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C. W. Muehlberger

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THE INVESTIGATION OF BOMBS AND EXPLOSIONS

C. W. MUEHLBERGER

(Concluded From Previous Issue)

Having made the preliminary inspection of a suspected bomb and opened the container in accordance with the aforementioned safety precautions so as to permit access to the explosive and the ignition mechanism, these may then be separated, examined and chemically analyzed. The analysis of the explosive, the blasting cap and fuse will be discussed in order.

EXAMINATION OF THE EXPLOSIVE

Physical Examination:

Where this is a liquid, it should be handled with extreme caution. Care should be taken not to permit nitroglycerine or similar nitric esters to come into contact with the skin. Absorption through the intact skin is quite rapid and the resulting effects of lowered blood pressure, headache and nausea are very unpleasant and in rare cases may prove dangerous. A single drop of nitroglycerine rubbed on the skin will produce a violent throbbing headache in most persons who have not developed a tolerance to it through long and frequent exposure. In handling either liquid or solid explosives, rubber gloves should be worn to prevent contact with liquid nitric esters. The volume and weight of liquid should be recorded and the color and degree of fluidity of the material should be noted; also the character of its odor, if any. The acidity of the liquid should be tested, using blue litmus paper moistened with distilled water. Nitric esters such as nitroglycerine which contain free acid due to insufficient washing during the manufacturing process or to subsequent hydrolytic decomposition are very unreliable and capricious in their behavior. Such acidity, if observed, should be neutralized by treatment with dilute sodium bicarbonate (baking soda) solution to prevent possible accident. The *refractive index* (N_D) should be determined, using an Abbé type refractometer. From the readings on this instrument, the *dispersion coefficient*

may also be calculated with a fair degree of accuracy. The refractive index and, to a lesser degree, the dispersion coefficient, give excellent clues as to the chemical nature or identity of the fluid. If the volume of fluid permits, one may determine the *specific gravity* of the unknown liquid by the use of a hydrometer or a Westphal type of specific gravity balance. If the volume of liquid available is small (5 cc. or one teaspoonful or less), the specific gravity may be determined by means of a small specific gravity bottle (pycnometer) or a small specific gravity pipette. The latter may be used where the volume of liquid available amounts to only a few drops.

As a crude but rapid test of the sensitiveness of the liquid to shock, two or three small drops are permitted to fall on a small piece of filter paper; this is then placed upon an anvil and struck sharply with a hammer. If the liquid is sensitive and explosive, a slight explosion will occur. Needless to say, this test should not be made in the immediate vicinity of the bulk of the liquid under investigation.

Where the suspected explosive is a solid substance, a record should be made of its weight, color, and consistency (whether solid, dry and crumbly, or sticky). If in the form of sticks, the length, diameter and weight of each stick should be noted; the type of crimp in the paper wrapping at each end of the stick should be observed. The type of crimp affords information as to whether



FIGURE 9

End of Dynamite Sticks Showing Differences in Crimping.
Left: 6-fold machine crimp. *Right:* 4-fold hand crimp.

the sticks of "powder" were machine-loaded or hand-loaded. (Figure 9.) The stick or sticks may then be carefully unwrapped and the wrappings saved for further identification. The explosive contents or any solid suspected of being an explosive should be stored in clean, dry, tightly-stoppered, wide-mouth bottles. This is necessary to prevent possible absorption of moisture from the air. In most instances, dynamite sticks bear an identifying label stamped or printed on the waxed-paper wrapper. This label usually gives the brand name of the "powder," its explosive strength as compared to the percentage of nitroglycerine in a standard "straight" dynamite of equal explosive force, and the name of the manufacturer. Such labels give an excellent clue as to the nature of the contents of the stick, but for proof beyond reasonable doubt, as is necessary in a criminal case, the composition of the explosive must be established by a chemical analysis.

The explosive material should be examined with a hand lens or low-power microscope to determine its homogeneity. Frequently it may be separated mechanically into various constituents which may subsequently be identified.

Chemical Analysis of Explosive:

It is not feasible to present in this paper a *complete* method of chemical analysis. Nevertheless, the procedure outlined here is satisfactory in most cases.²²

As in most other chemical analyses, this consists of two steps: first, a qualitative analysis to determine *what* constituents are present; and, second, a quantitative analysis to determine *how much* of each constituent is present. To some extent these procedures may be combined. There probably is no scheme which will fit every explosive or possible mixture of explosives, but the following method has been found to give satisfactory results in most cases:

(1) *Ether extraction:* About 5 grams of explosive weighed into a tared 150 cc. Erlenmeyer flask is covered with 15 cc. of pure

²² For a more complete and exhaustive description of analytical technique, the reader is referred to other more detailed discussions:

(a) Snelling, W. O. and Storm, C. G., "The Analysis of Black Powder and Dynamite," U. S. Bureau of Mines, Bulletin No. 51 (1913); (b) Storm, C. G., "The Analysis of Permissible Explosives," U. S. Bureau of Mines, Bulletin No. 96 (1916); (c) Taylor, C. A. and Rinckenbach, W. H., "Explosives: Their Materials, Constitution and Analysis," U. S. Bureau of Mines, Bulletin No. 219 (1923); (d) Taylor, C. A. and Rinckenbach, W. H., "Analysis of Detonating and Priming Mixtures," U. S. Bureau of Mines, Technical Paper No. 282 (1922). See also reference No. 15 of bibliography at end of this paper.

