$W = n\,RT$$

Where: W is work in foot-pounds per pound

n is moles of gas per unit weight

R is the gas constant (2,782)

T is the isochoric flame temperature

From the equation, it can be seen that the volume of gas produced is of considerable importance as well as the heat liberated. An example of this is the equal work values of the pyrocellulose and M15 compositions, which have heat of explosion values of 861 and 800 calories per gram and gas volume values of 0.04106 and 0.04640 mole per gram, respectively.

(4) The kinetic energy of the projectile is represented by the area under the curve in figure 79, multiplied by the cross-sectional area of the projectile. This is equal to—

$$W = \frac{1}{2}mv^2$$

Where: W is the work performed
m is the mass of the projectile
v is the velocity of the projectile

From this, the weight of propellent charge, and the energy value of the composition, the efficiency of the weapon can be calculated.

c. Rockets.

(1) Solid propellants for rockets are primarily of two types. The more common type is a double-base composition consisting principally of a colloided mixture of nitrocellulose and nitroglycerin. The other type consists of a heterogeneous mixture of an organic fuel, an inorganic oxidizing agent, and a binding agent. In either case, the mode of burning and the limitations under which the compositions are used are the same.

(2) Most rocket motors are constructed to accommodate maximum pressures developed by the propellants of the order of 3,500 psi. When the propellent charge is ignited, the pressure within the rocket chamber generally increases within 0.005 to 0.05 second to a maximum value determined by the burning rate of the propellant and the diameter of the nozzle orifice. Thereafter, the charge burns at a nearly constant rate and the steady-state pressure is maintained constant or decreases very slowly until the propellant is completely consumed. The time required for complete combustion may vary from a fraction of a second to a minute or more. The limitations on maximum pressure are governed by the strength of the rocket motor and the maximum mass rate of gas discharge that can be permitted for a given end use. The pressures within the motor can be changed readily by changes in powder composition, burning surface area, and ratio of nozzle area to burning surface area.

(3) It has been found that each propellent composition has a critical pressure below which it burns nonuniformly and gives erratic ballistic effects, and a higher critical pressure above which it burns in an unpredictable manner and often ruptures the motor by apparent detonation. A comparison of the critical pressure values of several types of composition is afforded by the approximate values given in table 9–3.

Table 9–3. Critical Pressure Values of Rocket Propellants

Type of composition	*Critical pressure, psi at 25° C.*	
	Lower	*Upper*
Double-base	400–800	4,000–12,000
Composite	100–400	2,500– 5,000
Single-base	5,000	(*)

*Not established.

The very high lower critical pressure value of single-base powder precludes the use of such a composition in rockets.

(4) The transformation of heat energy into kinetic energy of flow causes a force to act in a direction opposite to the flow of the exhaust gases from a rocket motor. This force, termed *thrust,* is a function primarily of the velocity at which the gases leave the rocket nozzle and the weight of the gases and is independent of the velocity of flight. Thrust is expressed in pounds. In order to develop a high thrust value, there must be employed a grain or charge with a large burning surface, so that a high rate of mass flow is realized.

(5) The duration of burning of a rocket propellent charge, most of which occurs under steady-state pressure, is determined by the web of the grain and its burning rate. In order to obtain long duration of thrust, only a small porportion of the propellant can burn in unit time. Since the combustion chamber of a rocket has fixed dimensions and capacity for propellant, the thrust may be made great but of short duration or vice versa if the lower and upper critical pressure limits and the bursting pressure of the rocket are not exceeded. The product of force and duration is termed *impulse* and is expressed as pound-seconds. This may be used for comparing different, as well as equal, weights of charge.

(6) The impulse per unit weight of propellant is termed the *specific impulse* and is expressed in pound-seconds per pound of propellant. It may be considered as the pounds of thrust developed per pound of propellant consumed per second and can be used for comparison of propellent compositions. It is known also as the *performance index.* As the space in a rocket motor available for the propellant is limited, the density of the propellant is important and specific impulse may be calculated on both a unit weight and unit volume basis. Table 9–4 gives comparative specific impulse values for various propellent compositions used in rockets.

Table 9–4. Specific Impulse Values of Propellent Compositions

Propellent composition	*Specific impulse, lb-sec/lb*
M7	237
M13	242
T2	228
T5	234
T8	210
T9	165

It is to be noted that the specific impulse values for propellent compositions do not take into account the weight of the motor and therefore are of value only in comparing one propellant with another.

(7) The effect of initial temperature on the rate of burning of a World War II rocket propellant is much more pronounced than in the case of cannon propellants. Decrease in initial temperature causes marked decrease in burning rate and in the maximum pressure obtained. The results of comparative tests at various temperatures are given in table 9–5.

Table 9–5. Effects of Initial Temperatures on Burning Characteristics of a World War II Rocket Propellant

Initial temperature, ° C.	*Time of burning, sec*	*Pressure, psi*
55.5	2.5	2,500
20	4.25	1,000
−11	6	750

As the impulse is about the same in each case, change in initial temperature does not materially affect the range of the rocket. The velocity of the rocket and its ballistics may be affected by change in initial temperature, so that uniformity is poorer at low and high temperatures than at normal temperature. Furthermore, if rockets are to be fired on some occasions at relatively high initial temperatures, a heavier motor must be provided to withstand the high pressures developed during such firings. The need to provide a heavy motor to permit high temperature firings limits the velocities that can be developed at lower pressures. For this reason, every effort is made to utilize propellent compositions having low temperature sensitivity. One of the chief advantages of the recently developed propellants, such as the T–9 composition, is their very low temperature sensitivity as compared with that of the earlier rocket propellants containing nitroglycerin.

(8) In addition to the effects of change in initial temperature resulting from changes in chemical reaction rates,

change in temperature may have important physical effects that are reflected in the ballistic performance of a rocket propellant. If the grain of double-base powder is of large diameter or is restricted (inhibited), storage at or exposure to high and low temperatures may cause the development of gas pockets within the grain or cause it to crack. This results in increase of the burning surface, and burning in gas pockets or cracks may create sufficient pressure to cause rupture of the rocket motor when fired. On the other hand, exposure of grains of uncolloided, composite type propellant to low temperatures may cause changes in the physical properties of the binding agents and result in cracking of the grains. When fired, such grains burn as erratically as those of colloided powder cracked by exposure to high and low temperatures.

(9) Because of the foregoing effects of change in temperature on the chemical reaction rates and physical structures of rocket propellants, the high and low temperatures at which rockets can be fired today generally are within the limits −40° C. (−40° F.) and 49° C. (120° F.). In view of the global nature of modern warfare and the necessity for using ammunition under extremely cold and hot climatic conditions, much modern development effort in connection with rocket propellants is in the direction of compositions of low temperature sensitivity from both chemical and physical viewpoints.

9-6. Sensitivity

a. Propellants are so insensitive to shock and friction, even when they contain high percentages of nitroglycerin, that they present no practical problem from this viewpoint. While dry nitrocellulose is very sensitive to impact, colloidal dispersion of this and the retention of a small percentage of volatile solvent or other gelatinizing agent renders the material quite insensitive. From a practical viewpoint, the sensitivity hazards of propellants are chiefly in connection with heat and spark.

b. While not exploded or even ignited by ordinary shocks or frictional effects, some propellants can be ignited by the friction and impact effects of small-arms bullets. If the propellant is confined in a standard packing box and ignited by a caliber .30 bullet, ignition may be followed by detonation if the container is covered and nearly surrounded by other containers.

c. If a quantity of propellant is ignited, it may burn or undergo explosion. Whether burning or explosion takes place depends upon the composition of the powder and the degree of confinement. For this reason, manufacturing buildings, generally, are of light construction, so that not much pressure can be developed in the event of a fire. Several years after World War I, a large quantity of pyrocellulose powder, stored in standard packing boxes, underwent spontaneous ignition and burned without explosion.

d. Experiments have shown that the confinement afforded by the upper part of a column of propellant may be sufficient to cause explosion if the powder at the bottom of the pile is ignited. Various powder compositions were found to have correspondingly varying critical heights of column, the critical height varying inversely with the energy content of the composition. For the same composition, the critical height was found to vary directly with the average web of the powder. If the temperature of the powder is 50° C. instead of 20° C., the critical height of a composition is only 30 to 50 percent of that at the lower temperature. Propellants for small arms, consisting chiefly of nitrocellulose, were found to have critical height values of 15 to 20 inches. Single-base cannon powders of larger grain sizes and containing moderants have critical height values of 30 inches or more.

e. Propellants are sensitive to electric sparks only if the material is in the form of a dust. Some test results indicate the dust of a powder to be much more ignitible by a spark of static electricity than is nitrocellulose. Dry-house and blending-tower fires have occurred that were attributed to the primary ignition of powder dust by sparks of static electricity. Maintenance of the relative humidity of the atmosphere above 60 percent has been found to prevent such spark discharges and ignitions. The humidity must be measured *where the operation is going on.*

f. Propellants can be detonated by initiating compounds or booster explosives. Sand tests have indicated E. C. powder to be more sensitive to initiation and slightly more brisant than TNT. Double-base propellants are even more sensitive and brisant, but single-base propellants, such as the pyrocellulose and M1 compositions, are so insensitive that only detonations at low rates take place and low brisance values are obtained. In spite of this, single-base powders for caliber .30 and 20-mm ammunition can be detonated by a charge of tetryl even when immersed in water.

g. Summarizing, it may be said that nitrocellulose propellants have the inherent properties of detonating high explosives. However, they are particularly sensitive to heat and represent chiefly fire hazard unless under confinement, when burning may be transformed into detonation.

9-7. Stability and Stabilizers

a. The stability of colloided propellants centers around that of nitrocellulose, which is one of the least stable of military explosives and is used in grades having different nitrogen contents and levels of stability. As stated previously, materials known as stabilizers are added to counteract the undesirably low stability of nitrocellulose. However, the presence of moisture is known to increase the rate of decomposition of nitrocellulose at a given temperature; and generally it is impracticable to remove all moisture from a propellant during the course of manufacture. The various propellent compositions, therefore, vary widely with respect to stability, and different lots of the same composition may differ measurably when manufactured.

b. If a propellant is stored for a lengthy period at ordinary or elevated temperatures, the nitrocellulose undergoes decomposition at a rate dependent upon the temperature. The acid products of decomposition immediately react with and are neutralized by the diphenylamine, centralite, or other stabilizer present. The rate of decomposition of the nitrocellulose thus is prevented from being accelerated, as it would be if no stabilizer was present. When deterioration has progressed to the point that the stabilizer content has been reduced to a few tenths of 1 percent, this neutralizing action is no longer significant and the rate of decomposition becomes accelerated. The presence of an acid odor and, finally, visible red fumes indicate that the propellant has become definitely unstable.

c. The decomposition of nitrocellulose, as well as any other explosive present in a propellant, is exothermic. When the rate of decomposition is low, any heat liberated is lost by conduction under the usual conditions of storage. When the rate of decomposition becomes accelerated, such loss of heat liberated may be impeded if the propellant is stored in bulk. As a result, there develops in the mass of powder a region where increase in the temperature proceeds rapidly, since each increase in temperature causes further acceleration of the rate of decomposition. The increase in pressure within the tightly sealed box used for storage also contributes to acceleration of the rate of decomposition. The final result is spontaneous ignition and destruction of the propellant along with that of nearby material. During the period following World War I, there were a number of incidents of the spontaneous ignition of smokeless powder stored in bulk in localities having a warm or temperate climate.

d. Because of the hazardous nature of badly deteriorated propellants and their impaired ballistic values, it has been necessary to establish a surveillance system for propellants stored in bulk or in components of ammunition. In the case of bulk storage, a strip of tenth-normal methyl violet paper is inserted in the box of powder and inspected after one year for bleaching indicative of significant deterioration. Propellants loaded in ammunition having cartridge cases 3 inches in diameter or less have been found to represent no hazard of spontaneous ignition, because of the proportion of metal to propellent charge and the high heat conductance of the metal. Propellants loaded in larger cartridge cases, separate charges, and assembled charges are subjected to periodic sampling and testing for deterioration.

e. The stability of a propellent composition is affected by the purity of the materials used in its manufacture and the thoroughness of execution of the details of manufacture as well as the nature of the composition and the presence of moisture. If the nitrocellulose used is not purified, so as to be essentially free from cellulose sulfate, the stability of the composition is much reduced. The presence of any inorganic acid in any of the raw materials also has a pronounced effect in decreasing stability. The problem of making available propellants of satisfactory stability, therefore, has necessitated strict control of the details of manufacturing and continued surveillance of the product as well as the use of materials of rigidly controlled purity. This last has included strict control of the purification process for nitrocellulose as well as rigid test requirements for the material.

f. While propellants manufactured of impure materials have been known to become dangerously unstable within 5 years after manufacture, those manufactured carefully from pure materials withstand storage in a temperate climate for many years. Pyrocellulose powders with no added stabilizer, consisting only of colloided nitrocellulose and residual solvent (ether and ethanol), have been known to remain stable during storage for 40 years. Modern single-base compositions are indicated to be even more stable. A double-base propellant, containing 30 percent of nitroglycerin and 5 percent of petroleum jelly, was found to be of a high order of stability after 30 years of storage; and more modern double-base compositions are indicated to be even more stable.

g. The use of stabilizers as such in propellent compositions was preceded by the incorporation of a material, such as amyl alcohol or rosaniline, that indicated decomposition and the liberation of free acid by developing a characteristic odor of valerianic acid or by a change in the color of the powder. The materials currently used as stabilizers react with products of the decomposition of nitrocellulose to form stable solid compounds and so remove free acid and oxides of nitrogen as rapidly as they are formed.

(1) Diphenylamine $(C_6H_5)_2NH$, a compound melting at 53° C., has been used as the chief stabilizer for single-base propellants and to a lesser extent in powders containing nitroglycerin. It is capable of reacting directly with nitrogen dioxide as well as acids. Upon reaction during the decomposition of nitrocellulose, it has been found to form the following compounds successively, a number of these being present simultaneously:

N-nitrosodiphenylamine.
2-nitrodiphenylamine.
4-nitrodiphenylamine.
N-nitroso-2-nitrodiphenylamine.
N-nitroso-4-nitrodiphenylamine.
4,4′, 2,4′, 2,2′ and 2,4-dinitrodiphenylamines.
N-nitroso-4, 4′-dinitrodiphenylamine.
N-nitroso-2, 4′-dinitrodiphenylamine.
2, 4, 4′ and 2, 2′, 4-trinitrodiphenylamines.
2,2′, 4,4′-tetranitrodiphenylamine.
2,2′, 4,4′, 6-pentanitrodiphenylamine.
Hexanitrodiphenylamine.

The detection of any one of these in a propellant, therefore, does not indicate that the powder has become unstable. As diphenylamine is somewhat reactive with nitrocellulose, too much of this stabilizer should not be used; and 1 percent or less of the material, generally, is employed.

(2) 2-Nitrodiphenylamine, $C_6H_5.NH.C_6H_4.NO_2$, is an effective stabilizer for propellent compositions and has been found to have value in preventing the

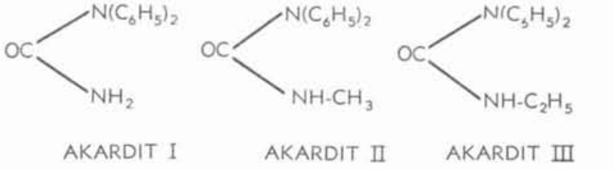
formation of gas bubbles in such compositions. Although it contains a nitro group and so has some explosive value, it is so insensitive that it cannot be caused to explode when subjected to the impact, sand, and explosion temperature tests. Heat tests at 100° and 120° C. indicate 2-nitrodiphenylamine to be very stable. The crystalline compound has a freezing point of 74.6° C., and it is very soluble in molten dinitrotoluene at 70° C. As it is one of the intermediate compounds formed from diphenylamine upon reaction during the decomposition of nitrocellulose, 2-nitrodiphenylamine acts as a stabilizer by undergoing further nitration with the ultimate formation of hexanitrodiphenylamine.

(3) Centralite I or ethyl centralite, symmetrical diethyldiphenylurea, $OC[N(C_2H_5)(C_6H_5)]_2$, was developed in Germany for use in double-base propellants. In addition to acting as a stabilizer, it is a gelatinizing agent for nitrocellulose and acts as a waterproofing agent. Unlike diphenylamine, it can be used in relatively large proportions; and some propellent compositions contain as much as 8 percent of this material. Like diphenylamine, it is nitrated by the products of decomposition of nitrocellulose. The following compounds are formed successively, as many as four of these being present simultaneously, as deterioration of the powder proceeds.

4-nitrocentralite.
4,4′ dinitrocentralite.
N-nitroso-N-ethylaniline.
N-nitroso-N-ethyl-4-nitraniline.
2,4, dinitro-N-ethyl-aniline.

Centralite II or methyl centralite, symmetrical dimethyl diphenylurea, $OC[N(CH_3)(C_6H_5)]_2$, also has been used as a stabilizer, but is not considered to be as effective as the ethyl analogue.

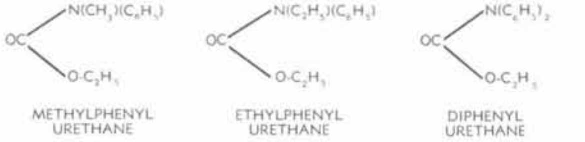
(4) Akardits, compounds with the structures indicated below, have been used extensively in German propellent compositions and are claimed to be more effective stabilizers than centralite or diphenylamine.



RA PD 212856

Figure Y. Akardits.

(5) Phenylurethanes, compounds with the structures indicated below, also have been used in German propellent compositions, but are considered to be less effective stabilizers than centralite. The methyl and ethyl compounds have greater gelatinizing action on nitrocellulose than centralite or the akardits.



RA PD 212857

Figure Z. Phenylurethanes.

(6) Petroleum jelly (petrolatum) has been used for many years by the British as a stabilizer for nitroglycerin-nitrocellulose propellants termed cordite. The stabilizing action of the material is ascribed to the presence of unsaturated hydrocarbons, which react with and neutralize acid products of decomposition.

h. Because of the high coefficient of acceleration of rate of decomposition of nitrocellulose (decomposition increases 3.7 times per each 10° C. rise) and the comparable effect of increase in temperature on the rate of decomposition of nitroglycerin, the more elevated the temperature used for testing propellants, the less representative the result is of stability at storage temperatures. For this reason, tests made at the lowest practicable temperature yield the most significant results. Since such tests require very long times for completion, they are not suitable for acceptance testing and no longer are used for surveillance testing. Acceptance tests are made at high temperatures and require but short times. They indicate solely if a given sample or lot has high temperature resistance characteristics normal for the composition, rather than stability in storage at ordinary or slightly elevated temperatures. The 134.5° and 120° C. heat tests are used for the acceptance testing of single-base and double-base propellants, respectively. The 65.5° C. surveillance test, once used for the purpose indicated by its name, is used for experimental purposes when an indication of true stability is desired.

(1) *65.5° C. surveillance test.* A 45-gram sample of the propellant is placed in a flint-glass bottle having a ground-glass stopper. The bottle is placed in an oven or chamber maintained at 65.5±1.0° C. (fig. 9–11). Visual inspection is made daily for the appearance of red fumes, which is considered completion of the test; and the test result is expressed as the number of days required to bring about the evolution of red fumes. Test values of 20 days or less are considered to indicate a condition of hazardous instability calling for immediate disposal of the propellant. Similar tests at 50° and 80° C. have been used experimentally. Although the procedure is lengthy, it has been found practicable, by means of periodic surveillance tests, to observe the gradual and then accelerated deterioration of propellants under adverse conditions of storage.

(2) *Heat tests.* The 134.5° C. heat test is used for testing single-base powders and nitrocellulose, while the 120° C. heat test is used in connection with double-base propellants. In either case, tests are made simultaneously of five portions of the sample. A 2.5-gram portion of the sample is placed in a pyrex glass test tube having an internal diameter of 15 mm, external diameter of 18 mm and length of 290 mm. A piece of normal methyl violet test paper, 70 mm in length and 20 mm in width is placed vertically in the tube so that its lower edge is 25 mm above the test material. The tube is stoppered with a cork that has a 4-mm diameter hole cut in it to prevent build-up of pressure within the tube. Each of the five tubes is placed in the appropriate constant temperature bath, maintained at 120.0±0.5 or 134.5±0.5° C. (fig. 9–12), which is so designed that no more than 7 mm of the tube projects from the well in which it is placed. At 5-minute intervals, each tube is withdrawn one-half its length and replaced quickly, any change in color of the test paper being noted. The time of observation that reveals the test paper in any of the five tubes to be completely changed to a salmon-pink color is recorded as the salmon-pink value of the sample. Heating is continued and the time of observation that reveals any of the tubes to be completely filled with red fumes is recorded. After the red fume value is determined, heating is continued further to determine whether explosion of the material in any of the five tubes takes place in less than 5 hours of heating. When the 134.5° C. heat test is used for testing nitrocellulose, only two tubes are processed, the first observation being made after 20 minutes of heating. Prior to testing, the nitrocellulose is dried for 4 to 5 hours at 40° to 42° C.,

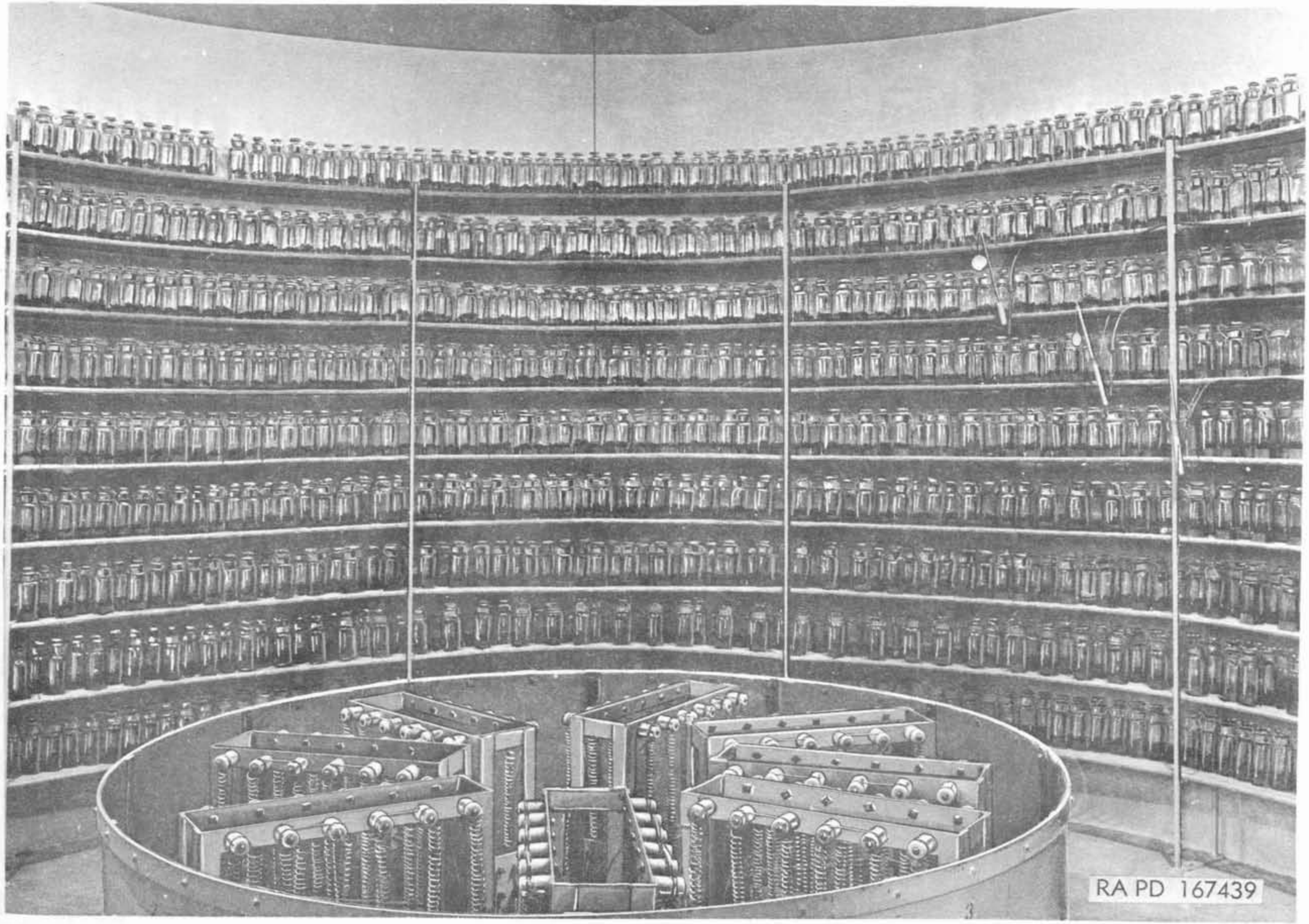


Figure 9–11. Surveillance test chamber.

or for 1 to 1.5 hours at 50° to 52° C., or overnight at room temperature, with further drying for 0.5 hour at 40° to 42° C. Prior to inserting the test paper in the tube, the 2.5-gram portion of the nitrocellulose sample is pressed into the lower 2 inches of the tube.

i. Representative surveillance and heat test values for standard propellent compositions are given in appendix A.