dry sulphuric (ethyl) ether. The flask is shaken at intervals for a period of about five minutes and any lumps of explosive are broken up with a flat tipped glass stirring rod. The ether is then decanted off and filtered through a 7 cm. dry ashless filter paper fitted into a small, dry, glass funnel. The filtered ether extract is collected in a tared, dry 100 cc. beaker. Care is taken in decanting the ether solution, so as to retain as much as possible of the insoluble residue in the flask. This residue is extracted with two additional 15 cc. portions of ether and these extracts again decanted through the same filter paper into the beaker. A final extraction with 15 cc. of ether is made and the filtered extract collected in another tared, dry beaker. These extracts are then evaporated at a temperature not exceeding 50° C. until the ether has been eliminated. The beakers are then placed in a desiccator and dried at room temperature. As a drying agent in the desiccator, anhydrous barium perchlorate has been found to give excellent results. The beakers are weighed and the weight of extracted material noted from the increase in weight of the beakers. If the extraction has been complete, the second beaker containing the fourth ether extract should contain a negligible amount of residue.

The nature of the ether extract will give some idea of the probable explosives which it contains. If the extract is an oily pale yellow fluid liquid, it is probably a nitric ester of a polyhydric alcohol such as nitroglycerine or ethylene glycol dinitrate, or mixtures of various members of this group. If the extract is liquid but extremely viscid, it is probable that the nitric ester has been gelatinized with nitrocellulose (nitro-cotton). If the residue is crystalline or contains crystals, these may consist of sulphur or of polynitro-aromatic compounds such as TNT.

The liquid portion of the extract may be decanted away from the crystalline solids and its refractive index and specific gravity determined as previously outlined. This liquid should also be tested for (a) its solubility in water; (b) its solubility in glacial acetic acid; (c) its solubility in chloroform; (d) the formation of nitrites when the liquid is hydrolyzed with a small amount of normal alcoholic sodium hydroxide, and (e) its nitrogen content as determined by the nitrometer.²³

Liquid nitric ester explosives are insoluble in water and are decidedly heavier than water. They are fairly soluble in glacial

²³ Cope, W. C. and Taylor, G. B. "The Determination of Nitrogen in Substances Used in Explosives," U. S. Bureau of Mines, Technical Paper No. 160 (1917).

acetic acid and chloroform, although cellulose nitrate (nitro-cotton) is poorly soluble in chloroform. They yield a large quantity of nitrite when hydrolyzed with alcoholic sodium hydroxide, the reaction proceeding at room temperature with the evolution of heat. The nitrite in the reaction product may be identified by any of the usual chemical tests and the reaction may be utilized for the determination of small quantities of nitroglycerine.²⁴

The nature of the nitric ester may be determined by its refractive index, specific gravity and volatility when placed in a highly evacuated vacuum desiccator over concentrated sulphuric acid. In a vacuum, the dinitrates of the glycols are quite volatile, whereas nitroglycerine is stable and fails to lose weight progressively. The refractive index and specific gravity of various nitric ester explosives is given in the following table:

	Specific Gravity	Refractive Index	Actual % Nitrogen in Commercial Product
Nitroglycerine	{1.601 at 15° C. {1.591 at 25° C.	1.4721 at 20° C.	18.3%
Ethylene glycol dinitrate ²⁵	{1.496 at 15° C. {1.489 at 20° C.	1.4473 at 20° C.	18.2%
Propylene glycol dinitrate	1.384 at 15° C.		16.7%-16.8%

The crystalline material may be washed with glacial acetic acid to remove the remaining adherent nitric ester, recrystallized from warm carbon tetrachloride and its melting point determined. A small amount of the recrystallized material should be transferred to a small white porcelain evaporating dish and treated with alcoholic sodium hydroxide solution. The colors given by various nitro explosives vary from blue to red. Mononitro aromatic compounds give practically no color, while trinitro derivatives give reddish colors. Dinitro derivatives in general give purplish or bluish colors.²⁶ Sulphur dissolves in alcoholic sodium hydroxide

²⁴ Crandall, L. A., "The Rate of Elimination of Glyceryl Trinitrate From the Blood Stream After Intravenous Administration in Dogs," *Journal of Pharmacology and Expt. Therapeutics* 37:283-285 (1929).

²⁵ Rinckenbach, W. H., "The Properties of Glycol Dinitrate," *Industrial and Engineering Chemistry* 18:1195-1197 (1926).

²⁶ For a detailed account of these color reactions, see *supra* note 22(c) at page 122, and also Colver, E. de W. S., *High Explosives* (1918) 234-236.

solution to give a light yellow color. Uncrystallizable solid residue may contain some rosin or some paraffin wax from the wrapper dipping.

(2) *Water extraction:* The residue left in the Erlenmeyer flask following the ether extraction is allowed to dry spontaneously at a temperature of about 50° C. until the odor of ether is no longer noticeable. The flask is then cooled and weighed. The contents of the flask are then extracted at room temperature with successive portions of distilled water in a fashion similar to the ether extraction, filtering the decanted water extract through the same filter paper used for the ether extracts. The extraction is continued until a 15 cc. extract, examined by means of a dipping refractometer, gives the same refractive index as distilled water.

To make qualitative tests, exactly half of the water extract is employed. The remaining half is put aside for quantitative analysis. The qualitative tests consist of tests for chloride, sulphate, nitrate and oxalate, also for sodium, potassium, ammonium, barium and lead. The test for chlorate is made by acidifying a portion of the solution with dilute sulphuric acid and saturating it with sulphur dioxide gas. The flask is then tightly stoppered and allowed to stand over night. The excess of sulphur dioxide is then removed by bringing the solution to the boiling point and silver nitrate solution is added. If the silver chloride precipitate is heavier than that obtained by the direct treatment of the solution with silver nitrate, chlorates are present. If other acid radicals are found to be absent, it is safe to assume that the residue consists of nitrates.

If qualitative tests have shown that chlorates are absent, the portion of solution reserved for quantitative analysis is transferred to a 250 cc. beaker and evaporated to dryness on a steam bath. The beaker is then dried in a thermostatically controlled drying oven at 105° C. until the weight is constant. The increase in the weight of the beaker represents the water-soluble salts, usually sodium nitrate, or ammonium nitrate, possibly potassium nitrate from black powder or potassium chlorate or sugar from chlorate explosives. *Chlorate explosives should never be heated or subjected to violent physical force.* It is suicidal to violate this rule.

In practically all commercial dynamites the water-soluble salts consist of sodium nitrate (in "extra" dynamites) or ammonium nitrate (in "ammonia" dynamites). Where this is the case, the relative amount of sodium nitrate and ammonium nitrate may be determined by dissolving the weighed residue and

dividing the solution into two equal portions. One of these is rendered alkaline with sodium hydroxide and the liberated ammonia distilled into a measured excess of standard acid solution, as in the usual Kjeldahl procedure. The amount of ammonia present is found by determining how much of the standard acid has been neutralized. From this quantity of ammonia, the equivalent percentage of ammonium nitrate is calculated. The other half of the solution is placed in a weighed platinum evaporating dish and treated with several cubic centimeters of strong sulphuric acid. This mixture is then evaporated on a sand bath until heavy white fumes of sulphur trioxide are evolved. Heating is continued until all sulphuric acid has been volatilized and the residue is dry. The dish is finally heated in a muffle furnace at 800° to 1000° C. for several hours to volatilize ammonium salts, then cooled in a desiccator and weighed. The increase in weight represents sodium sulphate resulting from the sodium nitrate originally present. The weight of sodium nitrate can be calculated from the weight of sodium sulphate so obtained by multiplying by 1.1967. One should always make a test of the solubility of the resulting sulphated residue to make certain that it is water-soluble and also to ascertain by flame tests or by means of the spectroscope, that no potassium is present.

If a residue is found to contain only sodium nitrate and ammonium nitrate, the relative percentage of these may be approximated fairly accurately by heating for 8 to 12 hours in a muffle furnace at 350° C. At this temperature ammonium nitrate is volatilized or decomposed, whereas sodium nitrate is not affected. The loss of weight resulting from such heating represents the amount of ammonium nitrate present.

In the case of chlorate explosives, the water extract containing this substance must first be treated with a reducing agent to change the chlorate to the inexplusive chloride. The most suitable reducing agent for this purpose is sulphur dioxide gas. The quantity of chlorate present may be estimated by determining the quantity of chloride resulting from the reduction. Following this reduction, the metallic ion associated with the chlorate group may be discovered by evaporating the reduced solution with sulphuric acid and examining the sulphated residue. This metal is usually potassium. Potassium chlorate may be suspected in explosives where the water soluble material remains in solution so long as the solution is kept warm, but crystallizes out in hexagonal plates when the solution is cooled.

(3) *Acetone extraction:* The residual insoluble material left after the cold water extraction is dried at a moderate temperature (not in excess of 60° C.), and then extracted with warm acetone. The acetone extracts are filtered through the same filter paper used in previous extractions with ether and water. The filtered acetone extracts are transferred to a tared 150 cc. beaker and evaporated on a water bath at not to exceed 60° C. (acetone boils at 56° C.). In most instances, there will be practically no extract left upon evaporation of the acetone. A small stain of rosin or incompletely extracted nitro-cotton which escaped ether extraction, or a few yellow crystals of sulphur may occur. Where considerable white gummy residue is encountered in the acetone extract, it is probably due to nitro-starch. In this country, nitro-starch in non-military explosives suggests that the explosive was originally manufactured by the Trojan Powder Company at Allentown, Pennsylvania.

(4) *Carbon disulphide extraction:* Carbon disulphide is an exceedingly inflammable volatile liquid (boiling point = 46° C.) and must be used with the greatest of care to prevent serious fires or explosions. The dried residue left from the acetone extraction is treated with successive portions of freshly distilled carbon disulphide, each portion being allowed to remain in contact with the material for about ten minutes. The extracts are combined in a weighed beaker and allowed to evaporate in a warm place (about 30° to 40° C.) until the solvent has been completely volatilized. The residue, if any, will probably consist of sulphur (melting point = 119° C.). This can be further identified by burning a small amount on a platinum spatula placed in a Bunsen burner flame. If the material is sulphur, it will burn with a pale bluish flame, producing fumes having the odor of sulphur dioxide.