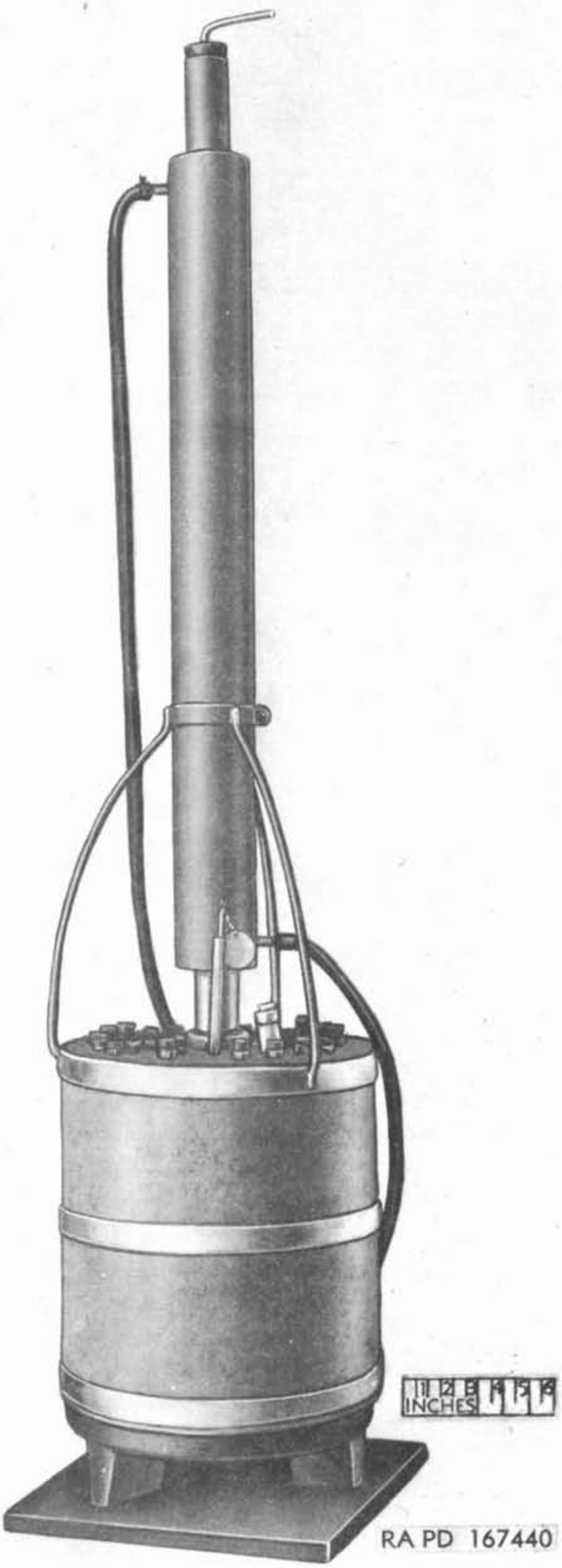


Figure 9–12. Apparatus for heat test at 120° or 134.5° C.

CHAPTER 10
UNITED STATES PROPELLANTS

10-1. Classes

a. The black powder originally used as a military propellant has been displaced completely by propellant compositions generally referred to as smokeless powders and almost invariably containing nitrocellulose. As these propellants are not used in the form of a powder and liberate varying amounts of smoke when burned, the term is a misnomer.

b. From the viewpoint of compositions, modern propellants can be divided into four classes. These are described in (1) through (4) below.

(1) *Single base*. These compositions contain nitrocellulose as their chief ingredient. In addition to a stabilizer, they may contain inorganic nitrates, nitrocompounds, and nonexplosive materials, such as metallic salts, metals, carbohydrates, and dyes.

(2) *Double base*. This term has been applied generally to compositions containing both nitrocellulose and nitroglycerin. However, in view of the purposes accomplished by nitroglycerin and the use aboard of substitutes for nitroglycerin, which serve the same purposes, a better definition of a double-base composition is one containing nitrocellulose and a liquid organic nitrate having the property of gelatinizing nitrocellulose. Like single-base propellant, double-base propellants frequently contain additives in addition to a stabilizer.

(3) *Triple base*. This term is applied to propellants containing three explosive ingredients with the major ingredient nitrocellulose. The other two explosive ingredients are usually nitroglycerine and nitroguanidine. As in the double base propellant, the nitroglycerine may be substituted by other liquid organic nitrates having the property of gelatinizing nitrocellulose. The nitroguanidine in the formulation produces a lower flame temperature, greater amounts of gases with lower flame temperature thus reducing tube wear while producing higher force. As in single and double base frequently contain additives in addition to a stabilizer.

(4) *Composite*. Most composite propellant contains neither nitrocellulose nor an organic nitrate. They generally consist of a physical mixture of a fuel such as metallic aluminum, a binder, normally an organic polymer (generally a synthetic rubber which is also a fuel), and an inorganic oxidizing agent such as ammonium perchlorate. These are heterogenous physical structures.

c. The use of propellant compositions is not in accordance with the foregoing classification. While single-base compositions are used in cannon, small arms, and grenades, double-base compositions are used in cannon, small arms, mortars, rockets, and jet propulsion units and triple base compositions are used in cannon units. Composite compositions are used primarily in rocket assemblies and jet propulsion units. The choice of a propellant for a specific use is determined by ballistic and physical requirements, rather than on the basis of composition. As a given composition may be suitable for use in several different applications, it is not practicable to classify propellants on the basis of use.

10-2. Single Base

a. *Pyrocellulose Powder*.

(1) The first nitrocellulose propellant standardized by the US Army and Navy was that termed pyrocellulose powder, because of the grade of nitrocellulose used in its manufacture. As first manufactured, pyrocellulose powder consisted only of carefully purified nitrocellulose having a nitrogen content of 12.60 ±0.10 percent and completely soluble in a mixture of ether and ethanol. The nitrocellulose was gelatinized by means of this solvent and extruded in the form of a cord with one or more perforations. This was air dried, so as to remove almost all of the volatile solvent, and the grains in the lot were blended thoroughly. The amount of *residual solvent* left in the powder varied from 1 to 5 percent, increasing with size of grain. About 1909, there was first added 0.5 percent of diphenylamine to act as a stabilizer, and during World War I, this ingredient was increased to 1 percent. This war period also saw the introduction of the *water-dry* process, which permitted the completion of manufacture in a fraction of the time required for the air-drying process.

(2) Pyrocellulose powder was unduly hygroscopic and gave bright flashes when fired in cannon. It was also considered to be of less than satisfactory stability, but defects in this direction were due largely to faulty manufacture and storage. It remained standard until the standardization of FNH (flashless, nonhygroscopic) and NH (nonhygroscopic) single-base propellants about 1930, when it became a substitute standard. Pyrocellulose powder was extensively manufactured and used by the Navy during World War II.

(3) Pyrocellulose powder was analyzed for nitrocellulose, diphenylamine, residual solvent, and external moisture contents. External moisture was determined by heating a sample at 100°C. Diphenylamine was determined by extraction with ether and nitration of the evaporated extract to the insoluble tetranitrodiphenylamine, which was weighed and calculated to the basis of diphenylamine. Nitrocellulose was determined by disolving the residue from the ether extraction in a 2:1 ether-ethanol mixture, precipitating the nitrocellulose by the addition of water and weighing the dried precipitate. By subtracting from 100 percent the sum of the percentages of nitrocellulose, diphenylamine, and eternal moisture, there was obtained the percentage of residual solvent present in the powder. This consisted of approximately 90 percent ether and alcohol and 10 percent water.

(4) Pyrocellulose powder was required to develop a salmon-pink color in not less than 60 minutes and not to explode in 5 hours when subjected to the 134.5°C. heat test. A minimum 65.5°C. surveillance test value of 140 days or less, depending upon the size of grain, was required for a time but was discarded after World War I. A compression test of individual grains in each lot was made to insure that the powder was not brittle. It was required that grains be compressed at least 35 percent of their length before cracks appeared.

(5) Pyrocellulose powder, as indicated by 65.5°C. surveillance tests, has less than half the stability life of the M1 and M6 powders, which displaced it. It has a calculated force value of 339,000 foot-pounds per pound, as compared with values of 314,000 and 328,000 for the M1 and M6 compositions. In spite of the satisfactory ballistic value of pyrocellulose powder, its hygroscopicity, the harmful effect of absorbed moisture on its stability, and the tendency to flash when fired in cannon render it less satisfactory than the M1 and M6 compositions.

b. E. C. Powder.

(1) This partially colloided propellant, one of the earliest nitrocellulose compositions developed, is used in caliber .30 blank ammunition. It has the composition —

	Percent
Nitrocellulose*	80
Barium nitrate	8
Potassium nitrate	8
Starch	3
Aurine dye	0.25
Diphenylamine	0.75

*Nitrogen content, 13.20 percent.

It consists of agglomerates having a rough surface. It is one of a type of propellants known as bulk powders used in shotgun shells. It is manufactured by either of two processes, in either of which only part of the nitrocellulose is gelatinized by means of a mixutre of acetone and ethanol or benzene or a mixutre of butyl acetate and benzene. In either case, the process is controlled, so that the product passes through a No. 10 sieve and a minimum of 97 percent passes through a No. 12 sieve and is retained on a No. 50 sieve.

(2) Because of the presence of some ungelatinized nitrocellulose, E.C. powder is distinctly more sensitive to friction than completely colloided powders. The ungelatinized nitrocellulose and potassium nitrate present also cause the powder to be much more hygroscopic than completely colloided powders. When exposed to an atmosphere having a relative humidity of 90 percent at 30°C., a representative sample absorbed 6.2 percent of its weight of water. Specification requirements permit the presence of 2.0 percent of moisture.

(3) Tests have shown E.C. powder to have the explosive characteristics shown in table 10-1.

Table 10-1. Explosive Characteristics of E.C. Powder

Impact test, cm	19
Pendulum friction test	Snaps
Explosion temperature test, °C.	199
Sand test, percent TNT value	107
Minimum detonating charge, gram mercury fulminate	0.22

These data indicate that the composition is sufficiently sensitive and powerful to be used as a high explosive as well as a propellant. It is because of this that the powder has been used in blank ammunition. E.C. powder, because of its high burning rate, is not used as a military propellant, but it has been used in shotgun shells, 65.5°C. surveillance tests have shown E.C. powder to be of the same order of stability as pyrocellulose powder.

(4) The specification for E.C. powder requires that it be of the prescribed granulation, comply with functioning test requirements when loaded in hand grenades and caliber .30 blank ammunition, and in the 134.5°C. heat test give a salmon-pink color in not less than 30 minutes and fail to explode in 4 hours.

(5) Analysis of E.C. powder: Barium nitrate is determined by oxidizing all organic material in a weighed sample, with nitric acid and precipitating barium as the sulfate. Potassium nitrate is determined by precipitating the potassium as dipotassium sodium cobaltinitrate, using the filtrate from the barium determination. Aurine dye is determined by extracting a sample of the powder with acetone and determining the aurine content

colorimetrically. Starch is determined by hydrolyzing the acetone-extracted sample, acidifying, adding a solution of potassium iodide, and titrating the liberated iodine with sodium thiosulfate solution. Diphenylamine is determined by steam-distilling a sample of the powder, extracting the distillate with ether, and precipitating the diphenylamine as tetrabromodiphenylamine. Nitrocellulose content is calculated by subtracting from 100 percent the sum of the percentages of the other ingredients found to be present in the moisture-free powder.

c. Flashless and Smokeless.

(1) The class of propellants known as flashless and smokeless (formerly designated as FNH and NH) comprises compositions that are used chiefly in artillery. Whether a composition is flashless depends upon the gun in which it is used. For example, the M1 composition is flashless when used in a 75-mm gun, but in the 8-in. gun, the propellant is not flashless. It would be desirable to have flashless propellants for all weapons, but his objective has not been attained as yet. Single base propellants of this class are not truly nonhygroscopic, but are so much less hygroscopic than pyrocellulose powder that moisture pick-up from external moisture during handling and storage is negligible. They differ from pyrocellulose powder, also, in having a much lower rate of moisture absorption as well as final moisture content when in equilibrium with air of given temperature and humidity.

Table 10-2. Standard Artillery Propellant Compositions

	M1 (percent)	M2 (percent)	M6 (percent)	M8 (percent)	M9 (percent)	M10 (percent)	M15 (percent)	M17 (percent)	M26 (percent)	M26A1 (percent)	M30 (percent)	M31 (percent)
Nitrocellulose (12.60%N)											28.0	20.0
Nitrocellulose (13.15%N)	84.2		86.1			98.0	20.0	22.0	67.25	68.70		
Nitrocellulose (13.25%N)		77.45		52.15	57.75							
Nitroglycerin		19.5		43.0	40.0		19.0	21.5	25.00	25.00	22.5	19.0
Nitroguanidine							54.7	54.7			47.7	54.7
Dinitrotoluene	9.9		9.9									
Dibutylphthalate	4.9		3.0									
Barium Nitrate		1.4							.75			
Potassium Nitrate		.75		1.25	1.5				.7			
Potassium Sulfate						1.0						
Cryolite							0.3	0.3			0.3	0.3
Ethyl Centralite		.6		.6	.75		6.0	1.5	6.0	6.0	1.5	
Diphenylamine	1.0		1.0			1.0						
Graphite		.3				0.1*			.3	.3		
Diethylphthalate				3.0								4.5
2-Nitrodiphenylamine												1.5
	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

*Graphite added.

(2) The M1 and M6 compositions were developed during the period between World Wars I and II, the remaining flashless and smokeless compositions being developed during and after World War II. The standard compositions of this type are shown in table 10-2. It will be noted that the M15, M17, M30 and M31 composition are triple base type, while the others are single or double base type. In all these compositions, the dinitrotoluene, and nitroglycerin act as gelatinizing and moisture-proofing agents and contribute ballistic potential to the composition. Dibutylphthalate and diethylphthalate are gelatinizing and waterproofing agents, but are nonexplosive and contribute to flashlessness. Nitroguanidine is used as a finely divided powder dispersed throughout the nitrocellulose-nitroglycerin colloid and contributes to ballistic potential as well as acting as a flash-reducing agent. Potassium sulfate, potassium nitrate and cryolite serve as flash-reducing agents, while the diphenylamine and ethyl centralite act as stabilizers.

(3) The manufacture of a single base propellant of this class involves a sequence of 10 or more operations. In the first, the wet nitrocellulose is dehydrated after the moisture content has been reduced to approximately 28 percent by wringing. Dehydration is accomplished by pressing the nitrocellulose at low pressure so as to squeeze out some water, adding 95 percent ethanol, and pressing at about 3,500 psi. There is obtained a block containing 25 pounds of dry nitrocellulose and about one-third this much of 90 percent ethanol. The wet block is broken up into small lumps by means of a rotating drum containing iron prongs and a screen. The nitrocellulose is transferred to a water-cooled mixing machine of the dough-mixer type and, while this is in operation,

ether equal to approximately two-thirds of the weight of dry nitrocellulose is added. Any plasticizing agents and stabilizers to be included in the composition are dissolved in or mixed with the ether prior to addition to the nitrocellulose. After addition of the ether is complete, materials such as potassium nitrate are added. Mixing of the ingredients is continued for about 1 hour. There is produced a partially colloided mixture, which resembles dry oatmeal in appearance. By pressing this at approximately 3,000 psi, so as to form a block, the degree of colloiding is increased rapidly. This effect is further increased and uniformity of the mixture is improved by subjecting it to a pressure of about 3,500 psi in a macaroni press. Here the material is squeezed through a series of screens and perforated plates and emerges in a form resembling that of macaroni. This is pressed again to form a block of well-colloided material. This is placed in a graining press and extruded through a carefully designed die by the application of pressure, the material emerging as a cord with one or more cylindrical perforations. By means of a cutting machine, the cord is cut into pieces of predetermined length. Removal of the volatile solvent, with shrinkage of the grains to their final dimensions, is accomplished by three operations. In the solvent recovery operation, the powder is placed in a large tank and warm air or other gas is passed through the mass. With careful control, so as to prevent surface-hardening, the temperature of the air is gradually increased to not more than 65°C. The solvent recovery operation requires from 2 to 14 days, depending upon the size of the grain, and reduces the solvent content to approximately 6 percent. The water-dry operation consists of placing the partially dried powder in water at about 25°C. and gradually increasing the temperature to a maximum of 55°C. After a number of days, the residual solvent is reduced to 0.3 to 5 percent, depending upon the grain size. The powder now is air dried to remove surface moisture and screened to remove dust and grain clusters. The final operation before packing is to blend all the powder in a lot, which may vary from 50,000 to 500,000 pounds depending on the type of powder. This is accomplished by transferring the powder from one bin to another by gravity flow, the bins being of conical shape. This blending improves uniformity of the lot with respect to composition and external moisture content.

(4) Propellants of the M1 and M6 class have maximum moisture absorption values of approximately 1 percent, as compared with 3 percent for pyrocellulose powder. While they have less tendency to flash when fired in cannon, they produce somewhat more smoke. This is a distinct disadvantage when firing is conducted during the daytime. The M1, M4, and M15 compositions have lower ballistic potential values than pyrocellylose powder. This is attributable to lower heat of explosion values, which also causes the development of lower flame temperatures and so tends toward less erosion of guns.

(5) Specification requirements for flashless and smokeless propellants include velocity and pressure effects when fired in a weapon and certain chemical and physical characteristics. In addition to having a specified composition, the maximum contents of moisture, volatile solvent, and ash are prescribed. The hygroscopicity of the propellant is required to not exceed a certain maximum, and multiperforated powders are required to have a minimum degree of compressibility without cracking. The single-base flashless and smokeless powders are required to comply with certain 134.5°C. heat test requirements, while propellants having the M15 composition are subjected to the 120°C. heat test. Grain dimensions are required to have certain relationships and be such as to yield the desired ballistic effects with a charge of suitable size.

(6) The methods of analysis of the several compositions comprising the flashless and smokeless class are so lengthy and varied that reference is made to the appropriate specifications. Hygroscopicity is determined as the percentage of moisture held when the powder absorbs no more after being exposed to an atmosphere of 90 percent relative humidity at 30°C. In the case of the M10 composition, hygroscopicity is determined as the percentage of moisture absorbed when powder is exposed to an atmosphere of 90 percent relative humidity at 30°C., after it has been brought into equilibrium with air of 20 percent relative humidity at 30°C. Compressibility is determined by cutting off the ends of grains, so that the length is equal to the diameter, slowly compressing the grain until the first crack in its wall appears, and measuring the degree of compression at this point. This is calculated to a percentage basis. 134.5°C. and 120°C. heat tests are made as described in paragraph 9-7.

d. Small Arms Powders.

(1) Both single- and double-base propellants now are used in small arms. The earlier type of single-base powder for this purpose was known as IMR. The compositions of this and other single-base powders are shown in table 10-3.

Table 10-3. Compositions of Single-Base Powders for Small Arms

	IMR (percent)	I (percent)	II (percent)	III (percent)	IV (percent)
Nitrocellulose	[a]97.4	[b]99.3	[b]98.3	[c]99.0 99.6	[d]98.95
Tin	2.0	......	......	......	......
Potassium sulfate	......	......	1	......	......
Diphenylamine	0.6	0.7	0.7	0.4 – 1.0	1.05
	100.0	100.0	100.0	100.0	100.0
Graphite	Glaze	Glaze	Glaze	Glaze	Glaze
Dinitrotoluene	[e]6.75	Coat	Coat	Coat	4.0

[a] 13.15 percent nitrogen.
[b] 13.15 percent or 13.25 percent nitrogen.
[c] 13.10 percent minimum nitrogen.
[d] 12.95 percent nitrogen.
[e] Applied as coating.

The tin and potassium sulfate present in these compositions acts as an anti-flashing agent. The dinitrotoluene coating acts as a moisture-proofing agent, causes the first phase of the burning process to take place at a relatively slow rate, and has some antiflashing action. The glaze of graphite is added to facilitate the uniform action of automatic loading machines and to avoid the development of large static charges in blending and loading. Composition II is used for loading the caliber .30 carbine grenade cartridges, as well as caliber .30 and caliber .50 rifle ammunition.

(2) The above single-base propellants are manufactured in the same manner as FNH and NH powders, except for the application of a coating of dinitrotoluene and glazing with graphite. The coating is applied by rotating the powder and dinitrotoluene in a rotating drum (sweetie-barrel) heated above the melting point of the coating agent. The glaze of graphite is applied to the coated powder by tumbling in a rotating drum.

(3) The single-base powders for small arms are of a high order of stability and are particularly resistant to the effects of atmospheric moisture because of their dinitrotoluene coatings. They have approximately the same ballistic potential as pyrocellulose powder.

(4) Each of the compositions is required to comply with certain requirements when subjected to the 134.5°C. heat test and not to be unduly hygroscopic. There are no requirements with respect to compressibility. The contents of external moisture and volatile solvent are kept within narrow limits, but the diphenylamine content is permitted to vary from 0.5 to 0.9 or 0.75 to 1.25 parts per 100 of nitrocellulose. The propellants are granulated as single perforated cylindrical grains. Each powder is required to comply with a gravimetric density requirement and have prescribed ballistic effects when fired in a standard weapon.

10-3. Double Base

a. Prior to World War II, double-base propellants were used in the United States for mortar and small arms ammunition but not in cannon. Since then double-base compositions have been standardized for use in the smaller guns and the requirements for rocket propellants have resulted in the standardization of a number of such compositions. Double-base propellants are manufactured by two methods. The solvent process is similar to that used for single-base powders, except that a mixture of ethanol and acetone is used as the solvent and the solvent recovery procedure is omitted because of the hazard involved in recovering solvents containing nitroglycerin. The nonsolvent process is used when the nitroglycerin and any other colloiding agents constitute approximately 40 percent of the composition. In this process the wet nitrocellulose is mixed with the nitroglycerin. Ethyl centralite is mixed in and the bulk of the excess water is removed by centrifuging. The resulting paste is put in cotton bags and subjected to heated air currents, to reduce the moisture content. The remaining constituents are then blended with the partly dried paste. Repeated rolling between the heated steel rolls removes the rest of the water and completes colloiding of the nitrocellulose. The thickness of the sheet formed is controlled carefully and varies with its use. If the sheet is to be cut into flakes for use in small arms or mortars, the thickness is between 0.003 and 0.0125 inch; but if the sheet is to be extruded in the form of large grains for use in rockets, it may be as thick as 0.125 inch.

b. The standard double-base cannon powders have the M2, M5 and M26A1 compositions shown by table 10-4. They are used in the form of perforated grains.

Table 10-4. Double-Base Cannon Propellants

	M2 (percent)	M5 (percent)	M26A1 (percent)
Nitrocellulose (13.15 percent N)			68.70
Nitrocellulose (13.25 percent N)	77.45	81.95	
Nitroglycerin	19.50	15.00	25.00
Barium nitrate	1.40	1.40	
Potassium nitrate	0.75	0.75	
Ethyl centralite	0.60	0.60	6.00
Graphite	0.30	0.30	.30
	100.00	100.00	100.00

These compositions have considerably greater ballistic potential than the single-base M1 and M6 compositions. As judged by their 65.5°C. surveillance test values (335 and 265 days), they are considerably less stable than the M1 and M6 compositions.

c. Double-base mortar powders include those used for propellant charges and those used in the ignition cartridges, both being in the form of flakes. The compositions of such powders are shown by table 10-5.

Table 10.5. Compositions of Mortar Powders

	M8 Increment (percent)	M9 Ignition (percent)
Nitrocellulose (13.25 percent N)	52.15	57.75
Nitroglycerin	43.00	40.00
Diethylphthalate	3.00	
Potassium nitrate	1.25	1.50
Diphenylamine		0.75
Ethyl centralite	0.60	
	100.00	100.00

The M8 and M9 compositions have very high ballistic potential values, because of their high nitroglycerin contents. They are the least stable of the standard propellants, having 65.5°C. surveillance test values of only 225 and 175 days, respectively. This is due in part to the small grain size, as powders having large specific surfaces have been found to give lower test values than those in large grains.

d. Double-base propellants for small arms have been used for many years. At one time these were of the ballistite type, used in flake, disk, and grain forms. Examples of such compositions are shown in table 10-6.

Table 10-6. Double-Base Propellants for Small Arms

	Ballistite (percent)	Bullseye (percent)	Infallible (percent)
Nitrocellulose (13.25 percent N)	60	59.6	60.25
Nitroglycerin	39	40.0	39.00
Diphenylamine	1	0.4	0.75
	100.0	100.0	100.0
Graphite	Coat	Coat	Coat

These compositions have been replaced by double-base compositions containing less nitroglycerin, such as those shown in table 10-7.

Table 10-7. Compositions of Caliber .30 and Caliber .50 Propellants

	Percent	Percent	Percent
Nitrocellulose*	74.70	76.55	84
Nitroglycerin	19.90	20.40	15
Starch	2.25	2.30	
Barium nitrate	1.50		
Potassium nitrate	0.90		
Diphenylamine	0.75	0.75	0.75
Graphite			0.25
	100.0	100.0	100.0

---

*13.15 or 13.25 percent nitrogen.

The single-perforated grains having these compositions are coated with dinitrotoluene or centralite and glazed with graphite. Although they have somewhat less ballistic potential than the ballistite type of powders, they are more stable, cause less erosion of rifles, and have less tendency to flash.

e. The standardization of the caliber .30 carbine permitted the use of a double-base composition in the form of spheres 0.02 or 0.03 inch in diameter instead of flakes or grains (fig. 10-1). Commonly called ball powder, it is produced by dissolving wet nitrocellulose in a solvent, such as ethyl acetate, adding diphenylamine and chalk, and then nitroglycerin. Upon agitation and the addition of a protective colloid, the solution is dispersed in the form of small globules. When the volatile solvent is removed by heating, the powder solidifies in the form of spherical pellets. Before removal of the water by screening, the powder is coated with a deterrent, such as dinitrotoluene, centralite, or diphenylphthalate. It may then be dried and graphited. A wide variety of single- or double-base compositions may be produced by this process. An example of the compositions manufactured for use in carbine ammunition is as follows:

	Percent
Nitrocellulose (13.2 percent N)	89.3
Nitroglycerin	9.0
Sodium sulfate	0.15
Calcium carbonate	0.65
Diphenylamine	0.90

Such powder is indicated by 65.5°C. surveillance test values to be as stable as M1 powder. Because of the rapidity and economy with which ball powder can be manufactured and the flexibility of the process, the future utilization of ball powder in directions other than small arms ammunition is probable.

f. Essentially all the propellants for rockets are of the double-base or composite type, as are those for JATO (jet-assisted takeoff) charges. Rocket propellants are manufactured in much larger grains than the largest cannon powder grains. The smallest rocket grain is 0.25 inch in diameter and 2.7 inches long. Rocket grains may exceed 260 inches in diameter and 50 feet in length. The smaller grains of rocket powder are manufactured by the solvent process, but the larger grains are produced by solventless cast, cast double base and cast composite processes. Representative compositions of rocket propellants are shown in table 10-8.