(5) *Insoluble residue:* The residual material from these extractions will consist of otherwise insoluble inorganic substances such as calcium carbonate, magnesium carbonate, crushed limestone, magnesium oxide, infusorial earth, ferro-silicon, powdered metallic aluminum, antimony trisulphide, red phosphorus, or potassium perchlorate, and insoluble organic materials such as wood pulp, wood flour, ground rice hulls, ground oat hulls, ivory nut meal, corn meal, or charcoal.

A weighed portion of the dry residual material is placed in a small tared porcelain evaporating dish and heated in a muffle furnace at 700° to 800° C. for 12 to 18 hours. The dish is then

cooled in a desiccator and weighed. This consists of the inorganic fraction of the residue.

A second weighed portion of the residual material is treated with hot 10% hydrochloric acid (one part concentrated acid to three parts distilled water) and carefully observed for any gas evolution. If gas is evolved, its odor should be noted. Any odor of hydrogen sulphide, which somewhat resembles rotten eggs, is an indication that a sulphide (probably antimony trisulphide or "stibnite") is present. Gas evolved with no striking odor is probably carbon dioxide from limestone or carbonates of calcium or magnesium, although it may be hydrogen gas if metallic aluminum is present. Following this treatment with hydrochloric acid, the mixture is filtered, using an ashless filter paper. The residue on the filter paper is washed several times with boiling distilled water. The filtered solution is nearly neutralized with pure sodium hydroxide solution and pure hydrogen sulphide gas is passed into it. If no precipitate (orange due to antimony sulphide) forms, the solution is boiled to eliminate hydrogen sulphide and treated with ammonium hydroxide until barely alkaline to litmus. If metallic aluminum is present, its hydroxide will separate at this point in the form of a white gelatinous precipitate. The precipitate, if any, is filtered off and ammonium carbonate added to the clear filtered solution. If a white precipitate forms, it is the carbonate of either calcium or magnesium. The washed residue on the filter paper is dried, placed in a small weighed porcelain dish and heated in a muffle furnace at 700° to 800° C. for 12 to 18 hours. The residue, if any, is weighed and examined. It will probably consist of sand, clay or infusorial earth or dark metallic-appearing particles of ferrosilicon.

Another portion of the original residue resulting from the extractions with ether, water, acetone and carbon disulphide is examined microscopically. From this examination an experienced observer can determine the nature of the organic absorbent in cases where a chemical analysis of such a residue would be of little avail. Also stibnite, red phosphorus, powdered aluminum or ferrosilicon may be recognized by their appearance under the microscope.

(6) *Moisture*: Many explosives such as "extra" dynamites or "ammonia dynamites" absorb water readily if the waxed paper wrappings are cracked or broken and the contents are permitted to come into contact with moist air. In such explosives the sensitivity and explosive power of the "powder" is decreased by increasing

moisture content. If the moisture content rises too high the powder becomes so insensitive as to be non-explosive. It is, therefore, of great importance to determine the moisture content of an explosive in order to determine its probable sensitivity and explosive effect. In many instances, moisture in an explosive may be estimated by observing the loss in weight of a weighed sample when placed in a desiccator over an active water absorbent such as concentrated sulphuric acid or anhydrous magnesium perchlorate. The latter substance is preferred as causing less hazard from spilling. If, by accident, a sample of a chlorate explosive should be spilled in a sulphuric acid desiccator, the resulting explosion and spattering of acid might be very serious.

With explosives containing easily volatile constituents such as the nitrated glycols, the loss in weight in the desiccator is due in part to the evaporation of these substances. In such preparations, the direct distillation and measurement of the water gives more accurate results. The procedure is similar in principle to the Brown--Duvel method for determining moisture in grain products.²⁷ The weighed sample of dynamite is placed in a distilling flask containing a water-insoluble solvent such as carbon tetrachloride or benzol. The mixture is distilled into a receiver which traps the water which separates out, but permits the refluxing solvent to flow back into the distilling flask. The method must be carefully standardized with powders of known moisture content so as to correct for such factors as the solubility of water in the solvent and also the original water content of the solvent.

Summary of Chemical Analysis:

In a reasonably complete and accurate chemical analysis, the sum of the percentages of various constituents should approximate 100%. Due to slight inaccuracies caused by incomplete removal of solvents, incomplete drying of extracts and minor losses due to retention of material by filter paper, or undetermined constituents, the sum of the components may total 98% to 102%. Even with the most expert technique, it is only by accident that the sum of the determined constituents of a complex explosive add up to within .1% of the theoretical 100% value. Slight inaccuracies in analysis, while they should be avoided so far as possible by careful analytical

²⁷ Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists (3d ed., 1930), Washington, D. C., at pages 277 and 278.

work, nevertheless do not detract materially from the result. Thus, so far as explosive effect is concerned, it makes very little difference whether a "powder" contains 14.9% nitroglycerine or 15.1% nitroglycerine.

Black powder can usually be recognized by its form and contains charcoal (5-15%), sulphur (5-15%), and either sodium or potassium nitrate (70-90%). Potassium nitrate (saltpeter) powders are the older type used in ammunition, but the cheaper sodium nitrate pellet powders, while they absorb moisture more rapidly, are widely used for quarrying.

Smokeless powder (gelatinized nitrocellulose) can also be recognized by its grain form and its poor solubility in all ordinary solvents except acetone.