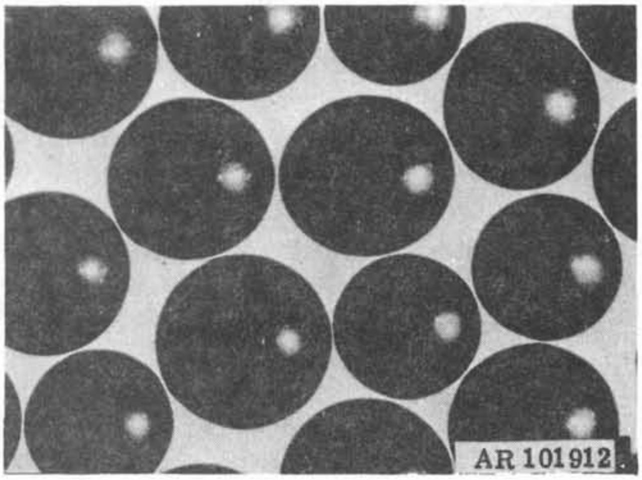


Figure 10-1. Ball powder, X25.

Table 10-8. Compostions of Rocket Propellants

	M7 (percent)	M5 (percent)	ARP (percent)
Nitrocellulose (12.6 percent N)		50.0	50.0
13.15 percent N)	54.7		
Potassium Perchlorate			
Potassium sulfate		10.5	
Carbon Black	1.2		0.2
Lead Salts		2.4	4.1
Triacetin			7.92
Ethyl Centralite	0.9		
2-Nitrodiphenylamine		2.0	1.67
Candelilla Wax	0.2		
	100.0	100.0	100.0

65.5°C. surveillance test values indicate the M7, M5 and ARP compositions to be of the same order of stability as pyrocellulose powder. The potassium perchlorate present in the M7 composition serves to increase the oxygen balance. Carbon black is used in the compositions, to improve uniformity of ignition and burning during its earliest phase. Lead salt is added to give more nearly constant rate of burning over the pressure range at which the powder is used (a so-called platonic agent). The specific impulse values of the above compositions vary widely. The use of such widely varying compositions is tailored to the ballistic requirements established for the rockets in which the propellants are used.

g. In addition to the usual requirements for propellants, rocket grains must be essentially free from fissures, pinholes, and similar structural imperfections. The presence of such physical discontinuities causes sudden change in the burning surface when these are reached during the burning process and may result in explosion and rupture of the rocket motor. While the smaller grains of rocket powder can be inspected visually for such imperfections, recourse is had to X-ray photography, fluoroscopy and ultrasonic testing for detecting significant voids, fissures, etc., in grains of the larger sizes.

10-4. Triple Base

a. Higher force nitroguanidine propellants were experimented with in the United States in the 1920's, but the present standard triple base propellant compositions (table 10-9) are based on the British Cordite N, disclosed to the United States during World War II. The American M15A1 propellant has about the same combustion temperature as the single base propellant M6 with correspondingly greater force for the M15A1. Since M15A1 has greater force than M6, a somewhat cooler propellant could be used effectively in guns chambered for M6 propellant.

Table 10-9. Triple-Base Cannon Propellant

	M15 (percent)	M17 (percent)	M30 (percent)	M30A1 (percent)	M31 (percent)
Nitrocellulose (12.60 percent N)			28.0	28.0	20.0
Nitrocellulose (13.15 percent N)	20.0	22.0			
Nitroglycerine	19.0	21.5	22.5	22.5	19.0
Nitroguanidine	54.7	54.7	47.7	47.0	54.7
Ethyl Centralite	6.0	1.5	1.5	1.5	
Diethylphthalate					4.5
2-Nitrodiphenylamine					1.5
Potassium Sulfate				1.0	
Cryolite	0.3		0.3		0.3
	100.0	100.0	100.0	100.0	100.0

b. For certain field uses, it is desired to give a projectile the highest possible muzzle velocity. The limitations on attainment of muzzle velocity by increasing propellant force is that gun erosion is a function of combustion temperature and increases rapidly above some temperature level. An economic balance between muzzle velocity and gun wear for weapons of 76mm or larger bore indicates that the cost in barrel life is too high to pay for added muzzle velocity if the gun chamber gas temperature is higher than about 3000°K. For rapid-fire weapons, the economic chamber gas temperature is considerably lower than 3000°K. Propellants containing nitroguanidine are considerably better than propellants without nitroguanidine in the attainment of force within a permitted maximum gas temperature.

c. The physical properties of the triple base propellants differ considerably from those of single base propellants in that the triple base propellants are opaque, chalky white unless glazed and generally exhibit lower physical strength. As the nitroguanidine-filled propellants are customarily manufactured by solvent extrusion, nonparalleled orientation of nitroguanidine crystals which are needle-like may cause bridging and to interfere with normal shrinkage during drying. The lower binder content may explain the physical properties deficiencies of these propellants.

d. The crystalline monopropellants such as nitroguanidine are generally more thermally stable than standard propellants, with the crystalline monopropellants becoming unstable only near their melting points. The aging of the triple base propellant, therefore, is considered to be due mainly to the nitrocellulose propellant binder. Stability tests dependent on measuring the loss in stabilizer content from a fixed weight of propellant generally indicate better stability for the triple base propellants than for ordinary single base propellants.

e. The manufacturing process used for the nitroguanidine triple base propellants in the United States has been uniformly solvent extrusion. The amount of solvent used is quite low and the propellant during extrusion is very soft so that it is sometimes necessary to dry the extruded strand partially before cutting, in order not to deform the cross section at the cut. Removal of solvent from the triple base propellant is rapid, due possibly to diffusion of solvent within the grain along the crystal-plastic interfaces. In order to make a good quality grain, it is necessary to use lower drying temperature gradients in order to avoid steep solvent gradients and resulting distortion and cracking.

10-5. Composite

a. The difficulty in manufacturing double-base rocket and JATO propellants in large grains and their undesirable variations in ballistic effects with change in initial temperature have led to the development of type of composition known as a composite propellant. Containing no nitrocellulose or nitroglycerin, it is an uncolloided, heterogeneous mixture consisting of an organic fuel, an inorganic oxidizing agent, and an organic binding agent.

b. A representative composite propellant is for the Pershing Missile which consists of:

		Wt. Percent
Ammonium perchlorate	oxidizer	68.0
Polybutadiene acrylic acid	binder	13.2
Aluminum	fuel	16.0
ERL-2795	Curing agent	2.8

This composition is manufactured by a simple mixing operation, cured and molded into grains, or poured directly into the rocket case and cured by heating in controlled ovens. Operational temperatures extend from -40° to 140°F. The high solid content requires great resin strength and good elastic properties to withstand low temperatures. Precise control of the oxidizer particle size is needed to control ballistic properties. Rockets, up to 260 inches in diameter, weighing up to 50 tons, have been fired successfully. The dense smoke produced limits these propellants to long range systems where the signature of the rocket does not easily reveal the firing point.

CHAPTER 11

FOREIGN PROPELLANTS

11–1. General

a. World War II witnessed the introduction of rocket propellants and their use by all the warring nations. The other major development in the field of foreign propellants was that of German and Italian modified double-base powders for artillery weapons and rockets. In these propellants, the nitroglycerin historically present in double-base propellent powders was replaced by diethyleneglycol dinitrate (DEGN) or, in some cases, by triethyleneglycol dinitrate (TEGN) or trimethylolethane trinitrate (metriol trinitrate).

b. The reason for the modification of double-base powders for ammunition for artillery weapons was that it was found possible, thereby, to increase very greatly the life of tube liners of these weapons. The high temperatures developed by nitroglycerin compositions caused rapid erosion and consequent short life of the tube liners. The Germans found that use of a nitroglycerin powder having a heat of explosion value of 950 calories per gram resulted in an effective gun life of 1,700 rounds; and when the nitroglycerin composition was modified so that the heat value was reduced to 820 calories per gram, the gun life was increased to 3,500 rounds. However, when the nitroglycerin was replaced by DEGN and the heat of explosion value thereby reduced to 690 calories per gram, gun life was increased to 15,000 or more rounds.

c. The benefits of this development were not confined to increase in gun life, as rocket propellants containing DEGN instead of nitroglycerin caused less nozzle-erosion and for this reason resulted in greater ballistic accuracy. In addition, since DEGN could be manufactured from synthetic organic chemicals and the production of nitroglycerin was dependent upon the production of glycerin from animal fats, the substitution of DEGN for nitroglycerin lifted a logistic ceiling.

d. The fact that DEGN is much more volatile than nitroglycerin is a disadvantage, particularly in hot climate warfare. For this reason, efforts were made to replace DEGN with somewhat similar less volatile liquid nitrates. TEGN and metriol trinitrate:

$$O_3NH_2C.CH_2.O.CH_2.CH_2.O.CH_2.CH_2NO_3 \text{ and } CH_3.C(CH_2NO_3)_3$$

were the two most promising materials developed for this purpose. However, the production of TEGN in adequate quantity proved difficult, and mentriol trinitrate was found to be such a poor plasticizer for nitrocellulose that it was necessary to use some metriol triacetate as a plasticizer.

e. A further development in the direction of double-base powders having low heat of explosion and high gas volume values was the development by the British and Germans of compositions in which nitroguanidine was substituted for part of the nitroglycerin or the DEGN. As nitroguanidine is essentially insoluble in the usual solvents and the nitrate esters, it is necessary that it be used in a very finely divided form and dispersed uniformly throughout the colloid formed by the rest of the composition.

11–2. British Propellants

a. Traditionally, British propellants have been of the double-base type and designated as cordites. Examples of those in use prior to World War II are the Mk I and MD cordites having the compositions shown in table 11–1.

Table 11-1. British Cordites

	Mk I (percent)	MD (percent)
Nitrocellulose*	37	65
Nitroglycerin	58	30
Mineral jelly	5	5
	100	100

*13.1 percent nitrogen content.

In such cordites, the mineral jelly (petroleum jelly or petrolatum) acts as a stabilizer, and a sample of the MD cordite has been found to be of apparently unchanged stability after 30 years of temperate-climate storage.

Table 11-2. Compositions of British Propellants

	Percent	Percent	Percent
Nitrocellulose	94.7	77.90	20.0
Nitroglycerin		14.85	19.0
Dinitrotoluene	4.1		
Nitronaphthalene		5.40	
Nitroguanidine			54.7
Tin	0.5		
Graphite	0.2	0.25	
Cryolite			0.3
Centralite	0.5	1.60	6.0
	100.0	100.0	100.0
(Percent nitrogen in nitrocellulose)	(13.1)	(13.0)	(13.15)

b. The necessities of World War II resulted in the British using single-base as well as double-base propellants, modifying cordite compositions so as to be suitable for use in rockets, and developing nitroguanidine compositions having low burning temperatures. Examples of the more modern British compositions are given in table 11-2.

11-3. German Propellants

a. During World War II the Germans used an extremely large number of propellent compositions of the single-base, double-base, and nitroguanidine double-base types. The double-base type included either nitroglycerin or DEGN as the liquid nitrate. In addition, there were at least two compositions that included PETN and constitute an unusual type of what must be considered single-base powder, since the PETN had not been dissolved, but was dispersed as discrete crystals.

b. Many stabilizers were employed in German propellants, in some cases singly and in other cases in combination with each other. Extensive use was made also of graphite, in some cases as a coating and in others as an ingredient. In one rocket powder composition, 0.3 percent of carnauba wax was used as a lubricant. Potassium sulfate was used as a flash suppressor in some artillery compositions. Dinitrotoluene or camphor was present in others and served as gelatinizing agents. Petroleum jelly was used as the stabilizer in one DEGN composition for 50-mm armor-piercing ammunition.

c. An usual feature of the use of German propellants was the employment of igniter powder bags made of nitrocellulose having a nitrogen content of 12.3 or 13.0 percent.

d. Tables 11-3, 11-4, and 11-5 give representatives of the various types of German propellent compositions.

Table 11-3. German Single-Base Compositions

	7.92 mm I (percent)	7.92 mm II (percent)	75 mm I (percent)	75 mm II (percent)	80 mm (percent)	Grenade (percent)	Pistol (percent)
Nitrocellulose	95.9	34.9	99.5	98.1	98.7	96.2	99.0
Dibutylphthalate						1.1	
Potassium sulfate						1.0	
PETN		64.7					
Graphite	0.5			0.1	1.0	0.3	
Camphor				1.8			
Centralite	3.6	0.4				1.2	1.0
Diphenylamine					0.3	0.2	
as-Diphenylurea			0.5				
	100.0	100.0	100.0	100.0	100.0	100.0	100.0
(Percent nigrogen in nitrocellulose)	(12.9)	(12.3)	(13.1)	(13.0)	(13.0)	(13.1)	(12.8)

Table 11-4. German Nitroglycerin Compositions

	37 mm (percent)	47 mm (percent)	80 mm (percent)	105 mm (percent)	150 mm (percent)	Mortar (percent)
Nitrocellulose	70.4	63.8	60.0	60.0	63.4	57.4
Nitroglycerin	27.3	30.0	39.0	38.8	33.0	41.6
Dinitrotoluene	—	—	—	0.4	—	—
Potassium sulfate	0.6	—	—	—	—	—
Graphite	—	—	0.2	—	0.1	0.2
Centralite	1.5	6.2	0.8	—	—	—
as-Diphenylurea	0.2	—	—	0.8	0.2	0.8
Ethylphenylurethane	—	—	—	—	1.5	—
Diphenylurethane	—	—	—	—	1.8	—
	100.0	100.0	100.0	100.0	100.0	100.0
(percent nitrogen in nitrocellulose)	(11.9)	(12.1)	(13.1)	(130.5)	(12.0)	(13.3)

Table 11-5. German DEGN and TEGN Compositions

	Percent	Percent	Percent	Percent	Percent	Percent
Nitrocellulose	[a]60.5	67.7	62.3	61.5	[b]43.5	58.5
DEGN	26.0	29.0	26.0	34.8	18.6	—
TEGN	—	—	—	—	—	25.1
Nitroguanidine	—	—	—	—	30.0	—
Dinitrotoluene	4.0	—	—	—	—	—
Nitronaphthalene	2.5	—	—	—	—	—
Hydrocellulose	3.0	—	—	—	—	—
Carnauba wax	—	—	—	0.3	—	—
Potassium sulfate	—	—	0.5	—	—	4.0
Graphite	0.10	0.1	0.3	—	0.1	0.1
Magnesium oxide	0.15	0.25	—	—	0.3	0.8
Petroleum jelly	—	—	3.5	—	—	—
Centralite	3.75	3.0	7.4	—	—	12.0
as-Diphenylurea	—	—	—	0.2	—	—
Akardite	—	—	—	—	0.5	—
Ethylphenylurethane	—	—	—	1.2	—	—
Diphenylurethane	—	—	—	2.0	3.25	—
Methylphenylurethane	—	—	—	—	3.75	—
	100.0	100.0	100.0	100.0	100.0	100.0
(Percent nitrogen in nitrocellulose)	(12.0)	(12.0)	(12.0)	(12.5)	(12.0)	(12.2)

[a] This composition, known as "universal powder," has a heat of explosion value of 730 calories per gram.

[b] This composition has a heat of explosion value of 750 calories per gram.

11-4. Italian Propellants

a. The propellants used by the Italians during World War II are very similar to those used by the Germans. The major differences are the lack of use of nitroguanidine and the use of metriol trinitrate by the Italians in formulating their compositions.

b. Examples of the compositions used by the Italians are shown in table 11-6.

Table 11-6. Italian Compositions for Propellants

	Percent	Percent	Percent	Percent	Percent	Percent
Nitrocellulose	97.0	64.0	62.0	64.0	*63.5	55.0
Nitroglycerin	—	28.0	33.0	—	—	—
DEGN	—	—	—	35.0	27.0	—
Metriol trinitrate	—	—	—	—	—	40.5
Cellulose acetate	—	—	—	—	5.0	—

Table 11-6—Continued

Phthalide		4.0				
Metriol triacetate						2.0
Camphor						
Graphite			0.3			
Tin						
Petroleum jelly		1.0	2.0			
Centralite	2.0	3.0	2.6	1.0	4.5	2.5
Diphenylamine	1.0					
as-Diphenylurea			0.1			
	100.0	100.0	100.0	100.0	100.0	100.0
(Percent nitrogen in nitrocellulose)	(12.5)		(12.1)	(12.25)	(10.0)	

*Cellulose acetate-nitrate.

11–5. Japanese Propellants

a. Because of the scarcity of animal fats from which to obtain glycerin and the necessity for using as fuel the petroleum that could have served as a raw material for the glycols during World War II, the Japanese used single-base propellants to a much greater extent than any other of the warring nations. At the end of the war, almost all of their technical data were destroyed prior to surrender, hence only a small amount of precise information became available thereafter.

b. Some known Japanese compositions are given in table 11–7.

Table 11–7. Japanese Compositions for Propellants

	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
Nitrocellulose	96.0	93.2	92.5	91.0	93.1	72.0
Nitroglycerin						19.5
Dinitrotoluene		5.5	5.0	8.0	5.0	
Tin			2.0			
Potassium nitrate						2.5
Graphite	0.2	0.3		0.4	0.4	
Diphenylamine	1.8	1.0	0.5	0.6		
as-Diphenylurea					1.5	6.0
	100.0	100.0	100.0	100.0	100.0	100.0
(Percent nitrogen in nitrocellulose)	(12.6)	(13.1)	(12.5)	(13.0)	(12.9)	(11.8)

11–6. Russian Propellants

a. In view of the one-way traffic in ammunition between the United States and Russia during World War II, and lack of informativeness on the part of the Russians, relatively little is known of the compositions of Russian propellants. Apparently, they used single-base powder for small arms, both single- and double-base powders for artillery, and double-base powder for rockets. There is no evidence that, like other nations, they used nitrated glycols or nitroguanidine in their propellants.

b. Known Russian compositions are given in table 11–8.

Table 11–8. Russian Compositions for Propellants

	76 mm A (*percent*)	*76 mm B* (*percent*)	*76 mm C* (*percent*)	*Caliber .30 rifle* (*percent*)	*Antitank Gun* (*percent*)
Nitrocellulose	98.9	99.3	98.7	96.9	64.4
Nitroglycerin					20.8
Dinitrotoluene					3.9

	76 mm A (percent)	*76 mm B (percent)*	*76 mm C (percent)*	*Caliber .30 rifle (percent)*	*Antitank Gun (percent)*
Graphite				0.3	0.3
Camphor				0.8	
Petroleum jelly					2.6
Centralite					1.3
as-Diphenylurea					6.7
Diphenylamine	1.1	0.7	1.3	2.0	
	100.0	100.0	100.0	100.0	100.0
(Percent nitrogen in nitrocellulose)	(12.7)	(12.6)	(12.8)	(13.0)	(13.1)

CHAPTER 12

IDENTIFICATION

12-1. General

a. Because of the use of inorganic as well as organic compounds as explosives and the presence of nitrate, nitro, nitramino, azide, diazo, and fulminic groups in these compounds, a systematic qualitative analysis of an unknown explosive is not practicable. The modern use of mixtures of explosives would render such an approach even more difficult. The identification procedures in *b* and *c* below and paragraphs 12-2 through 12-5, therefore, are based on the limited number of standard explosives and known characteristics that may be used to differentiate between these.

b. Before making any specific tests for identity, the unknown material should be examined for color, density, and particularly for sensitivity to flame, impact, and friction. The sensitivity tests may be made by subjecting a few grains of the material to the flame of a match and to impact and rubbing with a steel hammer, the material being placed on a steel block or hammer. Black powder flashes when exposed to a flame, but does not explode when struck lightly or rubbed with a hammer. Initiating explosives explode or flash when in contact with a flame and explode when struck or rubbed with a hammer. Solid noninitiating high explosives burn without explosion and are much less sensitive than the initiators to impact and friction.

c. Visual comparison of the color and crystal structure of the unknown material with the characteristics of standard explosives shown in parapraphs 7-1 through 7-29 will be of assistance in tentative identification. Should an unknown explosive be indicated to be of the sensitive type, special care should be taken in the removal of test samples, handling, and heating. Dense pressed pellets of explosive found to be, or suspected of being, of the sensitive type should be softened by soaking with water or alcohol prior to the removal of test samples.

12-2. Initiating Explosives

a. Lead Azide.

(1) Test the explosive for solubility in water. Lead azide is insoluble in water.

(2) Transfer 5 milligrams of the sample explosive to a 10-milliliter beaker and add 10 drops of a 10-percent ceric ammonium nitrate solution. A reaction accompanied with evolution of nitrogen gas is indicative of the presence of azide.

(3) Treat the solution of the sample obtained in (2) above with a few drops of a 10-percent solution of potassium dichromate. A yellow to reddish-yellow precipitate is indicative of the presence of lead.

(4) Transfer 2 milligrams of the sample to a 5-milliliter beaker. Add 5 ml of a 10-percent solution of ferric chloride solution. A red color is formed, which disappears slowly when a few milliliters of dilute mercuric chloride solution are added. This confirms that the explosive is lead azide.

b. Mercury Fulminate.

(1) Test the explosive for solubility in water. Mercury fulminate is insoluble in water.

(2) Transfer 10 milligrams of the sample explosive to a fritted glass crucible of medium porosity. Extract the sample with a 20-percent solution of

sodium thiosulfate, catching the washings in a 50-milliliter beaker containing 10 drops of a 1-percent phenolphthalein indicator solution. When the mercury fulminate is treated with solium thiosulfate, it dissolves with formation of alkali (NaOH), which changes the color of the indicator solution from colorless to red.

$$Hg(CNO)_2 + 2Na_2S_2O_3 + 2H_2O \rightarrow HgS_4O_6 + C_2N_2 + 4NaOH$$

(3) Transfer 10 milligrams of the sample to a 10-milliliter beaker and add 3 drops of concentrated hydrochloric acid solution and 2 milliliters of water. Transfer the solution to another beaker containing 1 drop of a 5-percent solution of potassium iodide. The formation of a bright red precipitate indicates the presence of the mercuric ion.

c. Diazodinitrophenol (DDNP). Dissolve 0.05 gram of the low-density greenish-yellow to brown explosive in acetone. Upon adding a larger volume of ice water, the explosive should appear as a bright yellow precipitate. Prepare a saturated solution of the explosive in 200 milliliters of water. Add to this 5 milliliters of a 20-percent solution of sodium hydroxide and mix. The evolution of a colorless gas and the appearance of a reddish-brown color in the solution indicate the explosive to be diazodinitrophenol.

d. Lead Styphnate. Wet 0.1 gram of the heavy light orange to reddish-brown material with 10 milliliters of water and then add slowly 10 milliliters of a 20-percent solution of ammonium acetate. Agitate the mixture until solution is complete. Add 10 milliliters of a 10-percent solution of potassium dichromate. The appearance of a bright yellow precipitate indicates the presence of lead. To 0.1 gram of the explosive in a beaker add 10 milliliters of a 10-percent solution of hydrochloric acid. Heat the mixture in a steambath and evaporate to dryness. Cool the beaker and contents and add 10 milliliters of ether. Mix the contents and allow to settle. Decant or filter off the ether solution and evaporate this to dryness at ordinary temperature. Dissolve the residue in **25** milliliters of water and add 0.1 gram of solid potassium cyanide. The absence of color indicates the explosive to be lead styphnate. Other tests will be included in revised specification JAN-L-757.

e. Tetracene. Wet 0.25 gram of the fluffy pale yellow explosive with 5 milliliters of a 10-percent solution of sodium hydroxide and warm the mixture in a steambath until solution is complete. Note if there is an odor of ammonia. Cool the solution and add 1 milliliter of a 5-percent solution of copper acetate. The appearance of a bright blue precipitate indicates the explosive to be tetracene. If 0.25 gram of the unknown explosive is not available, the identification could be carried out microscopically. See CWS Field Tab Manual 1-3-14 and MDRC Report on Microscopic Identification of Primers.

12-3. Noninitiating High Explosives

a. Many noninitiating high explosives can be identified by making a series of tests and comparing the results with those given in table 12-1. The tests are as indicated in (1) through (4) below.

(1) *Test 1.* Place 0.05 gram of the explosive in a 5-milliliter beaker, add 2 to 3 milliliters of distilled water and stir for 5 minutes.

(*a*) Observe color of liquid.

(*b*) Wet one end of a strip of Universal pH indicator paper and note any change in color.

(*c*) Add a drop of Nessler's reagent and note the color of any precipitate formed. Prepare the reagent by dissolving 5 grams of potassium iodide in a minimum quantity of cold distilled water and adding a saturated aqueous solution of mercuric iodide until a faint precipitate is formed. Add 40 milliliters of 50-percent potassium hydroxide solution. After the solution has clarified by settling, dilute to 100 milliliters with distilled water, allow to settle and decant.

(2) *Test 2.* Place 0.05 gram of the unknown material in an indenture of a white porcelain spot-test plate. Add two or three drops of a 65- to 68-percent aqueous solution of ethylene diamine and stir. Note the color of the solution (not the solid).

(3) *Test 3.* Place 0.05 gram of the unknown material in an indenture of a white porcelain spot-test plate and add three or four drops of a diphenylamine solution. Stir the mixture and, after 1 minute, note the color of the solution. Prepare the diphenylamine solution by dissolving 1 gram of diphenylamine in 100 milliliters of concentrated CP sulfuric acid.

Table 12-1. Tests of Noninitiating Explosives

	Water solution or extract			*Color effect of test with—*		
	Color	*Color of universal pH test*	*Color of PPT with Nessler's*	*Ethylenediamine*	*Diphenylamine*	*Thymol*
TNT	(Insoluble)			Maroon	Colorless	
Tetryl	(Insoluble)			Red	Blue	Green.
Picric acid	Yellow	Red	(No ppt)	Orange		
Explosive "D"	Yellow		Brown	Orange		
Haleite	None		(No ppt)	None	Blue[1]	Orange.
Nitroguanidine	None		White		Blue	Green.
Ammonium nitrate	None		Brown	None	Dirty green	Green.[1]
PETN	(Insoluble)			None	Dirty green	Green.
Nitroglycerin	None		(No ppt)		Deep blue	Green.
DEGN	None		(No ppt)		Deep blue	Brown.[2]
Nitrocellulose	None				Blue	Green.
Tritonal	(Insoluble)			Maroon	Colorless	
Tetrytol	(Insoluble)		(No ppt)	Maroon	Intense blue	Green.
Picratol	Yellow		Brown	Maroon		
Ednatol	None	Orange	(No ppt)	Maroon	Intense blue	Orange.
Amatol	None		Brown	Maroon	Dirty green	Green.
Ammonal	None		Brown	Maroon	Dirty green	Green.
Pentolite	None		(No ppt)	Maroon	Dirty green	Green.
Trimonite	Yellow	Red	(No ppt)	Orange	Red	
Tridite	Yellow	Red	(No ppt)	Orange		
Black powder[3]	None	No change			Blue	Green.

[1] Color appears immediately.

[2] Sometimes explodes mildly (puffs) upon addition of sulfuric acid.

[3] Tests of dried water extract.

(4) *Test 4.* Place 0.05 gram of the unknown material in an indenture of a white porcelain spot test plate. Add an equal amount of crystalline thymol and three drops of concentrated sulfuric acid. Stir the mixture and note its color after 5 minutes or more.

b. If the unknown material is not identified completely by the tests and data in paragraphs 12-1 and 12-2 and *a* above, tests should be made to determine whether it is one of those indicated in (1) through (8) below.

(1) *RDX.* Place 1 milligram of the white, unknown material in an indenture of a white porcelain spot-test plate and add not more than 1 milligram of thymol. Grind and mix the two intimately with the end of a glass stirring rod. Add three drops of concentrated sulfuric acid and stir. After 1 minute, add two drops of CP 95 percent ethanol, and stir the mixture. The appearance of a pink or rose color indicates the presence of RDX. If a blank test is made, a faint pink color is developed, but the effect of the presence of RDX is unmistakable.

(2) *Composition* A-3. Place 0.1 gram of the material in a 10-milliliter beaker and add two or three drops of acetone. Warm the mixture and allow to stand for 5 minutes. Evaporate the acetone by gently warming on a steambath, cool, and add 2 milliliter of carbon tetrachloride. Cover the beaker and warm the contents, occasionally swirling the mixture. Cool the mixture and allow the undissolved material to settle. Decant the supernatant liquid into a 5-milliliter beaker, evaporate to dryness, note if a waxy (not tarry) residue is obtained. Dry the undissolved material in the 10-milliliter beaker and test for RDX as described in (1) above.

(3) *Composition B*. Place 0.2 gram of the pale yellow to medium brown material in a 10-milliliter beaker and add 2 to 3 milliliters of chloroform. Cover the beaker. Warm and digest the mixture for 10 minutes with occasional swirling. Decant the supernatant liquid through a paper filter and evaporate the filtrate to dryness. Digest the insoluble residue in the beaker with three more portions of chloroform, discarding the decanted liquid in each case. Dry the insoluble residue by evaporating any adherent chloroform. If the original material was composition B, the residue from the chloroform solution consists of TNT and wax. Test the TNT and wax mixture with ethylenediamine and diphenylamine as described in *a*(2) and (3) above. The insoluble residue obtained by extraction with chloroform consists of RDX. Test it as described in (1) above.

(4) *Composition C-3*. Place 0.2 gram of the putty-like explosive in a 10-milliliter beaker and add 5 milliliters of benzene. Mix and digest for 10 minutes, crushing any lumps present. Decant the supernatant liquid the benzene with gentle heating. Note through a paper filter and evaporate whether a dark, tarry residue remains. Wash the insoluble residue left by benzene extraction with two or three 3-milliliter portions of a 2:1 ether-ethanol mixture and dry the washed residue. Test this as described under RDX ((1) above). To the decanted either-ethanol washings, add 15 milliliters of distilled water and heat the mixture until all ether and alcohol are removed. Note if there is separated a white precipitate, which is nitrocellulose. Catch the precipitate on a filter, wash with ethanol, dry by evaporation of the ethanol, and test for nitrocellulose as described in *a* above.

(5) *Torpex*. Place 0.2 gram of the explosive in a 5-milliliter beaker and extract with three 3-milliliter portions of acetone. Dry the insoluble residue and examine this under a microscope. Note if it has the characteristic appearance of metallic aluminum. Place 0.2 gram of the explosive in a 5-milliliter beaker and digest with two 3-milliliter portions of benzene, decanting the benzene into a small evaporating dish. Evaporate the benzene solution to dryness and test for TNT as described in *a* above. Dry the insoluble residue from the benzene extraction and test for RDX as described in (1) above.

(6) *Tritonal*. Place 0.2 gram of the explosive in a 10-milliliter beaker and add 5 milliliters of acetone. Stir, allow any undissolved material to settle, and decant the liquid. Wash the insoluble matter with two 5-milliliter portions of acetone, dry and examine under a microscope. Note if it has the characteristic appearance of metallic aluminum. Subject the explosive to the tests prescribed for TNT in *a* above.

(7) *Amatol*. Place 0.2 gram of the yellow

material in a 5-milliliter beaker, add 3 milliliters of distilled water, and stir for 5 minutes. Decant the liquid through a filter and evaporate to dryness. Test the dried solid as prescribed for ammonium nitrate. Dry the water-insoluble residue and test as prescribed for TNT in *a* above.

(8) *Ammonal.* Place 0.2 gram of the explosive in a 10-milliliter beaker and digest with 3 milliliters of distilled water. Decant the liquid through a filter and evaporate to dryness. Test the dried solid as prescribed for ammonium nitrate in *a* above. Digest the insoluble residue in the beaker with three 3-milliliter portions of acetone, decanting these through a filter. Dry the insoluble residue and examine under a microscope. Note if it has the characteristic appearance of metallic aluminum. Evaporate the filtrate to dryness by warming gently. Test the dried solid as prescribed for TNT in *a* above.

12–4. Black Powder

Place 0.2 gram of the black material in a 5-milliliter beaker, add 2 to 3 milliliters of distilled water, and stir for 5 minutes. Decant the liquid through a filter and catch the filtrate in a beaker. Evaporate this to dryness and subject the dried white solid to the tests shown in table 12–1. Dry the water-insoluble residue in the beaker, cool, and digest with two 5-milliliter portions of carbon disulfide, decanting these into an evaporating dish. Evaporate the carbon disulfide solution to dryness at room temperature. By means of a microscope, examine the yellow residue so obtained and the insoluble black residue from the carbon disulfide extraction. Note if they have the characteristic appearances of sulfur crystals and charcoal, respectively.

12–5. Propellents

Since the standard propellents are complex mixtures of nonexplosive as well as explosive compounds, and the separation of these compounds is rendered difficult by their dispersal in colloided nitrocellulose, simple identification tests for the various compositions are not practicable. Qualitative analysis of an unidentified propellent composition requires numerous solvent extractions, fractionation of the extracts, and tedious identification of the individual ingredients so isolated. The security classification of many of the standard compositions puts their identification outside the scope of this manual. The reader is referred to the applicable specifications for descriptions of the procedures involved in the analysis of such compositions.

CHAPTER 13

PYROTECHNIC AND TRACER COMPOSITIONS

13-1. General

a. Pyrotechnic compositions are low explosives that have but little exposure value, because of their low rates of combustion and the liberation of relatively gas per unit weight of composition. Solid products of combustion represent more of their mass than do the gases produced. They produce considerable light and so are used extensively for signal and illumination purposes. Although pyrotechnic compositions have been used for centuries for military purposes, such use did not become important until World War I, when the increased technical requirements of modern warfare necessitated the extensive use of signal and illuminating compositions. World War II saw the further development and application of such compositions for aerial observation, photography, and bombing purposes. Pyrotechnic compositions, today, are used not only for light but also for the heat, smoke, and/or sound. Therefore, tracer compositions, smoke compositions for signaling, spotting and tracking, delay fuze powder compositions, igniter compositions, and incendiary compositions could well be considered pyrotechnics. They all contain essentially similar ingredients, which undergo chemically similar reactions, namely exothermic reactions, which are used for the light, heat, smoke, and/or sound produced.

b. Physically, pyrotechnic compositions are mixtures of finely powdered elements and compounds, which are generally compressed in candle form. When signal and flare compositions burn, they radiate energy in the ultraviolet, visible, and infrared regions of the spectrum, but only the radiation in the visible region (4,000 to 7,000 Angstrom units or 400 to 700 millimicrons) is utilized for practical purposes.

13-2. Ingredients

a. The most important ingredients of a pyrotechnic composition are the fuel and the oxidizing agent. To these may be added other materials to intensify the color of the light produced, decrease the burning rate, act as a binding agent for the other ingredients, and serve as waterproofing agents. In some cases, one of the added materials may serve more than one of the foregoing purposes. The compositions generally are not stoichiometrically balanced, since the oxygen of the air plays a part in the cigarette burning of a pyrotechnic composition. This permits the use of an excess of fuel and consequently increased light intensity.

b. Powdered magnesium, aluminum, and alloys thereof are the fuels generally used but calcium silicide, charcoal, sulfur, silicon, zirconium, titanium, and metallic hydrides may be used. Materials added as color intensifiers, binding agents, and waterproofing agents also act as fuels if combustible.

c. The major oxidizing agent selected for use is usually determined by the desired color of light, luminous intensity, and burning rate. The nitrates of barium, strontium, sodium, and potassium, the perchlorates of ammonium and potassium, and the peroxides of barium, strontium, and lead are among the most important oxidizing agents that have been used. These are all compounds that can supply the oxygen necessary for combustion of most of the fuel present in the composition.

d. The most effective color intensifiers are compounds of chlorine. Organic chlorine compounds are preferable to inorganic compounds, because of the hygroscopicity of many inorganic chlorine compounds and their consequent incompatibility with the metals used as fuels. Very finely powdered copper has some value as a color intensifier when used in conjunction with an organic chlorine compound. Dechlorane, mexa chlorbenzene, polyvinyl chloride, and chlorinated waxes, rubbers, and plastics are the most important color intensifiers used. These agents are effective because of their decomposition during combustion and the formation of metallic chlorides, which produce characteristic color bands in the flame spectrum. The portion of the intensifier other than chlorine acts as a part of the fuel present in the composition. Since they are less reactive than metallic fuels, color intensifiers act to some extent as retardants of combustion. Certain intensifiers such as polyvinyl chloride, because of their physical characteristics, can serve also as binding agents.

e. Retardants are materials that are used to reduce the burning rate of the fuel-oxidizing agent mixture, with a minimum effect on the color intensity of the composition. Some retardants act only as inert diluents, while others take part in the combustion reaction at a much slower rate than the metallic fuels. Calcium carbonate, sodium oxalate, strontium resinate, titanium dioxide, polyvinyl chloride, ethyl cellulose, paraffin, linseed oil, castor oil, asphaltum, and sulfur are the most important retardants used. Certain of these serve other purposes as well. For example, sodium oxalate and polyvinyl chloride act also as color intensifiers, titanium dioxide is a source of incandescent solid particles in the flame produced by the composition, and the waxes and oils act as fuels, binding agents, and waterproofing agents, as well as retardants.

f. Binding agents are, generally, required in pyrotechnic compositions, because of the necessity for compressing these compositions into dense coherent candles and because of the nonadhesive characteristics of the metallic fuels and inorganic oxidizing agents used. Polyvinyl chloride, ethyl cellulose, metallic resinates, oils, waxes, and asphaltum have been used as binding agents. Binding agents of late have been polyester and sulfur plastics, which are polymerized in the pressed candle and vinyl alcohol acetate resin. As pointed out in *c* above, many of these serve a double purpose. In addition, they serve, in some cases, to reduce the sensitivity of compositions that otherwise would be unduly sensitive to shock and friction.

g. Waterproofing agents are necessary in many pyrotechnic compositions, because of the susceptibility of metallic magnesium to reaction with moisture, the reactivity of metallic aluminum with certain compounds in the presence of moisture, and the hygroscopicity of nitrates and peroxides. Waterproofing agents are applied as a coating on metallic fuels, such as a coating of dried linseed oil on magnesium, or as an ingredient uniformly distributed throughout the rest of the composition. In some cases, the metallic fuel is given a coating by treatment with a solution of acidic or acidified potassium dichromate. Waxes, resinates of metals, and natural and synthetic resins are used for distribution throughout the composition. Many of these serve also as binding agents.

13-3. Characteristics

a. The important functioning characteristics of a pyrotechnic composition are its luminous intensity (candlepower), burning rate, color, color value, and efficiency in light production. Necessary characteristics for photoflash compositions are peak intensity, time to peak, and integral light in the required exposure time, which at present is 40 milliseconds. Because pyrotechnic compositions are low explosives and must withstand loading operations, handling, and storage, other important characteristics are sensitivity to static, impact and friction, ignitibility, stability, and hygroscopicity. In other words, a composition must have acceptable explosives as well as pyrotechnic characteristics to be satisfactory for military use.

b. Luminous intensity is expressed in candles, the unit established by international agreement as equivalent to the light from the flame of a carefully specified tallow candle. The constancy of this unit is now maintained by means of standard incandescent light sources. Candlepower is luminous intensity in terms of candles. As the candlepower value of a composition varies almost directly with the area of the burning surface, it is customary to express luminous intensity in terms of candles per square inch.

c. Burning rate is expressed in inches per minute, when the composition burns after being pressed in cylindrical form and ignited at one end.

d. Color is a visual response of the normal human eye, which is most sensitive to yellow-green light and least sensitive to the violet and red rays near the ends of the visible part of the spectrum. Since the flames from pyrotechnic compositions emit light waves of a large number of different lengths, and since light of any one wave length produces a specific color sensation through the eye, the color and shade of color of a flame are determined by the summation of the effects of the different wave lengths. Spectrograms of green lights show bands in the blue and green regions of the spectrum, less intense bands and lines throughout the entire visible spectrum due to excited ions, and a continuous spectrum due to incandescent oxide particles. Red flame spectrograms have molecular bands in the red region representing strontium oxide and chloride, less intense lines and bands throughout the rest of the spectrum, and a white light continuum that dilutes the red color. Yellow flames, generally, owe their color to sodium lines and a continuum, but certain yellow lights are the result of a proper combination of red and green lights or the removal of blue light from white light.

e. Since changes in weather affect the apparent color as well as visibility of a flame, the color value (saturation) of such a light is a useful ratio. Color value is determined by using colored filters and an illuminometer. The designated color value is the ratio of the apparent light intensity, through the specified filters, to the total (white or unfiltered) intensity.

f. The light production efficiency of a pyrotechnic composition is calculated from its candlepower, burning rate, and density. It is expressed in candles per second per gram or per milliliter. It is useful in making comparisons of different compositions on a weight basis and, when expressed in terms of milliliters of pressed composition, it is of significance because of the volume limitations that are general in the design of pyrotechnic items.

g. Sensitivity and ignitibility are significant characteristics of pyrotechnic compositions for the practical reasons applicable to both low and high explosives.

h. While the ingredients used in the formulation of pyrotechnic compositions are each of a relatively high order of chemical stability, this does not assure similar stability of the physical mixtures of these ingredients. This is particularly true in the presence of moisture. New compositions, therefore, must be subjected to stability tests in the same manner as high explosives and propellants. Physical stability sometimes is critical. Because of slow oxidation of some of the minor ingredients, the pressed composition may become harder during storage. Extreme temperature changes during storage may produce a nonuniform composition. Such effects of physical instability cause marked changes in the burning rate and candlepower values of compositions and the uniformity of burning. New compositions, therefore, must be subjected to storage under adverse conditions, with subsequent functioning tests, to assure physical as well as chemical stability.

i. The critical nature of the hygroscopicity of pyrotechnic compositions is due not only to the adverse effects of the absorption of an inert diluent on the burning rate and candlepower characteristics, but also to the effect of moisure in promoting chemical instability. Powdered magnesium and aluminum, the principal metallic fuels, undergo reactions with moisture according to the following equations

$$Mg + 2\ H_2O \rightarrow Mg(OH)_2 + H_2$$
$$2\ Al + 6\ H_2O \rightarrow 2Al(OH)_3 + 3\ H_2$$

and absorbed moisture may bring about the reaction of other ingredients with these metals, their reaction products, or with each other. Spherical magnesium is less reactive with water because of the smaller specific surface, for the same granulation, and its higher purity. The frequently used nitrates of barium, strontium, and sodium in the presence of moisture react with the metallic fuels and cause deterioration of the composition. Strontium and barium nitrates can react with sodium oxalate in the presence of moisture to yield the much more inert oxalates of strontium and barium and the much more hygroscopic sodium nitrate. The hydrogen produced by the above reactions can reduce nitrates with the formation of inert hydroxides and gaseous ammonia.

j. The instability resulting from the absorption of moisture by a pyrotechnic composition may result in sufficient gas pressure, to cause distortion or rupture of the pyrotechnic assembly. The ignition of such pyrotechnic compositions may result in abnormally rapid burning or even an explosion. On the other hand, the effect of such deterioration through the action of moisture may be loss of ignitibility because of the formation of inert products of reaction.

k. While it is not practicable to formulate pyrotechnic compositions from only nonhygroscopic materials, the problem of hygroscopicity can be minimized by the use of less hygroscopic materials, the control of impurities that increase hygroscopicity, and the application of waterproofing agents. Examples are the use of less hygroscopic nitrates, the use of highly purified strontium nitrate instead of the technical grade, and the coating of powdered magnesium with linseed oil, which is air oxidized to a tough film prior to use.

13-4. Factors Affecting Characteristics

a. As in all other combustion processes, the rate of burning and the products of combustion of a pyrotechnic composition are affected markedly by the physical condition of the material and the extraneous conditions under which it is burned. Studies have shown the most important factors to be burning surface area, density (loading pressure) granulation of ingredients, purity of ingredients, type of candle case, and degree of confinement. All of these factors must be controlled within fairly narrow limits if reproducible results are to be assured.

b. With increase in the area of the burning surface (diameter of candle), there is an increase in total candlepower. Results of tests of a certain composition in this connection are given in table 13-1.

Table 13-1. Effects of Change in Area of Burning Surface

Burning area (sq in.)	*Total candles*	*Candles per (sq in.)*	*Burning rate (in./min)*
2.7	446,000	165,000	14.3
3.4	602,000	177,000	14.0
5.0	726,000	145,000	13.9
10.8	1,426,000	132,000	13.4

From these data, it appears that increase in burning surface causes decrease in the burning rate. This might be expected to result in a correspondingly uniform decrease in candlepower per square inch, but evidently there is an optimum burning area. With smaller burning areas, the rapid radiation of heat prevents the development of maximum candlepower in spite of maximum burning rate. With greater burning areas, less benefit from atmospheric oxygen is obtainable and the candlepower per square inch and burning rate decrease. These effects of change in burning area are not invariable and not equally pronounced for all compositions.

c. The density of a pyrotechnic composition, as used, is determined by the pressure employed in loading the composition in its case. Tests of compositions subjected to various loading pressures gave the results shown by table 13-2.

Table 13-2. Effects of Change in Density

Loading pressure PSI	*Composition A*		*Composition B*	
	Candles per sq in.	*Burning rate (in./min)*	*Candles per sq in.*	*Burning rate (in./min)*
5,800	78,300	9.8	41,400	7.0
10,000	81,800	9.1	39,100	5.9
14,400	90,000	9.0	41,400	5.9
17,600	93,000	9.0	39,000	5.3

From these representative data, it is apparent that, within practical limits, increase in loading pressure causes a decrease in burning rate and may cause an increase in candlepower value. Increase in candlepower value due to increased loading pressure is usual if the metallic fuel of the composition consists of magnesium, while the increase is not noted in aluminum compositions.

d. The burning characteristics of a pyrotechnic composition are significantly affected by changes in the granulation and particle shape of the principal ingredients. In general, as the granulation (particle size) is decreased, the rate of burning is increased because of the increase in total surface. At the same time, there is a more than proportional increase in luminous intensity, but the color value is decreased. Representative data in this connection are given in table 13.3.

Table 13-3. Effects of Change in Particle Size

Composition	Percent	Sieve number[1] Through	Sieve number[1] Held on	Sieve number[1] Through	Sieve number[1] Held on	Sieve number[1] Through
Barium nitrate	78	50	100	100	150	200
Aluminum, Grade A	3	50	100	100	200	200
Aluminum, Grade B	12	100	200	200	----------	200
Sulfur	5	80	----------	80	----------	80
Castor oil	2	----------	----------	----------	----------	----------
	100					
Burning rate, in./min	----------	3.1		3.2		3.4
Candles/sq in	----------	8,400		10,220		12,620

[1] *Sieve number*	*Sieve opening, inch*
50	0.0177
80	0.0070
100	0.0059
200	0.0029

e. Particle shape may have a substantial bearing on burning characteristics. Ground or flaked magnesium has a much greater specific surface than atomized magnesium of the same granulation because of the spherical shape of atomized magnesium particles. The burning rate values increase, with increasing surface area of magnesium. The substitution of atomized magnesium for ground magnesium of the same granulation has the effects on burning characteristics shown by table 13-4.

Table 13-4. Effects of Change in Specific Surface

Ground magnesium, percent	66.6	--------
Atomized magnesium, percent	--------	66.6
Sodium nitrate, percent	28.6	28.6
Resin, percent	4.8	4.8
	100.0	100.0
Candles per sq in	200,000	178,000
Burning rate, in./min	9.4	5.7
Density	1.56	1.65
Candle seconds/gram	50,000	69,200

f. From the foregoing, it is apparent that in order to obtain reproducible results from a given composition, it is necessary to exercise careful control over the granulations of the ingredients and in some cases the particle shape. The specifications for most ingredients of pyrotechnic compositions control the granulation by prescribed sieve tests and, where necessary, the particle shape is also prescribed.

g. Impurities in ingredients of pyrotechnic compositions may have adverse effects on the shade of color produced and so must be kept within well-defined limits. Usually, this is accomplished by prescribing maximum contents for impurities, which are known to be present in technical grades of materials and have harmful effects on burning characteristics.

h. The type of case into which a candle of pyrotechnic composition is loaded and burned has some effect on its burning characteristics.

The use of a metal case results in greater candlepower and burning rate values than when a paper case is used. Even different compositions of organic and easily combustible cases cause differences in burning characteristics, as shown by the test data in table 13-5 for a magnesium-sodium nitrate-resin composition.

Table 13-5. Effects of Combustible Cases

	Composition of case		
	Paper	Bakelite	Cellulose acetate
Candles per sq in	54,00	105,000	30,000
Burning rate, in./min	3.8	10.2	2.8

i. Because of the formation of gaseous products of combustion, the degree of confinement can have marked effects on the burning characteristics of a pyrotechnic composition. The greater the gas pressure, the greater will be the burning rate and luminous intensity, hence, under certain conditions the reaction may become so rapid as to result in an explosion. This is particularly true of photoflash compositions. Generally, pyrotechnic compositions are burned under conditions of atmospheric pressure, but if the normal burning rate is very high, only slight confinement is necessary to cause marked increase in the burning rate. In some cases, excessive slag formation and collection at the burning face can produce confinement and local explosions at the burning face.

13-5. Manufacture and Loading

a. Military pyrotechnic compositions are prepared by dry-blending or wet-mixing of the ingredients, the former being the more generally used method.

b. Metallic magnesium and magnesium-aluminum alloys to be used in pyrotechnic compositions were precoated with 3 percent by weight of linseed oil. However, because of the higher purity and smaller specific surface of atomized magnesium used in most flare compositions today, no linseed oil is necessary or used to coat the atomized magnesium. Where necessary, a more effective coating is obtained by immersing the metal powder briefly in a hot aqueous solution of sodium or potassium dichromate. As a result. there is formed on the surface of each particle a thin layer of insoluble chromate, which is a more effective moisture barrier than linseed oil, particularly at high storage temperature.

c. For dry blending, a conical blender having a capacity of 5 to 50 pounds is used. The blender is located in a well ventilated building or barricade with three concrete walls and is operated by remote control. All equipment must be grounded and every precaution taken to prevent the accumulation and discharge of charges of static electricity, as the dust produced within the blender makes it hazardous. The weighed ingredients are placed in the blender, together with bronze balls about 1 inch in diameter and weighing twice as much as the material to be mixed. For the more sensitive compositions, wooden balls or rubber stoppers are used. One-half hour of mixing, generally, is sufficient. The thoroughly mixed composition is transferred to airtight containers for temporary storage, prior to use in manufacturing pyrotechnic items. In large scale manufacture, cross-blending of batches is resorted to in order to insure uniformity throughout a lot of ammunition. This is accomplished by placing a portion of each batch in each of a number of containers and blending the contents of each container by means of the blender used for mixing the batch. For wet mixing, a Simpson intensive mixer is used, the mixing tools, mullers, and pan of which are made of stainless steel. The mixing tools, mullers, and scrapers are adjusted so as not to touch the pan. The weighed ingredients are placed in the pan and wet with a liquid, such as ethanol, acetone, or benzene. Sufficient liquid is used to form a thick paste. The operation of the mixer is continued until the composition becomes sufficiently granular to be screened but is not dry. The time required for this is from 20 minutes to 2 hours. The mixture is then granulated by screening, dried at about 80° C. in a steamheated oven, and transferred while warm to an airtight container.

d. Except for photoflash and some smoke compositions, which are used in a loose condition, pyrotechnic compositions are consolidated by pressing into cylindrical cases of paper or metal. For this operation, the case is held in a split brass or bronze mold. Loading is generally done in increments, employing a ram with a stepped face. In order to facilitate ignition of the pyrotechnic composition, the last increment loaded generally consists of a more ignitible "first fire composition." This is a blended mixture of one part of black powder and three parts of the pyrotechnic composition being loaded. The first fire composition of newer improved items, is a nonhygroscopic composition containing barium nitrate, silicon, zirconium hydride, tetranitrocarbazole, and laminac. The candles so manufactured vary from ½ to 6 inches in diameter and from ½ to 30 inches in length.