Dynamite is a very vague term applied loosely to a wide range of explosive mixtures. Following the world war, stocks of smokeless powder intended for military or naval purposes were ground up, mixed with a small amount of nitroglycerine to sensitize the mixture and loaded into the usual 8 inch x 1¼ inch sticks as a "dynamite" intended for agricultural use. Most dynamites employ nitroglycerine or related nitric esters as an important explosive in their composition.

High strength nitroglycerine dynamites may appear quite moist unless they are of the "extra" or gelatin types. Gelatin dynamites have a more sticky, putty-like consistency. Some dynamites (see No. 3 of the following table) contain no nitroglycerine or liquid nitric ester whatever, and hence do not cause a headache when brought into contact with the skin.

Chlorate explosive mixtures are chiefly found in fireworks, either for "Fourth of July" celebrations or for signaling. Occasionally one finds chlorate mixtures in bombs intended for criminal use, but these are usually made by amateurs. Experienced bomb makers have too high respect for the treachery of chlorate mixtures to risk their lives in the manufacture of bombs or infernal machines containing them.

The purpose of the chemical analysis is to enable the expert to testify that the mixture analyzed constitutes an "explosive," or a "high explosive," as the case may be. As has been previously pointed out, where an adequate quantity of explosive is available, an actual explosion test should be conducted in the bomb-proof.

Typical analyses are given below:

	No. 1 40% Strength Gelatin Dynamite	No. 2 40% Strength Extra Dynamite	No. 3 40% Strength Nitro- Starch Powder
Moisture	.25	.32	.54
Liquid Nitric Ester (Nitroglycerine)	22.72*	13.80
Sodium Nitrate	52.83	40.25	25.67
Ammonium Nitrate	7.65	34.65	48.92
Sulphur	4.85	3.72
Calcium Carbonate (Chalk)	1.21	1.07
Trinitrotoluene (TNT)	5.95
Dinitrotoluene (DNT)	1.80
Ferrosilicon	1.42
Nitro-Starch	13.18
Absorbent or Filler	8.52	5.88	4.17
Total	99.83	99.69	99.85

* Including small percentage of nitro-cotton.

EXAMINATION OF BLASTING CAPS

There are two general types of blasting or detonating devices in general use for setting off explosives. These are "squibs" which are used for igniting low explosive powder such as pellet or blasting powder, and "caps" used for detonating high explosives. These differ in that caps contain a detonating charge, while squibs merely produce a substantial flash. Squibs are shorter than blasting caps and are usually made with aluminum shells. A blasting cap, if used for exploding black powder, would simply scatter the powder without exploding it, while a squib, used with TNT, would be entirely ineffective in producing an explosion. Squibs are practically always set off by electrical ignition, whereas blasting caps are set off either by safety fuse or by electric current. Some electrically ignited blasting caps have a length of fuse enclosed in the cap so that there is a delay period between the passage of the electrical current and the detonation of the priming charge. Such caps may be obtained with rather accurately gauged delay periods of from one to ten seconds. Since, in the experience of the writer, squibs are not used

in bombs intended for criminal use, their examination will not be discussed here.

Physical Examination of Blasting Cap:

The cap should be carefully measured and described. If any crimp markings are noticeable from the attachment to a fuse, these should be protected and preserved for possible comparison with any crimping tool, pliers or other tool found in the possession of a suspect. The outside diameter of the cap, its length, weight, contour and markings of the base, apparent composition of the capsule, length of open space above the explosive mixture, location of any circumferential ridges in the external surface of cap (indicating location of a reinforcing capsule)²⁸ should all be observed and recorded. If the cap is designed for electrical ignition, the length, gauge, metallic composition and type of insulation of the wires should be noted; also the nature of the sealing compound by which

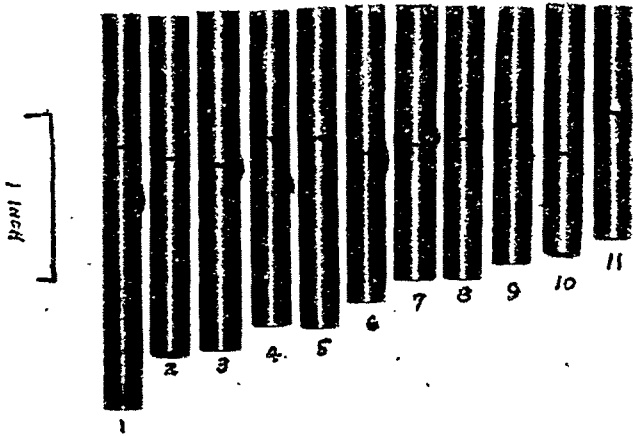


FIGURE 10

(1) Western, No. 8; (2) Atlas, No. 8; (3) Trojan, No. 7; (4) California, No. 8; (5) Du Pont, No. 8; (6) Western, No. 6; (7) Trojan, No. 6; (8) Du Pont, No. 6; (9) Hercules, No. 6; (10) Atlas, No. 6; (11) California, No. 6. (The black line on each cap shows the depth of loading.)