13–6. Testing

a. Performance Tests.

(1) *Consolidated compositions.*

(*a*) *Luminous intensity.* The luminous intensity of a linearly burning consolidated composition, be it a flare, signal or tracer, is determined in an experimental test vehicle or in the final item. Items to be tested are placed in a black walled chamber or hood located at the end of a dark test tunnel not less than 50 feet long. The candlepower value of the luminous intensity is determined by means of a Weston illuminometer. This instrument consists of a barrier layer photocell, filters, and a microammeter. The illuminometer is placed at a carefully measured distance from the point at which the candle or assembly is to be burned and is then calibrated by means of a standard incandescent white light source. During the burning of the item, illuminometer readings are taken at predetermined time intervals throughout the burning period and the average of these readings is calculated. The Westen illuminometer and its accessories are often replaced by a barrier layer cell equipped with filters and coupled to a graphic recorder. The output of the photocell is fed into the recorder which has been calibrated to give full scale readings for the anticipated candlepower output. A graphic record is superior to readings taken with the Weston illuminometer, because all variations during burning are recorded and thus are available for future study. The area under the curve thus obtained represents the candle-second value of the tested item. An integrator which gives the candle-seconds value of the composition directly, is coupled with the same photocell, thus avoiding the time-consuming and less accurate method of estimating or planimetering the graphic record.

(*b*) *Color value.* Color value is determined by the simultaneous use of a second photocell, equipped with a colored glass filter chosen so as to transmit only the wavelengths of light emitted by the color-producing spectral emitters in the flame. The color value is calculated as the ratio of the luminous intensity measured through the colored filter to the total luminous intensity. This method indicates any unusually low saturation of color, but measures only one spectral component and so does not determine the exact hue of the flame. The more precise method of color evaluation proposed by the ICI system uses three tristimulus values, $\overline{X}$, $\overline{Y}$ and $\overline{Z}$, for the various spectral colors. The values of $\overline{X}$, $\overline{Y}$ and $\overline{Z}$ are the amounts of the three ICI primaries required to color match a unit amount of energy having a specified wavelength. The $\overline{Y}$ tristimulus value was selected by conducting hundreds of tests on observers so that the average response curve of the human eye could be determined. The $\overline{Y}$ tristimulus curve is also used as the response curve for any photocell employed in photometry where luminosity or light intensity is measured. The $\overline{X}$ and $\overline{Z}$ curves were so selected that the areas they encompass are equal to that under the $\overline{Y}$ curve. Since the three tristimulus values are necessary to specify a color, a three-dimensional diagram would be needed to represent a color graphically. To avoid this, three other quantities were introduced, X, Y and Z. They are defined for spectrum colors as follows:

$$X=\frac{\overline{X}}{X+Y+Z}\quad Y=\frac{\overline{Y}}{X+Y+Z}\quad Z=\frac{\overline{Z}}{X+Y+Z}$$

and are called the chromaticity coefficients. From mathematical definition it follows that $X+Y+Z=1$, therefore, it is only necessary to specify two of the chromaticity coordinates to describe a color.

(c) *Burning rate.* The determination of burning time occurs simultaneously with the determination of candlepower and color value. To obtain burning rate, the burning time (obtained either by stopwatch or by measuring the baseline of the graphic record) divided into the previously obtained length of the composition candle, exclusive of the first fire layer, results in a value for burning rate; finally expressed in inches/unit time.

(2) *Non-consolidated compositions.*

(a) The performance characteristics of these high intensity short duration illumination sources in either experimental or end items are obtained by a coordinated instrumentation set-up, consisting of an oscilloscope and a camera. To obtain light output measurements the item is suspended above the ground, in an horizontal position, at a height sufficient to prevent any portion of the flash from hitting the ground. The item is oriented so that an end aspect is facing the photocell; the latter being positioned at a distance usually greater than 200 feet from the item, to prevent damage by fragments and to prevent saturation of the cell. A wire mesh neutral filter may be used to attenuate the luminous intensity of the flash to protect the cell. The output of the cell is fed into the oscilloscope which, prior to the test proper, was calibrated by a standard light source.

(b) The oscilloscope response is recorded photographically by either a still or a motion picture camera. Using a multibeam (2, 3, or 4) oscilloscope several ranges of luminous intensity may be recorded simultaneously or appropriate filters may be used on the photocells to obtain a spectral distribution of the luminous output. Usually one of the beams is modulated so as to produce a time record on the film. The output values of interest are the time-to-peak light, the peak intensity and the total duration, or the duration for some specific portion of the output. A similar arrangement may be placed at 90° to the end-on cell for side-on values.

(3) *Pyrotechnic delay compositions.*

(a) The performance characteristic of prime interest is burning time, which is obtained on the loaded item by means of a stopwatch or an electronic timer. The burning time interval is taken from the instant of initiation by squib or other initiating source and the appearance of a flash at the opposite end of the delay column.

(b) When an electronic timer is used,

the electrical pulse which functions the squib or initiator starts the counter. The flash at the end of the burning impinging on a photocell or ionization gage stops the counter.

(4) *Sound producing devices.* The intensity of sound producing devices such as whistles is determined by a calibrated decibel meter at a preselected distance. The frequency of the sound produced is obtained by a sound spectrum analyzer of frequency meter. The burning time is measured by stopwatch.

(5) *Incendiaries.* The heat output of an incendiary is determined by calorimetry, and is expressed as calories per gram. The effectiveness of an incendiary against various targets, such as wood, paper, and other combustibles is determined by attempting to ignite selected targets of known size and geometry by a fixed charge weight of the incendiary.

b. Additional Performance Tests. Tests such as transportation by truck and air, vibration, drop and temperature and humidity cycling are a few of the service tests conducted to determine the ability of an item to withstand rough handling and adverse environmental conditions. Specification MIL 5272C fully describes these as well as other specialized service tests.

c. Impact sensitivity and ignitibility tests of pyrotechnic compositions are made in the same manner as described for high explosives.

d. Laboratory stability tests of pyrotechnic compositions generally are 120° C. vacuum stability tests. These tests determine the inherent chemical stability of the composition but are insufficient for practical purposes because of the factors of hydroscopicity and physical instability involved in many pyrotechnic compositions. Consequently, a composition generally is tested for stability in storage by loading into an appropriate assembly and making functioning tests before and after storage for various periods under adverse conditions of temperature and humidity. Candlepower, burning rate, and color value determinations are those usually made in the course of the functioning tests. In this manner, the effects of physical instability and moisture absorption are detected as well as any chemical deterioration that may have occurred. Typical storage conditions employed are 50° C. for 2 years and several years at atmospheric temperature and humidity.

e. Hygroscopicity is determined by placing a weighed sample of the dry powdered composition in a humidor maintained at a fixed temperature and relative humidity and finding the increase in weight through moisture absorption during a certain period of time. Comparative values can be obtained at 30° C. and 90 percent relative humidity. A more informative method is to determine the critical humidity of the composition. This is the relative humidity at ordinary temperature below which moisture absorption is nil or negligible. Critical humidity is determined by placing weighed samples in a number of humidors covering a range of relative humidity and finding the minimum humidity above which moisture absorption is significant. Such data can be used for establishing whether a composition should be manufactured and loaded in rooms in which the relative humidity is maintained below a certain value.

f. Additional Laboratory Tests. Physiochemical changes which occur in pyrotechnic ingredients and compositions when subjected to heat, directly influence the ultimate performance of the compositions. Two complementary thermal analytical techniques can be used to observe and measure the changes that occur upon heating.

(1) The first, differential thermal analysis, determines the reactions that occur upon heating, such as transitions, fusion, boiling, and decomposition. A test sample and a thermally inert reference material, in individual containers, are heated simultaneously at a preselected rate in a vertical furnace. The differential output of thermocouples placed in each sample is recorded on one axis of an X-Y re-

corder, while the furnace temperature is recorded on the other axis. Analysis of the valleys and peaks obtained on the thermogram indicate the changes that have occurred upon heating. Endothermic reactions are indicated by a dip or valley in the curve while exothermic reactions are indicated by an upward trend or by peaks.

The second technique, thermogravimetric analysis indicates changes in the sample when heated at a preselected rate. A crucible containing the sample is placed on the pan or arm of a thermobalance and heated, while any weight changes that occur are detected by a transducer and the resultant output is recorded as a function of the furnace temperature. Analysis of the thermogram indicates loss of volatiles, decomposition, oxidation or vaporization.

13-7. Uses

a. Pyrotechnic compositions are used in a wide variety of ammunition items, many specialized uses having been developed since World War I. The most important uses are in projectiles, flares, photoflash cartridges and bombs, signals, tracers, simulated ammunition, target identification bombs and delays.

b. Illuminating projectiles, fired from artillery and 60-mm and 81-mm mortars are used to illuminate enemy territory at night. The compositions given in table 13-6 are representative of those used in illuminating projectiles.

Table 13-6. Compositions Used in Illuminating Projectiles

		Percent by weight			
	60-mm	81-mm	4.2 Inch	105-mm	155-mm
Sodium nitrate	36	36	36.3	36.3	38.5
Magnesium, Type I, 30/50	55	55	56		
Magnesium, Type III, Gran 18	----	----	----	----	53
Laminac A*	9	9	7.7	7.7	8.5
	100	100		100	100
Candlepower	320,000	500,000	850,000	450,000	1,000,000
Burning time, seconds	32	60	90	60	120

*Binder composition:

Alkyd-Styrene Resin	97.9%
Lupersol DDM	1.5%
Cobalt Naphthenate	0.6%

c. Flares are used for illuminating purposes and embrace a wide variety of tactical uses. In general, flare compositions produce yellow-tinted white light, as this has been found to be superior to pure white light with respect to visual observation. Trip, airport, ground, aircraft parachute, reconnaissance and landing, observation, bombardment, and tow-target flares are the principal ones used.

d. Trip flares are used to prevent enemy infiltration and are designed for imbedding just below the surface of the ground or attachment to tree or poles. A representative trip flare composition and its characteristics are shown in table 13-7.

Table 13-7. Typical Trip Flare Composition

	Percent (by weight)
Laminac A	9
Magnesium, Type I, 30/50	46
Sodium nitrate	45
	100.0
Candlepower	40,000
Burning time, seconds	55-70

e. Airport flares are used to provide illumination for landing airplanes in fog or rain or at emergency landing fields. They are designed to yield light intensities of 40,000 to 1,000,000 candles. Table 13-8 shows a representative composition.

Table 13–8. Composition Used in Airport Flares

	Percent		Percent
Aluminum, Grade A	2	Candlepower	60,000
Aluminum, Grade B	20	Candlepower per sq in	50,000
Barium nitrate	62	Burning rate, in/min	5.0
Strontium nitrate	11	Explosion temperature test, ° C.	600+
Sulphur	3.5		
Linseed oil	1.5		
	100.0		

f. Aircraft parachute flares are used for emergency night landings, observation purposes, and bombing operations where special techniques are applied. Representative compositions are given in table 13–9.

Table 13–9. Composition Used in Aircraft Parachute Flares

	Percent	Percent	Percent
Magnesium, Type III, 30/50	52	48	58
Sodium nitrate	39	42	37
Laminac A	9	8	5
Polyvinyl chloride	--------	2	--------
	100	100	100
Candlepower	1,900,000	1,500,000	3,000,000
Candles per sq in	165,000	72,000	156,000
Burning rate, in./min	6	3	6.0

g. Tow-target flares are towed behind airplanes by means of a long steel cable and provide practice targets for antiaircraft gun crews. The compositions used in tow-target flares are very similar to or identical with those used in trip flares.

h. Photoflash cartridges and bombs are used for nighttime aerial photography. For photography from altitudes up to 3,500 feet, a cartridge is used to obtain a peak light intensity of 120,000,000 candles and a total light of 1.4 million candle-seconds in 0.04 second. The composition used contains a 30/40/30 mixture of barium nitrate, aluminum, and potassium perchlorate. Ignition is effected by a small charge of lead azide. For high altitude photography, there is used a bomb containing 85 pounds of the same composition. This gives a peak intensity of 3 billion candles with a total light of 85 million candle-seconds in 0.04 second. The loose composition used is considerably more sensitive to spark and friction than most pyrotechnic compositions, consequently, great care must be taken in blending and loading this composition. Recent type photoflash bombs are loaded with a metal dust of atomized magnesium or magnesium-aluminum alloy powder, ignited by a central explosive burster in the form of metal dust explosion. This type of photoflash minimizes the vulnerability to fragment and bullet penetration. Current emphasis is on development of flash mixtures containing calcium, which are more eflective at higher altitudes than other photoflash mixtures.

i. Pyrotechnic signals are used extensively for communication between various elements of ground troops, ground troops and airplanes or vice versa, and airplanes in flight. White, green, yellow, and red lights are used alone or in combinations in the forms of stars, clusters, and blinkers. Tracers are also used as aircraft signals. Compositions employed in ground signals appear in table 13–10; in aircraft signals, in table 13–11.

j. In use, parachute signals burn for 20 to 30 seconds and have light intensities of 5,000 to 25,000 candles. Cluster signals burn for only 5 to 7 seconds, with luminous intensities of 2,000 to 35,000 candles. Blinker signals burn from 30 to 60 seconds, with light outputs of 2,500 to 15,000 candles.

Table 13–10. Compositions Used in Ground Signals

	White (percent)		Green (percent)		Red (percent)		
	No. 1	No. 2	No. 1	No. 2	No. 1	No. 2	No. 3
Magnesium, Type 1, Gran#4	--------	--------	--------	--------	--------	--------	17.0
Magnesium, Type 1, Gran#11	--------	--------	--------	16.0	--------	--------	2.0
Laminac A	5.0	5.0	5.0	--------	4.0	7.0	
Barium nitrate	--------	49.0	46.0	59.0			
Strontium nitrate	--------	16.5	--------	--------	48.0	42.0	4.30

Table 13-10—Continued

	White (percent)		Green (percent)		Red (percent)		
	No. 1	No. 2	No. 1	No. 2	No. 1	No. 2	No. 3
Potassium perchlorate	-------	-------	-------	-------	-------	9.0	21.4
Polyvinyl chloride	-------	-------	16.0	-------	15.0	12.0	
Hexachlorobenzene	-------	-------	-------	21.0	-------	-------	6.4
Copper	-------	-------	-------	2.0			
Sodium nitrate	38.0						
Asphaltum	-------	-------	-------	-------	-------	-------	7.4
Linseed oil	-------	-------	-------	2.0			
Graphite	-------	-------	-------	-------	-------	-------	2.8
Magnesium, Type 1, 30/50	57.0	29.5	33.0	-------	33.0	30.0	
	100	100	100	100	100	100	100

Table 13-11. Compositions Used in Aircraft Signals

	Red (%)	Yellow (%)	Green (%)
Magnesium, Type I, Gran. #11	18.0	18.0	17.0
Potassium perchlorate	24.0	16.0	13.0
Strontium nitrate	44.5	8.0	-----
Linseed oil	0.5	4.0	4.0
Hexachlorobenzene	6.0	12.0	13.0
Asphaltum	7.0	-----	-----
Barium nitrate	-----	25.0	53.0
Sodium oxalate	-----	17.0	-----

13-8. Visibility

a. The shorter the wavelength of light, the less its transmissibility through air, and the greater its absorption by particles of dust and moisture in the atmosphere. These factors result in considerable differences in the perceptibility of signals of different colors, even though they have the same luminous intensity and are viewed from the same distance. Adverse weather conditions magnify these differences. Illustrative of these variations in the visibility of signals are the results of tests at 1,468 yards given in table 13-12.

Table 13-12. Candlepower Requirements for Visibility

	Minimum candlepower for visibility Red	Amber	Green	White
Nighttime:				
Clear sky and air	0.09	0.18	0.25	0.23
Overcast sky, slight fog	0.29	0.37	0.53	0.28
Daytime:				
Clear sky and air	430	680	900	1000
Overcast sky, slight fog	180	190	360	290

From these data it is apparent that red light is the most easily visible under both nighttime and daytime conditions. The effects of adverse weather conditions are shown by the test data in table 13-13.

Table 13-13. Relative Intensities of Light Required for Visibility at Night Under Various Weather Conditions

Color of light	Clear sky	Light rain	Heavy rain	Light fog	Dense fog	Light snow	Heavy snow
Red	1.0	1.2	8.9	3.2	No Transmission	222	No Transmission.
Amber	2.0	2.1	33.5	4.1	do	835	Do.
Green	2.8	3.2	33.5	5.9	do	567	Do.
White	2.5	3.0	132.0	3.1	do	1,556	Do.

b. Blue light has such poor transmissibility characteristics that its use in pyrotechnic signals is impractical. This is particularly true in view of the impracticability of formulating compositions to give blue light of high luminous intensity. Violet light has been used to a slight extent, but it also has such poor transmissibility that its use is negligible.

c. Pyrotechnic compositions are used in a variety of other items, such as tracers, smoke signals, incendiary ammunition, whistles, gunflash simulators, V-2 rocket igniters, fuel igniters for ramjet engines and guided missiles, pathfinder beacons, etc. Use in the first three of these types will be dealt with in paragraphs 13–9 through 13–15.

With the growing complexity of the techniques of warfare, it may be anticipated that the future will witness an even more extended use of pyrotechnic compositions.

13–9. Smoke Compositions

a. These are mixtures of materials that undergo autocombustion with the liberation of a marked amount of smoke but no visible light. Because they are used for signaling purposes, they are classified as pyrotechnic compositions, but in reality they differ from true pyrotechnic compositions because of the relatively low temperatures developed and the absence of visible light during burning.

b. Smoke compositions may be divided into two types: those in which a product of the combustion reaction forms a dense voluminous smoke and those in which the heat of combustion of most of the ingredients serves to volatilize another ingredient to a smoke. Examples of these are the production of zinc oxide by the oxidation of metallic zinc and the volatilization of a colored dye. Smoke compositions frequently are loaded in a loose uncompressed condition.

c. Signaling by means of smoke is dependent upon the reflection of daylight and therefore is limited to daytime. White, black, and a wide range of colored smokes (except violet) can be used for this purpose. Smoke signals may be in the form of rifle grenade ammunition or drift signals. The rifle grenade signals produce smoke puffs that persist for 20 to 30 seconds.

d. Representative smoke compositions are given in table 13–14.

Table 13–14. Compositions Used in Smoke Ground Signals

	White (percent)	*Green (percent)*		*Yellow (percent)*	*Red (percent)*	
		No. 1	*No. 2*		*No. 1*	*No. 2*
Sugar	---	17.0	22.0	16.0	26.5	20.0
Ammonium perchlorate	27.6					
Potassium chlorate	---	28.0	31.0	26.0	35.0	31.0
1.4-D1-P-Tolvidinoanthraquinone	---	---	30.7			
Zinc oxide	34.6					
Sodium bicarbonate	---	---	3.0	---	1.0	
No. 3 Green solvent dye	---	36.0				
Yellow smoke B-10	---	---	10.8			
Asbestos	---	---	2.5	---	1.5	
Vinyl alcohol acetate resin	3.5	2.0	---	2.0	---	2.0
Aluminum	3.6					
Dechlorane	30.7					
Red dye (1-Methylaminoanthraquinone)	---	---	---	---	36.0	47.0
Yellow dye (Indanthrene golden yellow)	---	17.0	---	56.0		
	100	100	100	100	100	100

e. If the composition is one that yields a smoky product of combustion, the burning rate can be 5 to 6 inches per minute. In cases where a dye is to be volatilized with minimum decomposition, the combustion temperature and therefore the rate of burning must be kept to a minimum. Burning rates of 1.5 to 2 inches per minute are usual for such compositions.

f. Smoke compositions are not unduly sensitive and are of good stability although not unaffected by absorbed moisture. Their manufacture generally consists of simple mixing, the ingredients sometimes being dampened with a nonflammable liquid so as to reduce the friction and spark sensitivity of mixtures of fuels and oxidizing agents.

13–10. Tracer Compositions

a. General.

(1) Tracer ammunition for both small

arms and artillery is used for determining range and directing fire. In small arms cartridge tracers, the rear of the bullet has a cavity into which are loaded tracer and igniter compositions, using a pressure of 30,000 to 125,000 psi. Some artillery projectiles have a cavity in the base, into which tracer and igniter compositions are loaded under a pressure of about 110,000 psi. Other artillery shells have a separate tracer component, which is loaded and screwed into the projectile. In some types of artillery ammunition, the tracer composition serves also to provide self-destruction of the projectile after a definite time interval when fired. In such cases, a black powder relay at the bottom of the tracer cavity is ignited by the burning tracer composition, and the flame from the black powder primes an initiating charge, which detonates the high explosive bursting charge in the shell.

(2) In effect, the tracer composition is ignited by the hot gases resulting from burning of the propellent charge. However, since tracer compositions are relatively difficult to ignite, there is loaded on top of the tracer composition a much more easily ignitible igniter composition. This is generally done with a stepped ram. As a brilliant light from the igniter composition may dazzle the eye of the gunner and the completely visible trajectory of the round would betray the position of the weapon, a dim or delay igniter composition is used. Igniter as well as tracer compositions are classified as pyrotechnic compositions because of their compositions and their production of light when burned.

b. Compositions. Tracer compositions, which give red and white lights, are used in ammunition. Tracers burn for 3 to 20 seconds and have intensity values of 200 to 2,000 candles. Examples of tracer compositions are shown by table 13-15.

Table 13-15. Typical Tracer Compositions

	Red (percent)					White (percent)	
	No. 1	*No. 2*	*No. 3*	*No. 4*	*No. 5*	*No. 1*	*No. 2*
Dechlorane	-------	7					
Magnesium	27	42	-------	-------	28	-------	35
Magnesium-aluminum alloy	-------	-------	40	37			
Chlorinated rubber	-------	-------	5				
Strontium peroxide	26						
Strontium oxalate	5						
Strontium nitrate	33	44	55	56	55	-------	32
Barium peroxide	-------	-------	-------	-------	-------	-------	31
Calcium resinate	9	-------	-------	-------	-------	-------	2
Polyvinyl cloride	-------	-------	-------	7	17		
Polyethylene	-------	7					
Vinyl alcohol acetate resin	-------	-------	-------	-------	-------	1	
Potassium perchlorate	-------	-------	-------	-------	-------	30	
Zerconium	-------	-------	-------	-------	-------	69	
	100	100	100	100	100	100	100

13-11. Stability

The tracer compositions in paragraph 13-10 are quite stable with respect to heat, but some are particularly susceptible to deterioration by the absorption of moisture. This is because of the ready hydration of peroxides and magnesium. Accordingly, they must be loaded in rounds designed for protection against the absorption of moisture present in the adjacent propellant charge. Deterioration of the tracer composition results in loss of ignitibility and may cause swelling of the projectile.

13-12. Manufacture

Tracer compositions are generally manufactured by the wet method described and by simple dry blending of ingredients. Although dry blending is preferred for simplicity of processing, certain compositions must be wet blended (see wet method described) to obtain best results.

13-13. Tests

Tracer compositions are tested in the same manner as other pyrotechnic compositions except that the high pressures used for the loading of tracers makes the use of similar pressures desirable in the preparation of test samples. Accordingly, test samples generally are loaded in tracer bodies instead of candles. This permits tests under rotation, as well as static tests. The rotational test also employs an air blast to duplicate flight conditions. Most tracer compositions burn more rapidly under rotation then when burned statically. Selected samples of small arms ammunition are fired periodically over a prescribed range for general function, visibility, and length of trace.

13-14. Igniter Compositions

Igniter compositions used in conjunction with tracer compositions are designed to have much lower ignition temperatures and produce very little gas. This latter feature makes them much less luminous than the tracer composition. If practically no luminosity is developed, the composition is known as a dim igniter composition. Examples of igniter compositions are given in table 13-16.

Table 13-16. Igniter Compositions

	C (%)	D (%)	A (percent)	B (percent)	Dim (percent)		Delay (percent)
					A	B	
Magnesium	-------	16.5	17.0	12.0	6.0		
Magnesium-Aluminum Alloy	37.0						
Manganese	-------	-------	-------	-------	-------	32.5	
Barium peroxide	-------	80.5	81.0	87.0			
Lead chromate	-------	-------	-------	-------	-------	50.0	
Sulfur	-------	-------	-------	-------	-------	17.5	
Zinc stearate	-------	-------	-------	1.0			
Calcium resinate	-------	2.0	2.0	-------	9.0	-------	10.0
Polyvinyl chloride	7.0						
Graphite	-------	1.0					
Strontium nitrate	56.0						
Strontium peroxide	-------	-------	-------	-------	85.0	-------	90.0
	100	100	100	100	100	100	100

b. Compositions A and B have explosion temperature test values of only about 375° C., while that of the dim igniter composition B is 450° C. The explosion temperature for dim igniter A and the delay igniter is 300° C. These values are distinctly lower than those for tracer compositions. Igniter compositions are even less sensitive to shock than tracer compositions, and are very stable with respect to resistance to heat. However, those containing a peroxide are susceptible to deterioration through the absorption of moisture and their air-dust mixtures are readily exploded by an electric spark.

c. Because of these characteristics and the fact that relatively small quantities are used, igniter compositions are manufactured on a small scale by wet-mixing methods. Extreme precautions are taken to prevent the formation of air-dust mixtures or the spark discharge of static electricity. The binding agent is dissolved in an excess of a volatile solvent, such as carbon tetrachloride, and the fuel and oxidizing agent are wet thoroughly with the solution and mixed. The volatile solvent is then allowed to evaporate at room temperature, with intermittent stirring of the solid, under conditions of forced ventilation. If no soluble binding agent is used, the ingredients are mixed mechanically in the presence of an inert moistening agent, which later is removed by evaporation. Proper granulation is obtained by sieving the material while still damp and dustless.

d. Igniter compositions produce very little gas when burned, and they are less exothermic than tracer compositions (600 and 1,100 calories per gram, respectively). They owe their high ignition value to the formation of nonvolatile oxides, which become highly incandescent, and are the source of much radiant energy. These solid products of combustion are removed from the tracer cavity by the rush of gases produced by the tracer composition after ignition of the tracer has taken place.