²⁸ In many blasting caps, the explosive force of the initiating charge (fulminate-chlorate) is enhanced by pressing this explosive mixture into a small metal reinforcing cup or capsule. This is then pressed into the cap so as to lodge against the detonating charge. See Figure 13.

these wires are sealed into the end of the capsule of the cap. From these characteristics it is usually possible to determine the source of the cap, if manufactured in this country. (See Figures 10 and 11.) There are only seven companies in the United States manufacturing blasting caps and one of these (General Explosives Division of American Cyanamide and Chemical Corporation) makes only electric caps. The characteristics of these caps are given in the following tables. Delay caps are more difficult to identify, probably due to the fact that these are still in the development stage and are being modified from time to time.

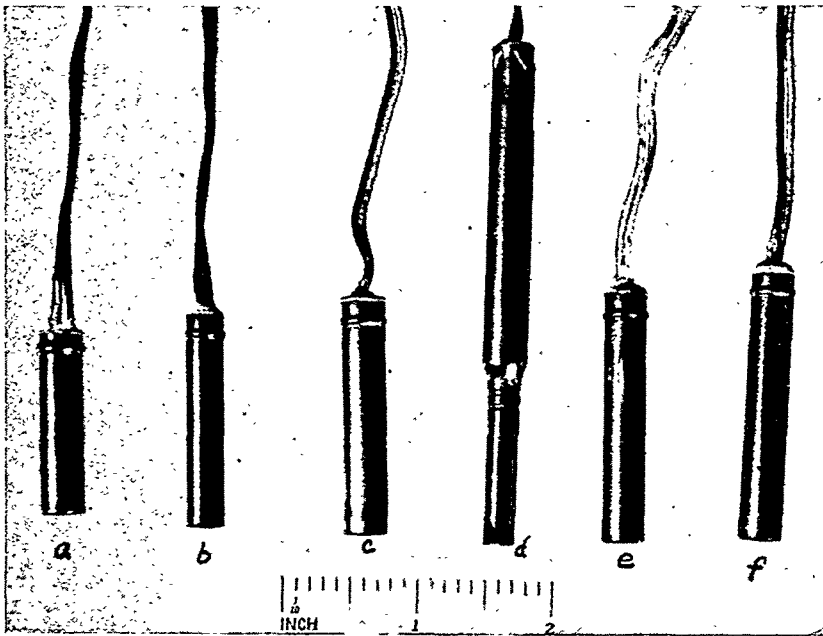


FIGURE 11

Types of Electric Blasting Caps: (a) Hercules, No. 6; Du Pont, No. 6; (c) California, No. 6; (d) Western ten sec. delay cap (base of dummy cap cut open); (e) Trojan, No. 6; (f) California, No. 8.

Analysis of Explosive Contents of Blasting Cap:

The first step in analyzing the explosive contents of a blasting cap is to get them out of the metallic capsule or shell. To do this safely without losing or exploding the contents requires some skill and patience. Where many caps are available for analysis, one

FUSE BLASTING CAPS

Manufacturer	Strength	Outside Diameter Length		Initiating Charge	Detonating Charge	Reinforced	Characteristic Marking on Base
		Inch	Inches				
Du Pont Co.	#6	.234	1½	Mercury Fulminate Potassium Chlorate	Tetryl	Yes	None
Du Pont Co.	#8	.237	1%	Fulminate-Chlorate	Tetryl	Yes	None
Hercules Powder Co.	#6	.234	1½	Dinitro Diazo Phenoxide	Tetryl	Yes	{ B S
Hercules Powder Co.	#8	.234	2	Dinitro Diazo Phenoxide	Tetryl	Yes	{ B S
Western Cartridge Co.	#6	.234	1%	Fulminate-Chlorate	Picric Acid TNT	Yes	{ WC CO.
Western Cartridge Co.	#8	.234	2½	Fulminate-Chlorate	Picric Acid TNT	Yes	{ WC CO.
California Cap Co.	#6	.233	1%	Fulminate-Chlorate	Tetryl	No	C
California Cap Co.	#8	.235	1%	Fulminate-Chlorate	Tetryl	No	C
Atlas Powder Co.	#6	.235	1½	Fulminate-Chlorate	Tetryl	No	None*
Atlas Powder Co.	#8	.235	2	Fulminate-Chlorate	Fulminate-Chlorate	No	None*
Trojan Powder Co.	#6	.249	1%	Fulminate-Chlorate	Nitro-Starch	Yes	None
Trojan Powder Co.	#7	.249	2	Fulminate-Chlorate	Nitro-Starch	Yes	None

* Atlas caps may be differentiated from Du Pont caps by their rounded edges of the base and the small pointed indentation in the center of the base. Du Pont caps have a more right-angled base and a larger indentation in the base which is in the form of a truncated cone.

ELECTRIC BLASTING CAPS

Manufacturer	Strength	Outside Diameter Length		Identifying Characteristics	
		Inch	Inches		
Du Pont Co.	#6	.274	1%	Red lacquered shell	
Du Pont Co.	#8	.273	1%	Red lacquered shell	
Hercules Powder Co.	#6	.314	1%-1½	{ B S } stamped in base	
Hercules Powder Co.	#8	.314	1% to 2%	{ B S } stamped in base	
Western Cartridge Co.	#6	.316	1%	{ WC CO. } stamped in base	
Western Cartridge Co.	#8	.316	1½	{ WC CO. } stamped in base	
California Cap Co.	#6	.318	1%	"C" stamped in base	
California Cap Co.	#8	.319	2	"C" stamped in base	
Atlas Powder Co.	#6	.275	1%	None	
Atlas Powder Co.	#8	.275	2¼	None	
Trojan Powder Co.	#6	.314	1%	None	
General Explosives Div. of Amer. Cyanamide and Chem. Corp.	} #6	.273	1½	None	
		#8	.273	2%	None

may resort to physical force in opening a cap. This, of course, must be done with due precaution for protecting the operator. Such techniques for opening caps are described by Taylor and Rinkenschach.²⁹ In criminal cases, there usually is only one cap available for analysis and therefore one cannot take a chance of losing it in an accident.