13-15. Delay Compositions

a. General.

(1) A delay element is a self-contained pyrotechnic device consisting of an initiator, delay column, and an output terminal charge or relay, all assembled into a specially designed inert housing. In some designs, one or more components may be omitted. When used as one of the components of an explosive train, the device provides a predetermined time delay between initiation and the ultimate detonation or deflagration of the main charge.

(2) The delay compositions themselves are homogeneous mixtures of oxidants and fuels which, upon ignition, burn for a definite time within certain allowable limits. Their primary functions in a delay element is to ignite and set off the rest of the train at a predetermined time.

(3) There are four basic designs in delay elements; these have certan desirable characteristics relative to ultimate application, and certain advantages and disadvantages. Mechanically, the element may be obturated (closed) or vented either externally or internally; chemically, the element may contain a nongaseous delay composition (in which case little or no gas is formed during the chemical reaction) or a gaseous delay composition (in which case large amounts of gas are found during the reaction).

(4) Obturated columns are generally not affected by changes in environmental pressure. Vented columns present a sealing problem compounded by difficulties in hygroscopicity and shelf life. In addition, these types may need a large venting volume and, unless carefully designed and loaded, often fail to propagate and ignite the relay detonator.

(5) Wherever possible, gasless powders are used in designing chemical components of the delay element. Almost insensitive to shock and vibration when pressed into the housing at 30,000 to 35,000 psi, these powders cannot normally be initiated directly by an initiating source; however, they may require a small quantity of igniter composition for activation. Blended dry, they are frequently brittle when pressed; thus, under high setback conditions or vibration, they are subject to cracking or disengagement from the relay. Introduction of a binder makes the delay column perform better; however, binders, which are wholly or partially of organic materials, render the composition gaseous.

(6) In view of the variations cited above, it becomes apparent that the intended end-item application of the delay element must be the guide in choosing the most suitable type. If timing considerations are critical, the choice between vented and obturated elements is obvious; if operational physical

abuse is certain, inclusion of a binder seems the correct approach. Use under extremes of climatic conditions or outside the earth's atmosphere would call for serious consideration of obturated designs.

b. Compositions.

(1) *Gas-producing delay compositions.* Burning rate is a direct function of pressure. This is so because burning of gas-producing materials depends on heat transfer between gaseous reaction products and the unburned solid. Thus, burning rates of such delays are greatly influenced by all factors which affect the gas pressure at the burning surface (i.e., all of the surface exposed to the gas including that of pores and cracks which the gas may penetrate). The largest class of gas-producing delays comprises black powder elements. Reproducible behavior of any delay requires that it burn as a continuous, homogeneous substance. Porosity can result in a discontinuous relationship between interface pressure and burning rate. Black powder delays are therefore loaded at 60,000 psi or more. When a long column is required, it is pressed in increments, each pellet being no longer than its diameter. Moisture has a considerable effect on the burning rate of black powder; therefore, black powder delay elements may be kept dry. Effects of temperature extremes on performance of black powder delay elements vary appreciably from one delay to another. The spread of data almost invariably increases at extreme temperatures.

(2) *Nongaseous delay compositions.* To overcome the disadvantages of black powder as a delay composition, nongaseous delay mixtures have been developed, making use of inorganic exothermal reactions similar to those used for thermite mixtures. Examples of nongaseous delay compositions and their burning rates are shown in table 13-17.

*Table 13-17. Burning Rates of Typical Delay Compositions**
(Pressed at 30,000—40,000 psi)

				Burning Rate Seconds/Inch	
Iron Oxide	*Zirconium*	*Silicon Dioxide*		*Obturated*	*Vented*
25%	65%	10%		0.02	-------
Barium Chromate	*Zirconium*				
72%	21%			0.24	-------
84%	16%			0.32	-------
Barium Chromate	*Boron*				
81%	19%			0.35	0.50
90%	10%			0.45	0.70
95%	5%			1.10	1.70
96%	4%			2.40	3.60
Barium Chromate	*Tungsten*	*Potassium Perchlorate*			
20%	70%	10%		-------	5.0
42%	48%	10%		-------	14.0
56%	34%	10%		-------	30.0
Barium Chromate	*70/30 Zirconium-Nickel Alloy*	*30/70 Zirconium-Nickel Alloy*	*Potassium Perchlorate*		
60%	26%	----	14%	2.0	-------
60%	16%	10%	14%	5.0	-------
60%	3%	23%	14%	12.0	-------

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*The range of compositions given for some of the combinations allows for wide latitude in adjustment of burning rates.

c. Manufacture. Dry blending of ingredients is used for mixtures known to be insensitive to impact, friction, and electrostatic discharges. Wet mixing is used for sensitive mixtures or those to which a liquid binder is added. Loading of delay mixtures into the delay elements is done by pouring a weighed increment into the element and consolidating it under pressure with a hydraulic press. When a terminal charge (e.g., a lead azide relay or flash charge) is called for, it is pressed in prior to the delay composition. If ignition of the composition requires some special igniter powder, this is pressed on top of the delay composition, forming the last increment.

d. Performance Tests. The most important characteristic of a delay element is its burning time or burning rate.

(1) The most accurate method for measuring burning time in general in-

volves use of an electronic chronometer. Timing starts when the initiating source (e.g., a squib or primer) functions; it stops when the terminal end of the charge flashes. A firing switch simultaneously actuates the initiating source and the chronometer. Photocells or ionization gages, actuated by the flash of the terminal charge, stop the chronometer. Burning rate is expressed in seconds per inch. Correction is made to allow for burning time contributions of the initiating source, and igniter and terminal charge compositions.

(2) The simplest technique for measuring burning time is by stopwatch. This method, although satisfactory for relatively long-burning delays, is unsatisfactory for burning times of 2 seconds or less.

e. Ignition Powders for Delays.

(1) A column of nongaseous delay composition is usually preceded by a charge of igniter mix. Igniters are necessary when delay compositions are too insensitive to be initiated directly by the agent used in the particular application. The following table gives compositions of typical igniter mixes used in nongaseous delay elements.

Table 13–18. Ignition Powders for Nongaseous Delay Elements

Fuel	*Oxidant*	*Inert*
Boron (30%)	Lead peroxide (70%)	
Boron (10%)	Barium chromate (90%)	
Zirconium (41%)	Ferric oxide (49%)	Diatomaceous earth (10%)
Zirconium (33%) Titanium (17%)	Ferric oxide (50%)	

(2) Compositions in table 13–18 differ generally from those in table 13–17 in that the nongaseous mixtures burn faster and are readily ignitible.

f. Properties of Delay and Igniter Powders. In addition to burning rates, properties of delay powders of interest include variability of burning rate, temperature coefficient of burning rate, pressure coefficient of burning rate, effects of storage (both wet and dry), effects of column diameter and obturation and mechanical characteristics. These are affected by such variables as ingredient particle size, particle size distribution, homogeneity and uniformity of the mixture, and impurities not readily detectable. To control these variables, relatively elaborate procedures have been established for the procurement, characterization and treatment of raw materials, and the mixing and subsequent treatment of the igniter and delay powders.

13–16. Simulator Compositions

a. General.

(1) Simulator compositions are used in a variety of pyrotechnic training devices designed to simulate the effect produced by service items of ammunition. Casings of cardboard, plastic or similar material render these devices harmless.

(2) Devices designed to simulate exploding HE projectiles, rifle grenades, etc. must produce a loud report accompanied by smoke similar in color to that of an explosive burst. To obtain a loud noise, simulating devices are charged with black powder or a mixture of aluminum powder, potassium perchlorate, and barium nitrate. The latter, which is similar to photoflash compositions in its properties, burns almost instantaneously, produces a loud report and a flash, and liberates a quantity of white smoke. Compositions liberating gray or black smoke must contain a certain amount of organic compounds rich in carbon (naphthalene, anthracene, etc.).

(3) Other simulating devices liberate a colored smoke when fired. These are used during maneuvers for marking hits of chemical or incendiary projectiles or bombs, bursts of mine fields, etc. Colored smoke compositions are

generally employed for charging these devices.

(4) Pyrotechnic whistle compositions in simulator devices are used to represent boobytraps, or warn against simulated shell bursts in training weapons. Picric acid and such other organic acids as gallic, salicylic, and benzoic having the same characteristic odor are the major ingredients in pyrotechnic whistles. Combinations of these acids with potassium chlorate, perchlorate or nitrate, compressed in short tubes, when ignited, emit a shrill whistling sound of varying pitch. Size, especially length of the tube in which the composition is pressed, determines pitch.

b. Uses. Pyrotechnic simulator compositions are used to simulate a variety of ammunition items, typical of which are the following:

(1) *Air burst simulators.* These simulate an artillery projectile burst by producing a puff of white smoke.

(2) *Boobytrap simulators.* These are used during maneuvers and in troop training to fill a need for a safe boobytrap. Functioning with a loud report and flash, this device teaches troops a proper respect for bona fide boobytraps. One type of boobytrap simulator contains a slowburning composition which produces a whistle that lasts for 3 to 4 seconds.

(3) *Ground burst simulator.* This provides battle noises and effects during troop maneuvers. Following a delay of 6 to 10 seconds after ignition, the simulator produces a highpitched whistle which lasts 2 to 4 seconds. After a fraction of a second, the simulator explodes, producing a flash and loud report.

(4) *Gun flash simulator.* This provides a flash closely resembling that of the 90-mm gun M2 series and the 155-mm howitzer M1 series.

(5) *Hand grenade simulator.* This, as does the gunflash simulator, provides battle noises and effects during troop maneuvers. Thrown like a hand grenade, the device burns for 6 to 10 seconds after ignition, producing a flash and a loud report.

CHAPTER 14

INCENDIARY COMPOSITIONS FOR PROJECTILES

14–1. General

Incendiary projectiles, as their name implies, are used to start fires. They are used primarily in air-to-air warfare but not to the exclusion of air-to-ground, ground-to-air, and ground-to-ground types of warfare. Aircraft fuel lines and fuel tanks are particularly vulnerable to this class of projectiles. However, incendiary bullets can and do initiate destructive fires in a large variety of other targets. While aircraft, ammunition and fuel dumps are among the principal targets for incendiary attack, incendiaries have proved to be effective against personnel armored vehicles.

Table 14–1. Examples of Incendiary Compositions

	Incendiary				*HEI*
	%	%	%	%	%
50/50 magnesium-aluminum alloy	48	48	49	22.5	---
Barium nitrate	50.5	50.5	40	---	---
Potassium perchlorate	---	---	10	22.5	---
Zirconium	---	---	--	50	---
TNT	---	---	--	5	---
Linseed Oil	1.5	---	--	---	---
Zinc Stearate	---	---	1	---	1
Asphaltum	---	1.5	--	---	---
RDX	---	---	--	---	64
Aluminum	---	---	--	---	35

14–2. Compositions

a. Incendiary compositions are used in small arms bullets and artillery projectiles. They must be sensitive to the force of impact of such projectiles and undergo burning rather than explosion. For maximum incendiary effect, they should produce a high percentage of solid incandescent products of combustion. They may be considered intermediate between thermite compositions, which produce no gases, and tracer compositions. Thermite compositions are not sufficiently sensitive to impact to be suitable for use in incendiary ammunition.

b. Such compositions have relatively high explosion temperatures between 400° to 600° C. The incendiary compositions are purposely not oxygen balanced, there being provided an excess of metal particles, most of which burn to their oxides in the air after expulsion from the projectile. Essentially, the only gas produced for the oxidizer-fuel types of compositions is nitrogen. The high explosive incendiary (HEI) compositions yield carbon dioxide and H_2O in addition to nitrogen from the decomposition of RDX. These gases provide the bursting force required to fragment the projectile body. The projectile body may be made of a combustible metal alloy to enhance the incendiary effect.

c. Since all incendiary reaction temperatures are above 3,000° C, the single most important attribute of incendiary efficiency is the burning time of the metal particles. This burning time is usually referred to as burst duration, or the length of time the cloud of metal particles burn upon expulsion from the projectile. The burst duration of course depends upon the composition formula with respect to rate of reaction, kind of metal, and metal particle size consistent with reliability of ignition and reaction of the ingredients. Burst durations of 30 to 600 milliseconds have been recorded depending upon composition formula, type of projectile and type of target.

14–3. Ignition

Small arms incendiary bullets are designed with a thin metal nose section which crushes upon impact expelling the burning incendiary material. Larger caliber, such as 20-mm projectiles, are fitted with a point sensitive mechanical fuze which is initiated upon impact with the target. Both kinds of projectiles are sufficiently sensitive to function against thin aluminum sheet approximately 0.020″ thick of the type used for the skin of aircraft. The rounds are bore safe and drop safe for handling.

14-4. Tests

a. Incendiary projectiles are tested by firing against specified replica targets, usually a simulated aircraft wing structure, at a specified distance from the gun. Percent ignition, time of burst duration, location and character of fire burst are recorded electronically and photographically.

b. Ignition of hydrocarbon aviation fuels is determined by firing against a replica target fitted with a fuel spray device located behind a selected aluminum plate to simulate a punctured self-sealing fuel tank. Efficiency of incendiary action is determined by progressively increasing the delay time of fuel spray after projectile burst upon the target plate.

CHAPTER 15

PACKING AND MARKING

15-1. General

a. The wide range in the sensitivity, stability, and hygroscopicity characteristics of explosives and propellents has required the development of appropriately varied types of packing. The sensitivity of initiating explosives to shock and friction, that of black powder to spark and flame, the decreased stability of some propellents in the presence of moisture, and the hygroscopicity of many propellents and black powder have neccessitated such extremes as packing in a wet condition and the use of airtight containers that must withstand a prescribed internal pressure. On the other hand, the nonhygroscopicity and relative insensitivity of some high explosives permit the use of cartons, which are collapsible and can be reused when these explosives are packed for interplant shipment or short term storage.

b. Bulk priming, pyrotechnic, smoke, tracer, and incendiary compositions are not subjected to shipment or storage, being manufactured and loaded at the same plant. Special packing containers, therefore, are not prescribed for these compositions.

c. The marking of containers for explosives and propellents is prescribed by drawings and U.S. Army and U.S. Navy general specifications and comply with regulations of the Interstate Commerce Commission. The name of the material, lot number, specification number, manufacturer's initials or symbol, contract number, date of manufacture, gross weight, cubical displacement, and the dangerous commodity designation required by the Interstate Commerce Commission regulations are the most general markings. Markings may include also grade and/or class, plant where manufactured, and a box number. Initiating explosives are marked to indicate their nature and a prohibition against storing or loading with other explosives.

15-2. Initiating Explosives

a. The sensitivity of explosives of this type to shock and friction necessitates that these be packed in a wet condition. Because of the low solubility of lead azide, mercury fulminate, diazodinitrophenol, and lead styphnate, water can be used as the wetting agent. However, if shipment or storage under low temperature conditions is anticipated, a mixture of equal weights of water and ethanol is permitted.

b. Approximately 25 pounds of the explosive, wet with not less than 20 percent of liquid, is placed in a duck or rubberized cloth bag and covered with a cap of the same material. The bag is tied securely so as to prevent leakage. Not more than six such bags are placed in a larger bag of the same material. The larger bag is tied securely and placed in the center of a watertight metal or wooden barrel, drum, or keg lined with a heavy, close-fitting, jute bag. The large bag containing the explosive is surrounded with well-packed sawdust that has been saturated with water or water-ethanol mixture. The bag forming a liner is closed by sewing before the barrel, drum, or keg is closed. Not more than 150 pounds (dry weight) of initiating explosive is permitted in a single container.

15-3. Noninitiating High Explosives

a. Nitroglycerin, as such, may not be shipped by freight or express and almost invariably is manufactured at the plant where it is to be used.

Nitrocellulose, because of its sensitivity to spark, is wet with at least 20 percent of water and packed in watertight drums.

c. Because of sensitivity to shock, RDX and PETN are wet with water or ethanol-water mixture, so that the paste or slurry contains not less than 40 percent of liquid. This paste or slurry is placed in duck, rubber, or rubberized cloth bags holding not more than 50 pounds (dry weight) of explosive. These bags of explosive are placed in a larger bag of the same material. The small bags are surrounded with water and the large bag is closed securely. This bag is then placed in a watertight barrel, keg, or drum. The dry weight of explosive in one container must not exceed 300 pounds. It is of interest that, during World War II, the Germans packed RDX in a dry condition.

d. Ammonium nitrate, because of its great hygroscopicity, is packed in moistureproof metal drums or paper bags. The metal drums are lined with paper and may be of the single-trip type. Single-trip drums and burlap-covered paper bags for packing ammonium nitrate have a maximum capacity of 100 pounds.

e. TNT, tetryl, explosive D, picric acid, haleite, and nitroguanidine are almost nonhygroscopic. For lengthy storage or oversea shipment, they are packed in wooden boxes lined with waterproof paper and holding 50 or 100 pounds of explosive (fig. 15–1). For interplant shipment or temporary storage, such explosives can be packed in fiber cartons which are lined with a waterproof bag and hold approximately 50 pounds of explosive (fig. 15–2). Such cartons are collapsible and can be reused, the paper bag being destroyed after emptying.

f. Binary explosives such as amatol, tetrytol, picratol, torpex, and tritonal generally are manufactured at the loading plants where they are to be used and are not subjected to packing. Pentolite, composition A–3, and composition B are sometimes shipped to loading plants or placed in storage. They are packed in a dry condition in 50-pound wooden boxes or fiber drums lined with moistureproof paper bags, since they are much less sensitive than the PETN and RDX from which they are made.

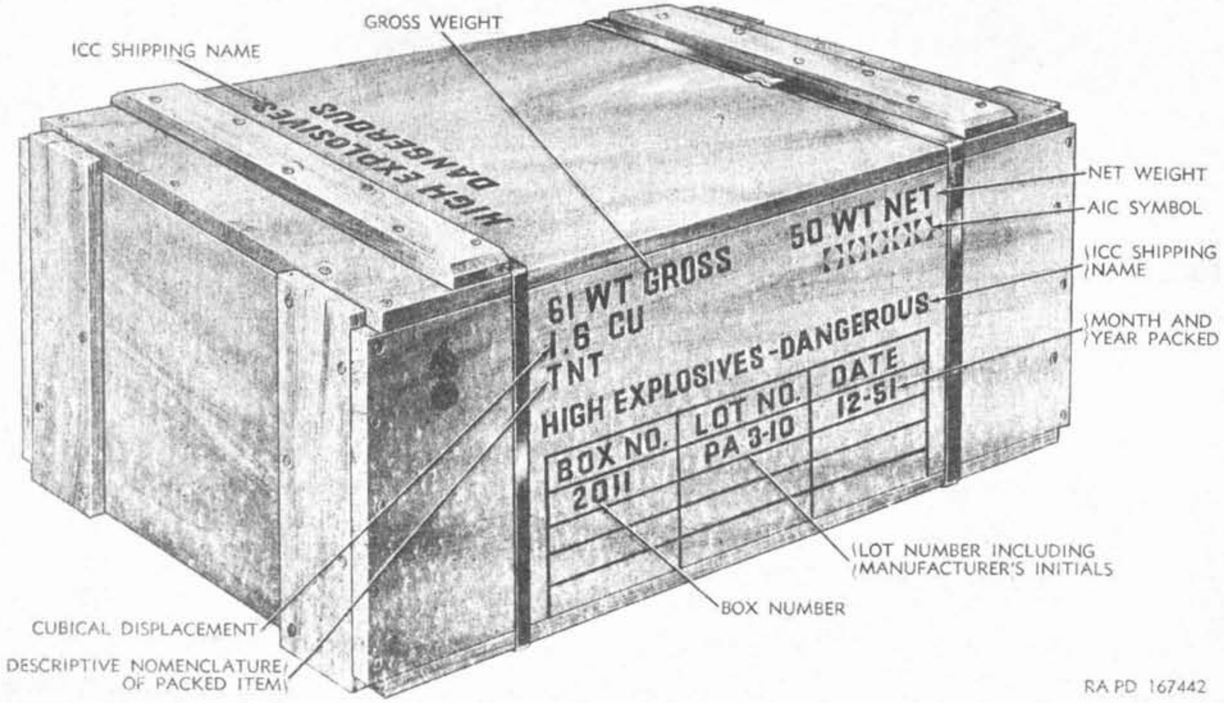


Figure 15–1. Wooden box for packing high explosives.

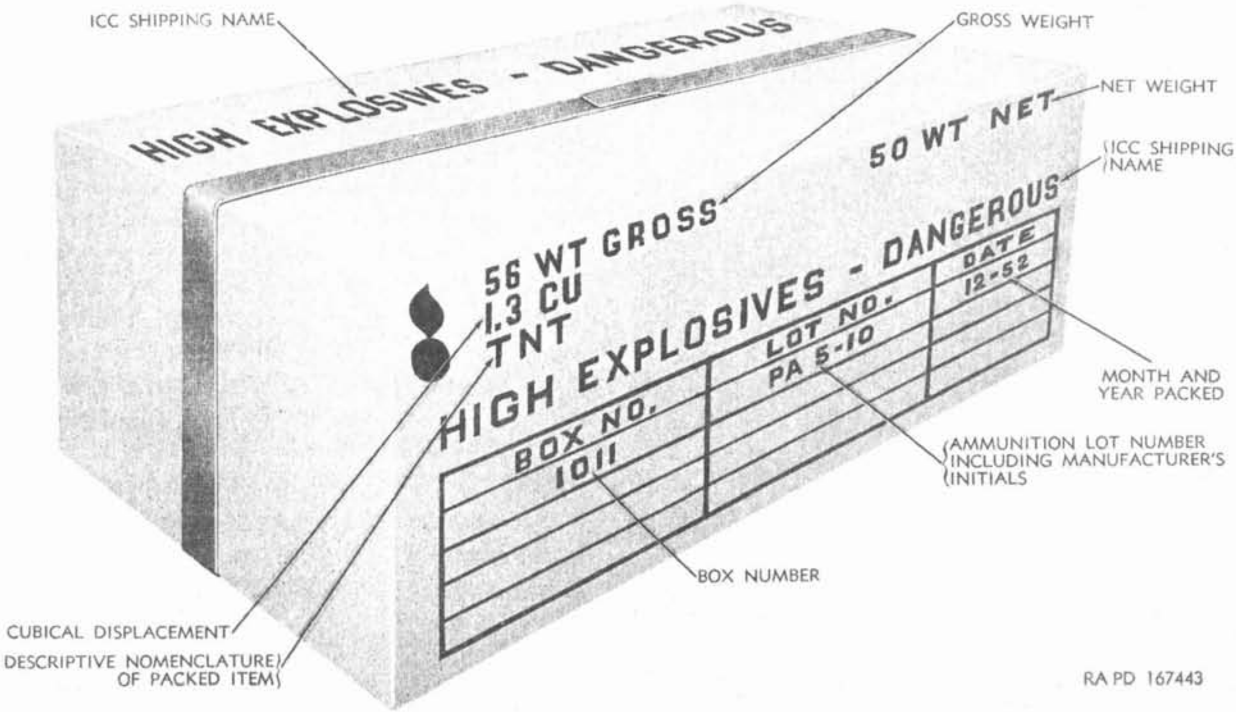


Figure 15-2. Fiber carton for packing high explosives.

Composition C-3, when shipped in bulk, is packed in wooden boxes holding 58 pounds and lined with oilproof and moistureproof paper bags.

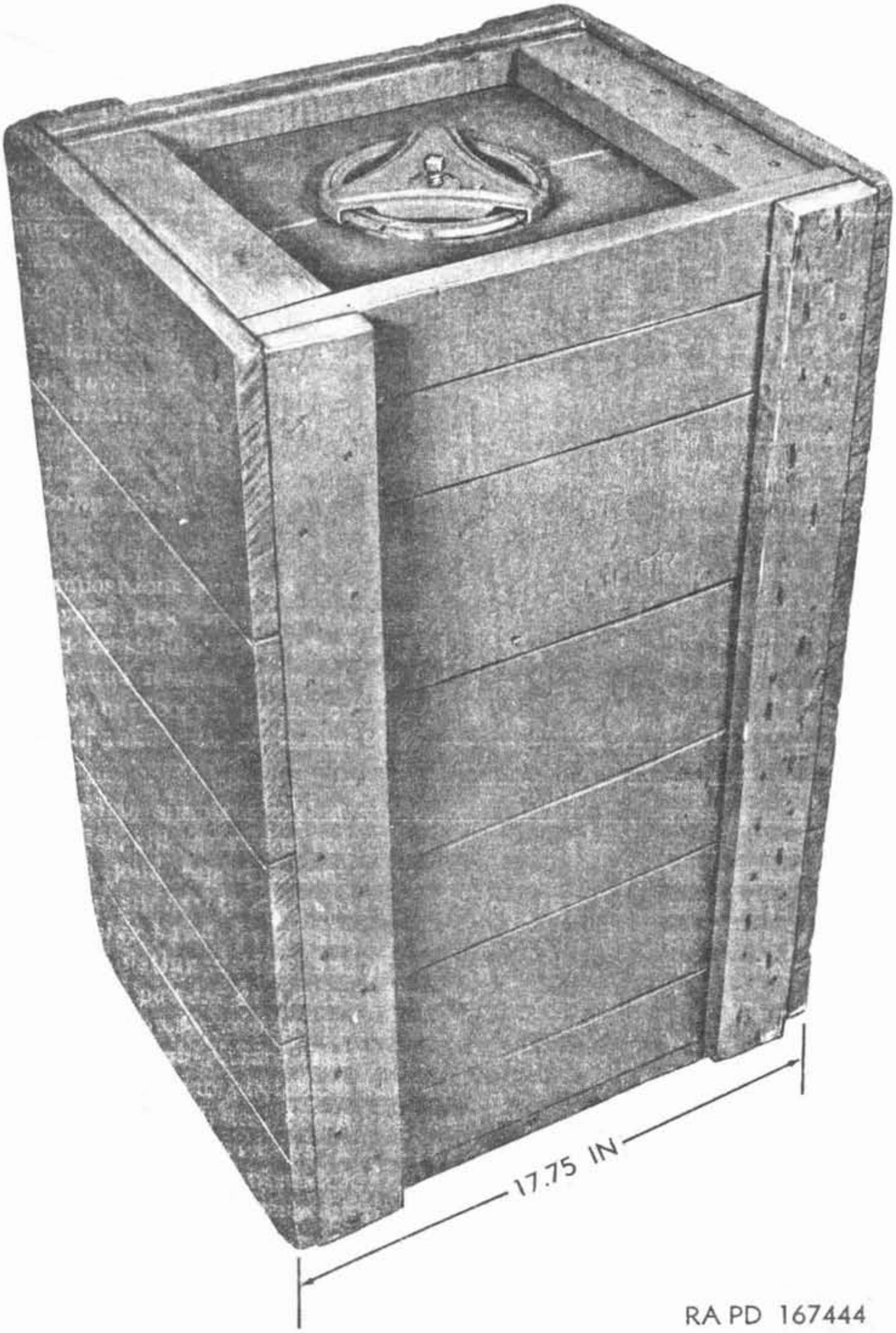
g. Military dynamites and demolition explosives such as the C-3 composition, like their commercial counterparts, are packaged in 1-pound sticks or blocks with paper; and these are packed in paperlined boxes holding 50 pounds of explosive. The paper used for packaging and packing generally is required to be oilproof as well as moistureproof.

15-4. Black Powder

Because of its extreme sensitivity to spark and its great hygroscopicity, black powder is packed in airtight steel drums, which hold 25 pounds of powder. These, sometimes, have small, slide-type gasketed closures.

15-5. Propellants

The hygroscopicity of nitrocellulose propellants in general and the adverse effect of moisture absorption on stability and ballistic value render necessary the packing of propellants in airtight containers. Copper-lined wooden boxes, tested for resistance to air pressure of 5 psi, were used formerly for all types of propellants and are still standard (fig. 15-3). These vary in size, holding as much as 150 pounds of powder. More recently, there have been standardized containers of stainless steel with a bonded outer layer of plywood and containers made of heavier, galvanized steel (fig. 15-4). Containers for propellants have relatively large, rubber-gasketed closures of the clamping type, with pressure applied by means of a screw. It has been found that propellants stored in such containers do not undergo change in moisture content even under the adverse conditions of tropical storage.



RA PD 167444

Figure 15-3. Wooden box for packing propellant.

RA PD 167445

Figure 15-4. Steel box for packing propellant.

CHAPTER 16

STORAGE

16–1. General

a. Because of the area of destructiveness of explosives and propellants in the event of accidental ignition or detonation, it has been necessary to establish rigid regulations for the storage of such materials.

b. On the basis of data for actual explosions, there was established the original American Table of Distances for the safe storage of explosives and ammunition. This has been revised on the basis of additional data, such as those given in R. Assheton's "History of Explosions" (1930) and C. S. Robinson's "Explosions, Their Anatomy and Destructiveness" (1944). This table shows the maximum distances at which damage to surroundings might be expected in the event of explosions of various magnitudes.

c. Explosions can cause death or injury to personnel (either directly or indirectly), damage to surrounding structures and vehicles, and ignition or initiation of other explosive material stored in adjacent buildings. Since these effects may be reduced or prevented by the barricading of magazines, control of quantities, or other means, the most recent regulations take into account inhabited building, public railway, public highway, magazine, and underground installation distances, as well as types of storage. For storage regulations, see TM 9–1300–206.

CHAPTER 17

SHIPMENT

17-1. General

a. There is an extensive requirement for interplant and oversea shipments of explosives and propellants, and shipments to and from reserve storage are particularly necessary during peace time. Miscellaneous shipments include those of salvaged explosive materials and explosives and propellents required for use in experimental and test work.

b. Railroads, ships, barges, and motor trucks are used chiefly for such shipments. With the exception of laboratory samples, all railroad shipments are made as freight, shipment of samples by express being permissible. Because of increased handling, shocks during transportation, and greater variety of hazards with changing environment, the shipment of explosives and propellants must be made with the utmost care. The problems of safe shipment are not unlike those of storage, and special regulations have been developed for the shipping of explosives and propellants as well as ammunition loaded with these.

c. While there were some accidents during World War II caused by the handling of ammunition, such as the loading and unloading operations incident to transportation, there was no explosion of explosives or propellants during actual transportation in the United States that was not caused by fire originating elsewhere than in the explosive material. In view of the enormous tonnages of such materials transported, chiefly by rail, this record is testimony to the effectiveness of the regulations for packing and shipping explosives and propellants.

17-2. Regulatons

a. Published regulations pertaining to explosives are listed in (1) through (8) below.

(1) Interstate Commerce Commission Regulations, Transportation of Explosives and Other Dangerous Articles by Freight.

(2) Interstate Commerce Commission, Motor Carrier Safety Regulations, Part Nos. 1 to 7 inclusive.

(3) Bureau of Explosives Pamphlets No. 6 and No. 6A.

(4) U.S. Coast Guard, Regulations Governing Transportation of Military Explosives on Board Vessels, and Regulations for the Security of Vessels in Port.

(5) U.S. Department of Commerce, Bureau of Marine Inspection and Navigation's Regulations Governing Transportation, etc., of Explosives.

(6) U.S. Civil Aeronautics Board, Civil Air Regulations, Part 49, Transportation of Explosives and Other Dangerous Articles.

(7) Freight Tariff No. 8.

(8) State and municipal laws and port and harbor regulations where applicable.

b. These regulations cover the inspection of freight cars, boats, and motor vehicles prior to loading with explosive materials, the loading and staying of shipments, the placarding of cars and trucks and the labeling of packages so as to indicate nature of shipments, the placement of freight cars in trains, and the inspection of shipments prior to unloading, as well as quantities of items permitted in individual cars, barges, and trucks.

17-3. Freight Shipments

a. Freight Tariff No. 8, publishing I. C. C. Regulations for the Transportation of Explosives and other Dangerous Articles by Freight, with supplements, establishes 13 classes of hazardous materials. Three of these classes comprise explosives, propellants, assemblies, and ammunition. The three classes include the following military explosives:

Explosives, Class A:

Lead azide
Mercury fulminate
Lead styphnate
Diazodinitrophenol
Black powder
Low explosives
TNT
Tetryl
Picric acid
Explosive "D"
RDX
Nitroguanidine
PETN
Haleite
Dynamite
Nitrostarch

Explosives, Class B:

Smokeless powder
Fireworks

Explosives, Class C:

Blasting caps (1,000 or less)
Primers
Cordeau detonant
Time blasting fuse (safety fuse)

b. It might appear that class A includes an unduly wide range of explosives from the viewpoint of sensitivity. However, when these explosives are packed as described in paragraphs 15–1 through 15–5, the factor of sensitivity may be considered fairly constant for all materials in this class. Class A explosives, therefore, are those which represent an explosion hazard in case of fire not in case of accident without fire. Transportation accident statistics substantiate this generalization. The transportation of niroglycerin, as such, by freight is not permitted, and it is not included in class A.

c. Explosives, class B, include explosive materials which are principally a fire hazard. Wet nitrocellulose, which might be expected to be included in class B, is included in the class of flammable solids. The transportation of dry nitrocellulose by freight is not permitted. Ammonium nitrate, although it has some explosive characteristics under extreme conditions, is not included in class B, but in the class of dangerous articles comprising oxidizing materials.

d. Class C explosives contain class A or class B explosives as components, but in restricted quantities. Such assemblies represent an explosion hazard in case of fire and might serve to initiate the explosion of any adjacent materials in classes A and B.

e. The establishment of these three classes simplifies the formulation of regulations for the proper separation of these and other dangerous articles during freight transportation. By such separation and the prescribing of appropriate packing, the extent of damage is minimized in case of accident or sabotage. The Bureau of Explosives of the Association of American Railroads, an official advisory body, does the test work and makes the recommendations upon which the Interstate Commerce Commission regulations for the freight transportation of explosives are based.

17–4. Motor Shipments

a. The Motor Carrier Safety Regulations of the Interstate Commerce Commission establish three classes of explosives that correspond closely, but not exactly, with those established for feight shipments. These are described in (1) through (3) below.

(1) Dangerous explosives, Class A, includes (*a*) through (*c*) below.

(*a*) High explosives that can be detonated by a blasting cap, including dry nitrocellulose, dry nitrostarch, and fireworks that can explode en masse.

(*b*) Black powder and low explosives.

(*c*) Blasting caps and electric blasting caps. Items of this class may be shipped in a common motor carrier, which consists of a truck to which no form of trailer is attached, or a semitrailer attached to a tractor, but to which no form of trailer is attached. Liquid nitroglycerin is not included in this class, but may be transported in special trucks that are not common carriers.

(2) Less dangerous explosives, Class B, includes smokeless powders for cannon and small arms, and fireworks not subject to explosion en masse.

(3) Relatively safe explosives, Class C, include assemblies such as squibs, primers, and Cordeau detonant.

b. Ammonium nitrate is not classified as an explosive for motor transportation, but as an oxidizing material. Separate regulations apply to the transportation of oxidizing materials.

17–5. Boat Shipments

Coast Guard Regulations also establish classifications of Class A, dangerous explosives; Class B, less dangerous explosives, and Class C, relatively safe explosives. This division into classes corresponds to that established by I. C. C. Regulations for motor transportation.

17–6. Airplane Shipments

Civil Air Regulations permit limited shipments of explosives and propellents by airplane. The packaging, labeling, and classification of such materials are in accordance with I. C. C. regulations for freight and express shipments. Passenger aircraft may transport no class A or class B explosive, except picric acid, explosive D, and TNT; and these must be shipped as medical or chemical materials, wet with at least 10 percent water, and in outside containers having maximum contents of 16 ounces. Class C explosives, except blasting caps, may be carried on passenger aircraft, but individual outside containers may inclose not more than 50 pounds of such explosives. Cargo aircraft may transport explosives permitted in passenger aircraft and also explosives other than blasting caps that are packed, labeled, and otherwise acceptable for express shipment in accordance with I. C. C. Regulations.

CHAPTER 18
DISPOSAL, DESTRUCTION AND DECONTAMINATION

18-1. General

In the course of manufacturing, loading, shipment, storage, and reconditioning operations, quantities of scrap, impure, deteriorated, and waste explosives and propellents are accumulated that cannot be used. Obsoletion of grades and the accumulation of these surplus materials sometimes present the problem of disposal or destruction. In the event that the operation of a manufacturing or loading plant is discontinued, the serious problem of decontamination of the plant equipment, structures, and ground presents itself. Because of their complete or almost complete insolubility in water and their toxic effects, explosives and propellents generally cannot be disposed of by solution and elimination as sewage. Submergence, burning, detonation, or decomposition by chemical agents must be resorted to.

18-2. Disposal

The term disposal covers the elimination of explosive material without alteration in form or identity. Burial in the earth is not an accepted practice, but dumping at sea is permitted under controlled conditions. Regulations require that explosives and propellants be removed from their containers and dumped not less than 10 miles from shore at depths of not less than 500 fathoms. The regulations permit only explosives that contain a water soluble constituent and that are desensitized by partial solution to be disposed of by dumping at sea.

18-3. Destruction

a. Burning generally is the preferred method for destroying explosives and propellents, but this is not always practicable and detonation or decomposition may be resorted to. Explosives of the initiating type cannot be burned, hence relatively large quantities are detonated. Smaller quantities are decomposed chemically.

b. In carrying out burning operations, the maximum quantity of a given explosive or propellant is that known to be safe for surrounding areas should it detonate en masse. Instances are on record of the detonation, while being burned, of TNT, tetryl, pentolite and explosive D. Explosives and propellants are burned in layers not more than 3 inches thick, except that unopened boxes of dynamite may be burned and layers of wet nitrobody sludge may be 12 inches deep. Loose, dry explosives may be burned in layers in direct contact with the ground, but wet explosives and boxed dynamite must be placed on a layer of combustible material. If it is found that wet explosive will burn only incompletely and with difficulty on the combustible bed, the bed may be sprinkled with fuel oil, which is not a volatile flammable liquid (must have a flash point higher than 100° F.). The layer of explosive or propellant is ignited by a train of combustible material, such as excelsior, arranged so that it and the bed of explosive material burn in the direction from which the wind is blowing (fig. 18-1). Ignition of the combustible train is accomplished by means of a black powder squib initiated by an electric current or time blasting fuse (safety fuse).

c. The destruction of explosives by detonation should be carried out in a pit not less than 4 feet deep, the explosive being covered with not less than 2 feet of earth. Where space permits, the use of a pit may be dispensed with. The maximum quantity to be detonated at one time is that which will not affect the nearest structures by blast effect or missiles resulting from the explosion. Detonation is effected by demolition blocks initiated by electric blasting caps.

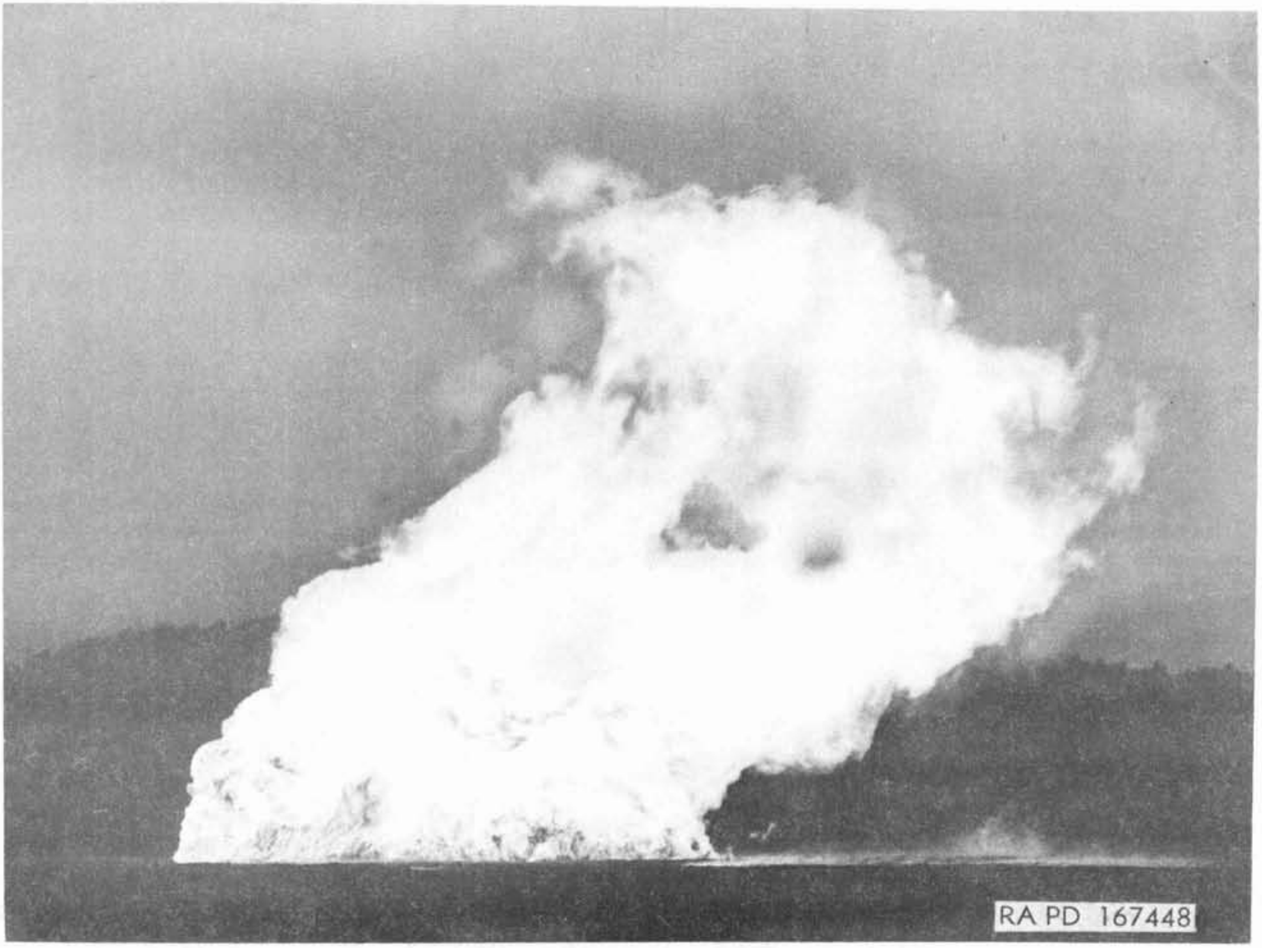


Figure 18-1. Burning of unserviceable propellant.

d. The destruction of explosives and propellants by burning or detonation is an operation to be carried out only with extreme care, because of the hazards involved in preparing the material for burning or detonation as well as the actual destruction. Careful attention should be given to the provisions of the AMC Safety Manual, AMC Regulation 385-224, in carrying out such operations.

e. Destruction of explosives by chemical decomposition is standard for only lead azide, mercury fulminate, and nitroglycerin, but methods for decomposing other explosives are available and may be used conveniently for disposing of small quantities of waste materials from laboratories or other sources.

f. Black powder can be disintegrated and desensitized by leaching with water, which dissolves the nitrate present. The washings must be disposed of separately from the residue. The residue of sulfur and charcoal is combustible but nonexplosive.

g. Lead azide can be decomposed as directed in (1) through (4) below.

(1) Mixing with at least five times its weight of a 10-percent solution of sodium hydroxide and allowing the mixture to stand for 16 hours with occasional stirring. The resulting supernatant solution of sodium azide is decanted and disposed of by drainage into the ground.

(2) Dissolving in a 10-percent solution of ammonium acetate and adding a 10-

percent solution of sodium or potassium bichromate until no more yellow lead chromate is precipitated.

(3) Wetting with 500 times its weight of water, slowly adding 12 times its weight of a 25-percent solution of sodium nitrite, agitating, and then slowly adding 14 times its weight of a 36-percent solution of nitric acid or glacial acetic acid. A red color produced on addition of ferric chloride solution indicates lead azide to be still present. Toxic fumes may be liberated during this process.

(4) Dissolving in 50 times its weight of a 15-percent solution of ceric ammonium nitrate. The azide is decomposed with the evolution of nitrogen.

h. Mercury fulminate can be decomposed by adding it, with stirring, to at least 10 times its weight of a 20-percent solution of sodium thiosulfate. Some cyanogen (a poisonous gas) may be evolved during this process.

i. Diazodinitrophenol can be decomposed by adding the water-wet material to 100 times its weight of a 10-percent solution of sodium hydroxide. The compound is decomposed with the evolution of nitrogen.

j. Lead styphnate can be decomposed by dissolving it in at least 40 times its weight of a 20-percent solution of sodium hydroxide or 100 times its weight of a 20-percent solution of ammonium acetate and adding a solution of sodium dichromate equal to half the weight of styphnate in 10 times its weight of water.

k. Tetracene can be decomposed by adding it to boiling water and continuing boiling for some time.

l. TNT can be decomposed by adding it slowly, while stirring, to 30 times its weight of a solution prepared by dissolving 1 part of hydrated sodium sulfide ($Na_2S.9H_2O$) in 6 parts of water.

m. Tetryl can be decomposed by dissolving it in 12 times its weight of a solution prepared by dissolving 1 part of hydrated sodium sulfite ($Na_2SO_3.7H_2O$) in 4 parts of water. The sulfite solution may be heated to 80° C., if desired, to facilitate decomposition of the tetryl.

n. Ammonium picrate (explosive D) can be decomposed by dissolving it in 30 times its weight of a solution prepared by dissolving 1 part of hydrated sodium sulfide in 6 parts of water.

o. Picric acid can be decomposed by dissolving it in 25 times its weight of a solution prepared by dissolving 1 part of sodium hydroxide and 21 parts of hydrated sodium sulfide in 200 parts of water. Some hydrogen sulfide and ammonia are evolved.

p. RDX can be decomposed by adding it slowly, so as not to interrupt boiling, to 25 times its weight of a boiling 5-percent solution of sodium hydroxide. Boiling should be continued for one-half hour after all the RDX has been added.

q. PETN can be destroyed by dissolving in eight times its weight of technical grade acetone and burning the solution in a shallow iron or enameled container. If decomposition is preferred, warm the acetone solution to 40° C. (105° F.), agitate, and add 7 parts by weight, for each part of PETN, of a solution prepared by dissolving 1 part of hydrated sodium sulfide in 2 parts of water and heating to 80° C. (175° F.). The aqueous solution should be added at such a rate that the acetone solution does not boil. After mixing is complete, agitation should be continued for one-half hour.

r. Haleite can be decomposed by addition to hot, dilute sulfuric acid. Nitrous oxide, acetaldehyde, and ethylene glycol are evolved. It can also be decomposed by adding 1 part to 5 times its weight of a 20-percent solution of sodium hydroxide.

s. Nitroguanidine can be decomposed by dissolving it in 15 times its weight of 45-percent sulfuric acid at 25° C. and warming the solution until gas is evolved. Heating is continued for some time thereafter.

t. Nitrocellulose can be decomposed by adding it with agitation in portions to five times its weight of a 10-percent solution of sodium hydroxide that has been heated to 70° C. (160° F.). Agitation is continued for at least 15 minutes after all the nitrocellulose has been added.

u. Nitroglycerin can be decomposed by adding it slowly to 10 times its weight of a 17.5-percent solution of hydrated sodium sulfide. The sulfide solution should be agitated during the addition and thereafter until solution is complete. Much heat is liberated by the reaction, but this does not represent a hazard unless agitation is interrupted.

v. DEGN can be decomposed in the same manner as nitroglycerin (*u* above).

w. Composition A-3 can be decomposed by the method described for the decomposition of RDX (*p* above).

x. Composition B can be decomposed by dissolving 1 part in 12 parts by weight of technical grade acetone and heating the solution to 45° C. (150° F.). While this is agitated vigorously, there is added slowly 12 parts by weight of a solution at 70° C. (160° F.) of 1 part of hydrated sodium sulfide in 4 parts of water. The addition should not be rapid enough to cause the temperature of the acetone solution to rise above 60° C. (140° F.). After addition is complete, agitation is continued for one-half hour.

y. Composition C-3 can be decomposed by adding 1 part slowly to a solution prepared from 1¼ parts of sodium hydroxide, 11 parts of water, and 4 parts of 95-percent denatured alcohol, and heated to 50° C. The mixture is heated slowly to 80° C. (176° F.) and maintained at this temperature for at least 15 minutes.

18-4. Decontamination

a. The cleansing of equipment, buildings, and grounds of explosive materials is a difficult, tedious, and sometimes hazardous operation. Because of the wide variety of materials, the existence of cracks, crevices, and cavities, and the possibility of explosions and the evolution of toxic or explosive gases, the operations and techniques must be made as simple as possible and various precautions taken to insure safety of personnel and completeness of decontamination. Serious accidents have occurred through the subsequent handling or heating of incompletely decontaminated equipment. For detailed safety measures, the reader is referred to the AMC Safety Manual and AMC Regulation 385-224.

b. Wherever practicable, decontamination is effected by the physical operations of washing, steaming, fire-flashing, and burning. Fire-flashing consists of exposing an item to sufficient flame to burn off any explosive present. Metal objects such as nitrators, centrifuges, tanks, piping, etc., are washed with water, steamed, and then flashed. A wood fire is used for the flashing operation, except for stainless steel objects, for which a low-temperature oil flame is employed. Equipment that is being decontaminated for standby purposes is not subjected to the fire-flashing operation. Wooden objects such as railing, paddles, etc., and buildings, such as dry-houses and packing materials, gaskets, etc., are destroyed by burning after preliminary cleaning. Earth that is so contaminated as to offer a fire or explosion hazard is wetted, scraped up, and burned at a burning ground.

c. Free acid present in equipment requires neutralization as well as washing, and a 5± percent solution of sodium carbonate (soda ash) is used for this purpose. Because of the uncertainty of complete removal of explosives in all cases by the physical methods described, chemical methods are used also to supplement these. Standard decontamination procedures include the chemical agents shown by table 18-1.

Table 18-1. Decontaminating Agents

Contaminant	*Agent*
Lead azide	Ceric ammonium nitrate
Mercury fulminate	Sodium thiosulfate
Nitroglycerin	Methanolic sodium sulfite
Nitrocellulose	Sodium hydroxide
Smokeless powder	Sodium hydroxide and acetone
TNT	Sodium carbonate and sellite
Tetryl	Sodium carbonate and sellite or acetone
Pentolite	Acetone

d. Loading plants, because of the use of a number of explosive materials, present particularly difficult problems of decontamination. The procedures appropriate to several different explosives manufacturing plants may be required for the decontamination of different parts of an individual loading plant.

e. With the growing complexity of modern military explosives and propellants, because of the introduction of new nonexplosive ingredients as well as mixtures of explosives, the problem of destruction and decontamination are increased correspondingly. New and special compositions, therefore, should be given careful technical consideration before destruction or decontamination operations are undertaken. While burning and washing operations suffice for most pyrotechnic, smoke and incendiary compositions, some of the newer compositions present explosion hazards when wet with water. Therefore, the rules in *a* through *d* above pertaining to explosives and propellant compositions apply equally to pyrotechnic, smoke, and incendiary compositions.