The writer has found that controlled acid corrosion of the capsule affords a safe way of opening the cap and getting out the contents. This is done by cutting two pieces of $\frac{1}{4}$ " diameter gum rubber tubing, each about 2" long. These are slipped over the blasting cap so that all of the cap is covered except for a narrow exposed ring about $\frac{1}{16}$ " wide. The rubber tubes may be shifted so that the ring of exposed metal may be located at any point in the length of the cap. Usually in a fuse blasting cap it is best to locate this exposed area about $\frac{1}{4}$ " to $\frac{3}{8}$ " below the upper edge of the explosive. The open ends of the rubber tubes are then closed with short lengths of $\frac{1}{4}$ " diameter glass rod. This combination is then dipped into dilute (10-15%) nitric acid solution. The acid slowly corrodes the metal (usually copper) of the exposed surface. By constant observation and control of the acid concentration, the corrosion may be carried to the point where the capsule has just been cut through. At this point the cap is removed from the acid bath, washed off with distilled water and dried. The rubber tubes are removed and the outside of the cap again wiped off, dried and the two parts of the cap weighed separately.

If the cap contains a reinforcing capsule, this may often be removed without force other than the use of the fingers. The contents of the parts of the cap may be safely removed from the copper shell by moistening with distilled water and probing the exposed surface of the adequately moistened explosive by means of a hard wood stick (about $\frac{1}{8}$ " diameter and $3\frac{1}{2}$ " long, sharpened to a rather blunt point). Metal or glass probes should not be used.³⁰ By observation of the color of the loosened material, one may obtain some information as to its probable chemical composition, and oftentimes a fairly accurate physical separation can be made of different cap constituents. Light greyish or brownish grey powdery material probably contains mercury fulminate (80-90%) and chlo-

²⁹ *Supra* note 22(d) at p. 12.

³⁰ If one is examining a fuse cap which does not contain a reinforcing capsule, it will not be necessary to cut the shell by means of acid corrosion. Such a cap may be safely unloaded by probing the well moistened explosive with the sharpened hardwood stick.

rate of potash (10-20%), while pale or darker yellow material is very likely a nitro-aromatic explosive such as tetryl, TNT, or picric acid. Dark brown, crumbly material (from Hercules caps) is probably dinitro diazophenoxide, while bluish white or bluish material (as in Trojan caps) is possibly nitro-starch diphenylamine combination. The loosened material is washed into a beaker and the newly moistened, exposed surface of the explosive further probed with the sharpened stick. In this way, the entire contents may be loosened and washed into the beaker. The detonating explosive is always found in the base of the capsule. The fragments of the capsule are washed out with acetone, dried, weighed and their length measured. From the decrease in length, one may estimate the weight of copper corroded away in the opening of the cap. The weight of the copper capsule and reinforcing cup subtracted from the original weight of the cap, give the weight of the explosive contents.

In analyzing the explosive contents of the cap, there is no simple outline which may be used in all cases. For details of analytical procedures the investigator is referred to Taylor and Rinckenbach's work and also to Kast and Metz's book.³¹ A procedure which we have found to be generally satisfactory for most caps is as follows:

The explosive contents of the cap contained in the beaker, along with considerable distilled water, are carefully crumbled by means of a flattened stirring rod. The beaker containing this powdered material is then permitted to stand at a slight angle so that all solid material will settle to one side of the bottom. The supernatant clear liquid is carefully decanted from this beaker into a dry tared beaker and evaporated nearly to dryness, then allowed to dry spontaneously at room temperature, finally placed in a desiccator over anhydrous magnesium perchlorate for twenty-four hours and weighed. The residue so obtained should be potassium chlorate, although it may also contain picric acid if this were present in the detonating charge. If the extract is markedly yellow, the dried residue should be treated with ether. This will dissolve any picric acid which may be present and leave the potassium chlorate behind. The chlorate may be determined by sulphur dioxide reduction to chloride, with subsequent determination of the chloride by means of silver precipitation as outlined under the analysis of explosives.

³¹ See Taylor and Rinckenbach, *supra* note 22, and also Kast and Metz, No. 15 of bibliography at end of this paper.

The material remaining from the water extraction is dried in a desiccator over anhydrous magnesium perchlorate and then extracted at room temperature with pure anhydrous benzol. The benzol extracts are combined and evaporated nearly to dryness. The remainder of the benzol is permitted to evaporate spontaneously and the residue finally dried in a vacuum desiccator and weighed. The extract contains tetryl, TNT, the remainder of the picric acid not previously removed in the water extract, also some dinitro-diazo phenoxide if this is present. The extract is re-dissolved in benzol and crystallization is allowed to take place slowly so as to obtain well formed crystals. These are then identified by their melting point³² as well as by their typical color reaction with alkalis.³³



FIGURE 12

Photomicrograph of Mercury Fulminate Crystals (100X).