APPENDIX A

CHARACTERISTICS AND DATA

Table A-1. Sensitivity Test Values of Explosives[1]

	Impact test with 2 kilogram weight		Pendulum friction test,[2] percent explosions	Rifle bullet test, percent explosions	Explosion temperature test, °C.	Minimum detonating charge, gram of—		
	PA APP. inches	BM APP. cm				Lead azide	Mercury fulminate	Tetryl
TNT	14	100	0	2	475	0.26	0.24	
Ammonium nitrate	31	100+	0	0				
Nitroglycerin	1	15	100	100	222			
Nitrocellulose[3]	3	9			230	0.10		
PETN	6	17	5	100	225	0.03	0.17	
Tetryl	8	26	0	70	257	0.10	0.19	
Picric acid	13	82		50	322	0.24	0.26	
Explosive "D"	17	100+	0	10	318			0.06
Nitroguanidine	26	47	0	0	275			0.10
Haleite	10	43	0	0	190	0.13	0.21	
RDX	8	33	20	100	260	0.05	0.19	
50–50 Amatol	12	95	0	0	265			0.05
80–20 Tritonal	10	73	0	60	470	0.30		
50–50 Pentolite	13	29	0	80	220	0.13	0.19	
70–30 Tetrytol	11	28	0	30	320	0.23	0.23	
52–48 Picratol	14	100+	0	0	285			0.06
55–45 Ednatol		52	0	0	190	0.22	0.22	
Composition B	13	75	0	15	278	0.17	0.22	
Torpex	8	40		100	260		0.18	
Composition A–3	16	100+	0	0	250	0.25	0.22	
Composition C–3	14	100+	0	40	280			0.08
Composition C–4	10	100+	0	0	290			0.10
Lead azide	5	11			340			
Mercury fulminate	2	5			210			
Lead styphnate	3	8			282			
Diazodinitrophenol	2	5			180			
Tetracene	2	7			154			
Black powder	16	49	0		427			

[1] For units, meaning, and methods of determination, see paragraph 28.

[2] With steel shoe.

[3] Nitrogen content, 13.3 percent.

Table A-2. Effects of Explosives[1]

	Brisance measured by—			Rate of detonation		Ballistic pendulum test percent TNT	Trauzl lead block test percent TNT	Relative blast effect, percent TNT	
	Sand test, grams	Plate dent test percent TNT	Fragmentation of shell percent TNT	At density	Meters per second			Pressure	Impulse
TNT	47.5	100	100	1.56	6,900	100	100	100	100
Ammonium nitrate	--------	--------	--------	0.9	2,700	--------	56	--------	--------
Nitroglycerin	58.7	--------	--------	1.60	7,700	140	185	--------	--------
Nitrocellulose[2]	48.7	--------	--------	1.20	7,300	125	--------	--------	--------
PETN	61.2	127	--------	1.70	8,300	145	170	--------	--------
Tetryl	54.0	--------	121	1.71	7,850	128	129	--------	--------
Picric acid	47.9	107	--------	1.70	7,350	109	103	--------	--------
Explosive "D"	36.8	--------	99	1.63	7,150	98	--------	--------	--------
Nitroguanidine	36.8	--------	--------	1.55	7,650	104	78	--------	--------
Haleite	52.0	121	134	1.55	7,750	136	122	--------	--------
RDX	59.0	131	--------	1.70	8,350	150	170	--------	--------
50-50 Amatol	38.5	--------	82	1.55	6,435	122	124	97	87
80-20 Tritonal	46.0	93	91	1.72	6,700	124	--------	113	118
50-50 Pentolite	54.0	121	131	1.65	7,450	126	122	105	107
70-30 Tetrytol	52.7	117	117	1.60	7,300	120	--------	--------	--------
52-48 Picratol	44.6	100	102	1.62	6,940	100	--------	100	100
55-45 Ednatol	49.0	107	124	1.62	7,340	119	120	108	110
Composition B	53.0	131	139	1.66	7,800	133	130	110	110
Torpex	58.2	120	126	1.81	7,600	134	131	122	125
Composition A-3	51.0	126	150	1.59	8,100	132	--------	--------	--------
Composition C-3	53.0	114	133	1.60	7,625	126	115	105	109
Composition C-4	55.7	115	--------	1.59	8,040	130	--------	--------	--------
Lead azide	16.7	--------	--------	4.0	5,100	--------	40	--------	--------
Mercury fulminate	22.1	--------	--------	4.17	5,400	--------	51	--------	--------
Lead styphnate	10.5	--------	--------	2.9	5,200	--------	42	--------	--------
Diazodinitrophenol	45.6	--------	--------	1.58	6,900	--------	--------	--------	--------
Tetracene	2.0	--------	--------	--------	--------	--------	53	--------	--------
Black powder	6.3	--------	--------	1.6	400	--------	10	--------	--------

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[1] For units, meaning, and methods of determination, see paragraphs 25, 30, 33, and 34.

[2] Nitrogen content, 13.3 percent.

Table A-3. Thermochemical Characteristics of Explosives[1]

	Heat of combustion, calories per gram at constant pressure	Heat of formation, kilogram calories per mole	Products of explosion[2]	
			Heat, calories per gram	Gas, milliliters per gram
TNT	3,589.5	16.37	925	730
Ammonium nitrate	--------	87.93	346	980
Nitroglycerin	1,603	90.09	1,486	715
Nitrocellulose[3]	2,409	--------	855	918.5
Nitrocellulose[4]	2,313	--------	965	883.2
PETN	1,974	119.4	1,385	790
Tetryl	2,914	−5.02	1,120	760
Picric acid	2,671.5	56.33	1,000	675
Explosive "D"	2,745	95.82	800	--------
Nitroguanidine	1,995	23.58	721	1,077
Haleite	2,490	20.13	1,276	908
RDX	2,307.2	−24.17	1,300	908
50-50 Amatol	--------	--------	980	860
80-20 Tritonal	--------	--------	1,470	560
50-50 Pentolite	--------	--------	1,220	--------
Composition B	--------	--------	1,240	--------
Torpex	--------	--------	1,500	--------
Lead azide	--------	−100.6	367	308
Mercury fulminate	--------	--------	429	315
Lead styphnate	--------	--------	368	440
Diazodinitrophenol	--------	--------	820	--------
Tetracene	--------	--------	658	1,190
Black powder	--------	--------	665	280

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[1] For units, meaning, and methods of determination, see paragraph 28.
[2] Water produced in gaseous form.
[3] Nitrogen content, 12.60 percent.
[4] Nitrogen content, 13.35 percent.

Table A-4. Stability Test Values of Explosives[1]

	75° C. international test, percent loss in weight	100° C. heat test, percent loss in weight in—		Vacuum stability test						
		1st 48 hr	2d 48 hr	Sample, gm	100° C.		120° C.		150° C.	
					ML	HR	ML	HR	ML	HR
TNT	0.04	0.1	0.1	5	0.1	40	0.4	40	0.7	40
Ammonium nitrate	0.0	0.1	0.0	5	0.3	40	0.3	40	0.3	40
Nitroglycerin	--------	3.5	3.5	1	11+	16	------	------	------	------
Nitrocellulose	--------	0.3	0.0	5	0.9	24	11+	16	------	------
PETN	0.02	0.1	0.0	5	0.5	40	11+	40	------	------
Tetryl	0.01	0.1	0.0	5	0.3	40	1.0	40	11+	12
Picric acid	0.05	0.03	0.09	5	0.2	40	0.5	40	------	------
Explosive "D"	0.12	0.1	0.1	5	0.2	40	0.4	40	0.4	40
Nitroguanidine	0.05	0.2	0.1	5	0.4	40	0.5	40	------	------
Haleite	0.1	0.2	0.1	5	0.5	40	1.5	40	11+	24
RDX	0.03	0.03	0.0	5	0.7	40	0.9	40	2.5	40
50-50 Amatol	--------	--------	--------	5	0.3	40	1.0	40	------	------
80-20 Tritonal	--------	--------	--------	5	0.1	40	0.2	40	0.8	40
50-50 Pentolite	--------	0.0	0.2	5	2.5	40	11+	16	------	------
70-30 Tetrytol	--------	0.1	0.1	5	3.0	40	11+	40	------	------
52-48 Picratol	0.0	0.0	0.05	5	0.4	40	0.7	40	0.7	40
55-45 Ednatol	--------	0.2	0.1	5	0.7	40	11+	24	------	------
Composition B	--------	0.1	0.1	5	0.7	40	0.9	40	11+	16
Torpex	--------	0.0	0.1	5	0.2	40	1.5	40	11+	16

	75° C. international test, percent loss in weight	100° C. heat test, percent loss in weight in—		Vacuum stability test						
					100° C.		120° C.		150° C.	
		1st 48 hr	2d 48 hr	Sample, gm	ML	HR	ML	HR	ML	HR
Composition A-3	------	0.15	0.15	5	0.3	40	0.6	40	------	------
Composition C-3	------	3.2	1.6	5	1.5	40	11+	18	------	------
Composition C-4	------	0.1	0.0	5	0.3	40	------	------	------	------
Lead azide	0.17	0.5	0.1	1	0.4	40	------	------	------	------
Mercury fulminate	0.18	([2])	([2])	------	------	------	------	------	------	------
Lead styphnate	------	1.5	1.5	1	0.3	40	0.3	40	------	------
Diazodinitrophenol	0.24	0.5	1.1	1	6.0	40	------	------	------	------
Tetracene	0.5	23.2	3.4	------	------	------	------	------	------	------
Black powder	------	------	------	5	0.5	40	0.9	40	------	------

[1] For units, meaning, and methods of determinations, see paragraph 29.

[2] Explodes.

Table A-5. Density Values of Explosives*

	Crystal or liquid	Pressure in pounds per square inch							Cast
		3,000	5,000	10,000	15,000	20,000	30,000	40,000	
TNT	1.654	1.34	1.40	1.47	1.515	1.55	1.59	1.59	1.56
Ammonium nitrate	1.725	------	------	------	------	------	------	------	------
Nitroglycerin	1.596	------	------	------	------	------	------	------	------
PETN	1.765	1.37	1.575	1.638	------	1.71	1.725	1.74	------
Tetryl	1.73	1.40	1.47	1.57	1.63	1.67	1.71	1.71	1.62
Picric acid	1.763	1.40	1.49	1.57	1.61	1.64	1.67	1.69	1.71
Explosive "D"	1.719	1.33	1.41	1.47	1.51	1.53	1.56	1.57	------
Nitroguanidine	1.715	0.95	------	------	------	------	------	------	------
Haleite	1.67	------	1.30	1.40	1.46	1.51	1.55	1.58	------
RDX	1.816	1.46	1.52	1.60	1.65	1.68	1.70	1.71	------
50-50 Amatol	------	------	------	------	------	------	------	------	1.56
80-20 Tritonal	------	------	------	------	------	------	------	------	1.72
50-50 Pentolite	------	------	------	------	------	------	------	------	1.65
70-30 Tetrytol	------	------	------	------	------	------	------	------	1.60
52-48 Picratol	------	------	------	------	------	------	------	------	1.62
55-45 Ednatol	------	------	------	------	------	------	------	------	1.62
Composition B	------	------	------	------	------	------	------	------	1.66
Torpex	------	------	------	------	------	------	------	------	1.81
Composition A-3	------	1.46	------	------	------	------	------	------	------
Composition C-3	1.60	------	------	------	------	------	------	------	------
Lead azide	4.38	2.62	2.71	2.96	3.07	------	------	------	------
Mercury fulminate	4.42	3.00	3.20	3.60	3.82	3.99	4.10	------	------
Lead Styphnate	3.02	------	------	------	------	------	------	------	------
Diazodinitrophenol	1.63	1.14	------	------	------	------	------	------	------
Tetracene	------	1.05	------	------	------	------	------	------	------
Black powder	------	1.32	1.41	1.55	1.64	1.70	1.775	1.85	------

* In grams per milliliter.

Table A-6. Compositions of Standard Nitrocellulose Propellants

Composition	M1 (percent)	M2 (percent)	M5 (percent)	M6 (percent)	M7 (percent)	M8 (percent)	M9 (percent)	M10 (percent)	M13 (percent)	M15 (percent)	M17 (percent)	M26 (percent)	M26A1 (percent)	M30 (percent)	M30A1 (percent)	M31 (percent)
Nitrocellulose:																
12.60 percent N	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	28.0	28.0	20.0
13.15 percent N	84.2	.....	.....	86.1	54.6	.....	.....	98.0	57.3	20.0	20.0	67.25	68.70	.....	.....	.....
13.25 percent N	.....	75.55	81.5	.....	.....	52.15	57.75	.....	.....	.....	.....	.....	.....	.....	.....	.....
Nitroglycerin	.....	19.95	15.0	.....	35.5	43.0	40.0	.....	40.0	19.0	21.5	25.0	25.0	22.5	22.5	19.0
Barium nitrate	.....	1.50	1.5	.....	.....	.....	.....	.....	.....	.....	.....	0.75	.....	.....	.....	.....
Potassium nitrate	.....	1.00	1.0	.....	.....	1.25	1.5	.....	.....	.....	.....	0.7	.....	.....	.....	.....
Potassium perchlorate	.....	.....	.....	.....	7.8	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Nitroguanidine	.....	.....	.....	.....	.....	.....	.....	.....	.....	54.7	54.7	.....	.....	47.7	47.0	54.7
Dinitrotoluene	9.9	1.0	.....	9.9	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Dibutylphthalate	4.9	.....	.....	3.0	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Diethylphthalate	.....	.....	.....	.....	.....	3.0	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Potassium sulfate	.....	.....	.....	.....	.....	.....	.....	1.0	1.5	.....	.....	.....	.....	.....	1.00	.....
2-Nitrodiphenylamine	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	1.5
Diphenylamine	1.0	0.75	0.75	1.0	.....	.....	0.75	1.0	0.2	.....	.....	.....	.....	.....	.....	.....
Ethyl centralite	.....	.....	.....	.....	0.9	0.6	.....	.....	1.0	6.0	1.5	6.0	6.0	1.5	1.50	.....
Graphite	.....	0.25	0.25	.....	.....	.....	.....	.....	.....	.....	.....	0.3	0.3	.....	.....	.....
Carbon Black	.....	.....	.....	.....	1.2	.....	.....	.....	*0.05	.....	.....	.....	.....	.....	.....	.....
Cryolite	.....	.....	.....	.....	.....	.....	.....	.....	.....	0.3	0.3	.....	.....	0.3	.....	0.3
	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

*Added.

Table A-7. Compositions of Rocket Propellants

Composition	T2 (percent)	T3 (percent)	T5 (percent)	T6 (percent)	T8 (percent)	T9 (percent)
Nitrocellulose:						
12.20 percent N	....	56.0	....	55.5	....	....
13.15 percent N	58.0	....	57.5	....	58.0	....
Nitroglycerin	30.0	27.0	39.2	27.5	22.5	....
Potassium nitrate	....	....	....	....	....	49.8
Dinitrotoluene	2.5	9.3	....	10.5	2.5	....
Explosive "D"	....	....	....	....	....	40.7
Triacetin	....	....	....	....	8.5	....
Potassium sulfate	1.5	....	1.5	1.5	....	....
Methyl cellulose	....	*0.11	....	....	....	....
Ethyl cellulose	....	....	....	....	....	4.5
Ethyl centralite	8.0	7.2	1.75	4.0	8.0	....
Carbon black	0.02	....	0.05	0.5	....	....
Lead stearate	....	0.5	*0.1	0.5	0.5	....
Calcium stearate	....	....	....	....	....	0.5
Chlorinated wax	....	....	....	....	....	4.5
	100.0	100.0	100.0	100.0	100.0	100.0

*Added.

Table A-8. Characteristics[1] of Propellant Compositions

Composition	Heat of combustion[2] calories per gram	Products[3] of explosion		Burning rate[4] at 25°C.	Force, 1,000 foot-pounds per pound	Specific impulse[5] pound-seconds per pound
		Heat, calories per gram	Gas, milliliters per gram			
M1	2,975	744	858	15	314	213
M2	2,275	1,138	685	29	368	234
M3	2,830	820	804	12	310	212
M4	2,945	797	783	....	325	216
M5	2,389	1,032	729	....	362	232
M6	2,780	796	842	14	328	219
M7	....	1,262	587	59	370	237
M8	....	1,225	671	67	380	239
M9	....	....	....	43	384	240
M10	....	949	762	38	352	229
M12	....	950	760	38	356	229
M13	....	1,244	640	55	390	242
M14	....	879	....	....	337	219
M15	....	800	836	18	337	223
T2	....	935	821	31	353	228
T3	....	798	874	....	....	216
T5	....	1,259	610	....	....	234
T6	....	873	842	28	338	218
T8	....	752	864	20	306	210
T9	....	....	....	65	....	165
Pyrocellulose	....	861	792	19	339	231
Cordite MD	....	1,025	940	....	....	....

[1] For units, meaning, and methods of determination, see paragraphs 4-8, 9-3, and 9-4.

[2] At constant volume.

[3] Water produced in liquid form.

[4] Inch per second per pound per square inch x 10-5.

[5] At 2,000 psi.

Table A-9. Stability Test Values of Propellant Compositions*

Composition	Surveillance test, days at—		Humidor test, days at—			120° C. heat test			134.5° C. heat test		
	65.5° C.	80° C.	50° C. 100 percent RH	65° C. 95 percent RH	80° C. 95 percent RH	SP. min	RF. min	EXPL. min	SP. min	RF. min	EXPL. min
M1	1,500	250	3,740	775	150	------	------	------	70	135	300+
M2	335	------	------	------	------	85	165	300+	------	------	------
M3	1,500	------	------	485	160	------	------	------	70	135	300+
M4	900	------	3,435	------	185	------	------	------	------	------	------
M5	265	60	------	------	------	100	120	300+	------	------	------
M7	800	115	------	------	------	90	180	300+	------	------	------
M8	225	28	------	------	------	55	70	300+	------	------	------
M9	175	------	------	------	------	55	75	300+	20	20	85
M13	625	132	------	------	------	80	180	300+	------	------	------
M15	------	------	------	------	------	95	300+	300+	30	35	300+
T2	1,390	600	------	------	------	85	250	300+	------	------	------
T3	1,575+	300	------	------	------	95	300+	300+	------	------	------
T5	725	------	------	------	------	80	170	300+	------	------	------
T6	1,400	350	------	------	------	90	200	300+	------	------	------
T8	1,225	750	------	------	------	70	150	300+	------	------	------
Pyrocellulose	600	125	730	125	53	------	------	------	95	160	300+
E. C	700	------	1,800	450	140	150	300—	300+	40	60	300+
Cordite MD	585	------	------	------	------	------	------	------	30	40	300+

* For units, meaning, and methods of determination, see paragraph 78.

Table A-10. Conversion Factors

Unit	*Factor*	*Product*
Inches	25.4	Millimeters.
Millimeters	0.03937	Inches.
Microns	0.001	Millimeters.
Augstrom units	0.0000001	Milimeters.
Square inches	645.16	Square millimeters.
Square millimeters	0.00155	Square inches.
Cubic inches	16.387	Milliliters.
Milliliters	0.061025	Cubic inches.
Cubic feet	0.028317	Cubic meters.
Cubic feet	7.48	U. S. gallons.
Cubic meters	35.315	Cubic feet.
Liters	61.022	Cubic inches.
Liters	0.264178	U. S. gallons.
U. S. gallons	231.	Cubic inches.
U. S. gallons	3.78533	Liters.
U. S. gallons	8.337	Pounds water at 15° C.
Kilograms	2.2046	Pounds.
Pounds	453.59	Grams.
Ounce	28.35	Grams.
Calories	4.1855	International joules.
Calories	0.0413	Liter-atmospheres.
Kilogram-calories	3.9685	BTU's.
BTU's	0.25198	Killogram-calories.
BTU per cubic foot	890.	Kilogram calories per cubic meter.
Kilograms per square centimeter	14.223	Pounds per square inch.
Atmospheres	1033.3	Grams per square centimeter.
Atmospheres	14.696	Pounds per square inch.
Atmospheres	760.	Millimeters of mercury.
Atmospheres	14.7	Pounds per square inch.
Gram moles	22.414	Liters at 0° C. and 760 mm of mercury.
R (gas constant)	1.9684	Calories per ° C. per mole.
Foot candles	1	Lumens per square foot.
Lumens	0.001496	Watts.

APPENDIX B

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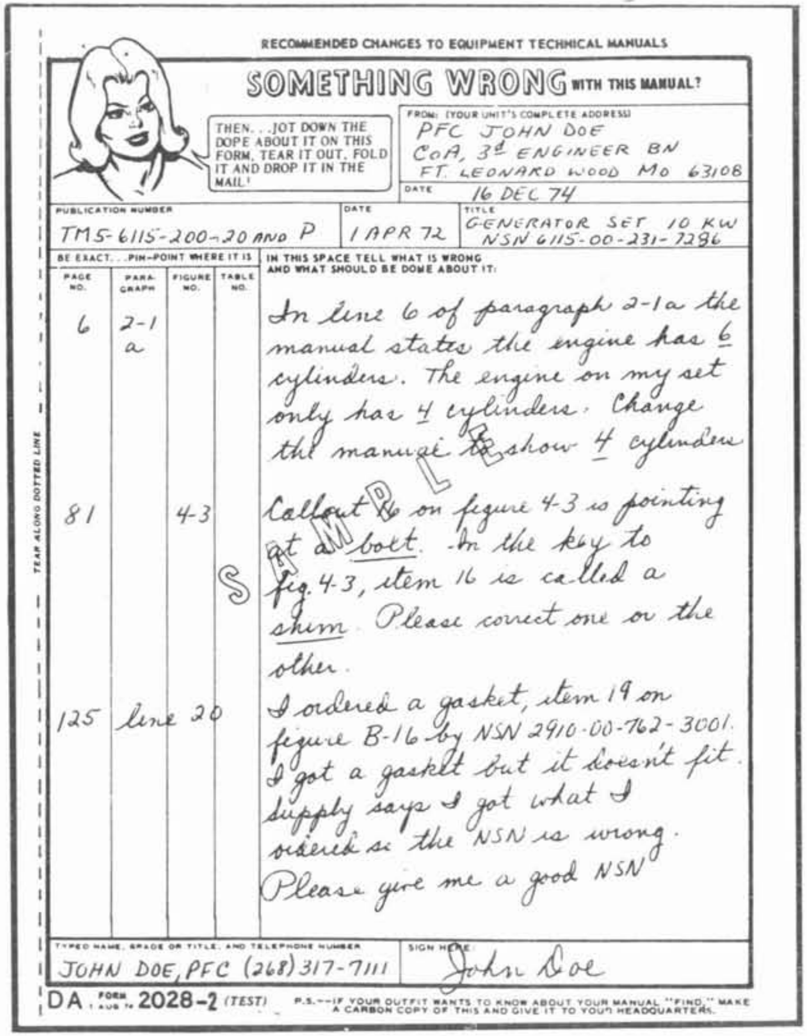
  - Picatinny (50)
- PG (2)
- Proc Dist (3)
- POE (2)
- MAAG (2)
- Mil Msn (2)
- JBUSMC (2)
- JUSMAGG (2)
- Log Con Ofc (2)
- MTMTS (2)
- EAMTMTS (2)
- WAMTMTS (2)
- Units org under fol TOE:
  - 3–500(AA-AC) (1)
  - 9–17 (2)
  - 9–22 (4)
  - 9–86 (4)
  - 9–500(BB, IA, KA-KC) (1)

NG: State AG (3); Units—Same as active Army except allowance is one (1) copy each.
USAR: None.
For explanation of abbreviations used, see AR 320–50.



RECOMMENDED CHANGES TO EQUIPMENT TECHNICAL MANUALS

SOMETHING WRONG WITH THIS MANUAL?

THEN. . .JOT DOWN THE DOPE ABOUT IT ON THIS FORM, TEAR IT OUT, FOLD IT AND DROP IT IN THE MAIL!

FROM: (YOUR UNIT'S COMPLETE ADDRESS)
PFC JOHN DOE
CoA, 3d ENGINEER BN
FT. LEONARD WOOD MO 63108

DATE 16 DEC 74

PUBLICATION NUMBER TM5-6115-200-20 AND P
DATE 1 APR 72
TITLE GENERATOR SET 10 KW NSN 6115-00-231-7296

BE EXACT. . .PIN-POINT WHERE IT IS PAGE NO.	PARAGRAPH	FIGURE NO.	TABLE NO.	IN THIS SPACE TELL WHAT IS WRONG AND WHAT SHOULD BE DONE ABOUT IT:
6	2-1 a			In line 6 of paragraph 2-1a the manual states the engine has 6 cylinders. The engine on my set only has 4 cylinders. Change the manual to show 4 cylinders
81		4-3		Callout 16 on figure 4-3 is pointing at a bolt. In the key to fig. 4-3, item 16 is called a shim. Please correct one or the other.
125	line 20			I ordered a gasket, item 19 on figure B-16 by NSN 2910-00-762-3001. I got a gasket but it doesn't fit. Supply says I got what I ordered so the NSN is wrong. Please give me a good NSN

SAMPLE

TYPED NAME, GRADE OR TITLE, AND TELEPHONE NUMBER JOHN DOE, PFC (268) 317-7111
SIGN HERE: John Doe

DA FORM 1 AUG 74 2028-2 (TEST) P.S.--IF YOUR OUTFIT WANTS TO KNOW ABOUT YOUR MANUAL "FIND," MAKE A CARBON COPY OF THIS AND GIVE IT TO YOUR HEADQUARTERS.

TEAR ALONG DOTTED LINE

DA FORM 2028-2 (TEST)

FILL IN YOUR UNIT'S ADDRESS

FOLD BACK

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CO A, 3d Engineer Bn
Ft Leonard Wood, MO 63108

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CUT ALONG DOTTED LINE

REVERSE OF DA FORM 2028-2 (TEST)

REVERSE OF DA FORM 2028-2 (TEST)

RECOMMENDED CHANGES TO EQUIPMENT TECHNICAL MANUALS

SOMETHING WRONG WITH THIS MANUAL?

THEN... JOT DOWN THE DOPE ABOUT IT ON THIS FORM, CUT IT OUT, FOLD IT AND DROP IT IN THE MAIL!

FROM: (YOUR UNIT'S COMPLETE ADDRESS)

DATE

PUBLICATION NUMBER	DATE	

BE EXACT. . .PIN-POINT WHERE IT IS				IN THIS SPACE TELL WHAT IS WRONG AND WHAT SHOULD BE DONE ABOUT IT:
PAGE NO.	PARA-GRAPH	FIGURE NO.	TABLE NO.	

TYPED NAME, GRADE OR TITLE, AND TELEPHONE NUMBER	SIGN HERE:

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DA FORM 1 AUG 74 2028-2 *(TEST)*

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