³² The melting point of common nitro explosives found in blasting caps is: Tetryl, 129-130° C.; Picric acid, 121.8° C.; TNT, 80.3° C.; Eutectic of TNT (66%) and Picric acid (34%) is 59.5-60° C. See Taylor and Rinkenbach, "The Freezing Point-Solubility Diagram of the System TNT-Picric Acid," *Industrial and Engineering Chemistry* 15:795 (1923).

³³ See note 26.

The residue remaining after the benzol extraction is dried at room temperature and extracted with pure anhydrous acetone. The acetone extracts are combined and evaporated nearly to dryness at a temperature not in excess of 35° C. The dried acetone extract is weighed. If there is any considerable amount of material extracted by the acetone it should be tested for its solubility in chloroform. If insoluble in chloroform it is not due to previously unextracted nitro-explosives but to nitro-starch.

The residue from the acetone extraction is moistened with water and examined under the microscope. Mercury fulminate is characterized by its typical diamond shaped crystalline form. (Fig. 12.) Lead azide is a fine white or light grey powder, while lead styphnate (trinitroresorcinat) forms orange yellow hexagonal crystals. It is barely possible that one might find stibnite (antimony trisulphide) present in such a residue. If so, it would be found as metallic black opaque crystalline fragments.

The mixture is treated with dilute hydrochloric acid to dissolve the residue. The resulting solution is tested for lead and for mercury. These metals may then be determined quantitatively—the lead by precipitation as sulphate and the mercury by electro-deposition from an alkaline cyanide solution on a weighed platinum gauze cathode.

A typical No. 6 fuse blasting cap has the structure and composition shown in Figure 13.

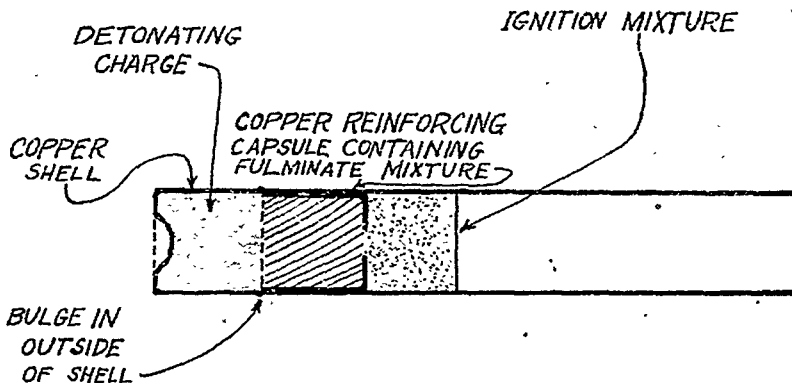


FIGURE 13

Section of a No. 6 Blasting Cap: Length—1½ inches; Outside diameter—.234 inches; Weight—2.10 grams; No identifying marks on base; Detonating charge—.249 grams tetryl; Exploding charge—.325 grams mercury fulminate (90%) and potassium chlorate (10%) mixture; Ignition mixture—100mg.

EXAMINATION OF FUSE

Except for the occasional use of pyrotechnic fuse, practically all fuse-ignited bombs intended for criminal purposes employ standard safety fuse such as is commonly used in mining, quarrying, and highway construction. Safety fuse has a diameter of .19 to .21 inch and has a core of black gunpowder. This powder may be in large granular form or in the form of a core molded or cast around the center thread. The composition and granular form of the powder controls its speed of burning. This velocity is ordinarily 30 or 40 seconds per foot but varies $\pm 10\%$ in various lots of fuse and also depends upon the altitude at which the fuse is used.³¹ This gunpowder is held in place by a number (usually ten) of spirally wrapped jute cords. These jute cords in turn are held in position by other wrappings of thread, paper or tape heavily covered in most instances by pitch for the purpose of making the fuse water-proof and preventing flashes of fire ("side-spitting") from issuing from the sides of the fuse during its burning. A type of detonating fuse known as "Cordeau" has been used to a considerable extent in commercial work but has not found application in criminal bombings. It consists of small lead pipe (.225 to .235 inch outside diameter) containing a core of cast TNT (.17 inch diameter). When attached to a blasting cap and detonated thereby, Cordeau fuse will transmit the explosive wave instantaneously throughout its length. The detonation speed of cast TNT is 17,500 feet per second.

Physical Examination:

The outside diameter of the fuse is measured and the outer covering described. (See Figure 14.) If a sufficient length of specimen is available, a one-foot strip should be ignited and its burning time noted with a stop watch. Another length of about 3 or 4 inches is opened by removing the outer covering or layer and progressively removing subsequent layers one by one until the powder core is reached. Before removing each layer, note should be made of the direction of winding as either "S" winding or "Z" winding (the slant of the winding as compared to the middle portion of the letters "S" or "Z"); also the number of turns per linear inch as indicating the pitch or slope of the winding. Where the windings are sealed

³¹ Snelling, W. O. and Cope, W. C., "The Rate of Burning of Fuses as Influenced by Temperature and Pressure," U. S. Bureau of Mines, Technical Paper No 6 (1912).

Hall, C. and Howell, S. P., "Investigations of Fuse and Miners' Squibs," U. S. Bureau of Mines, Technical Paper No. 7 (1912).

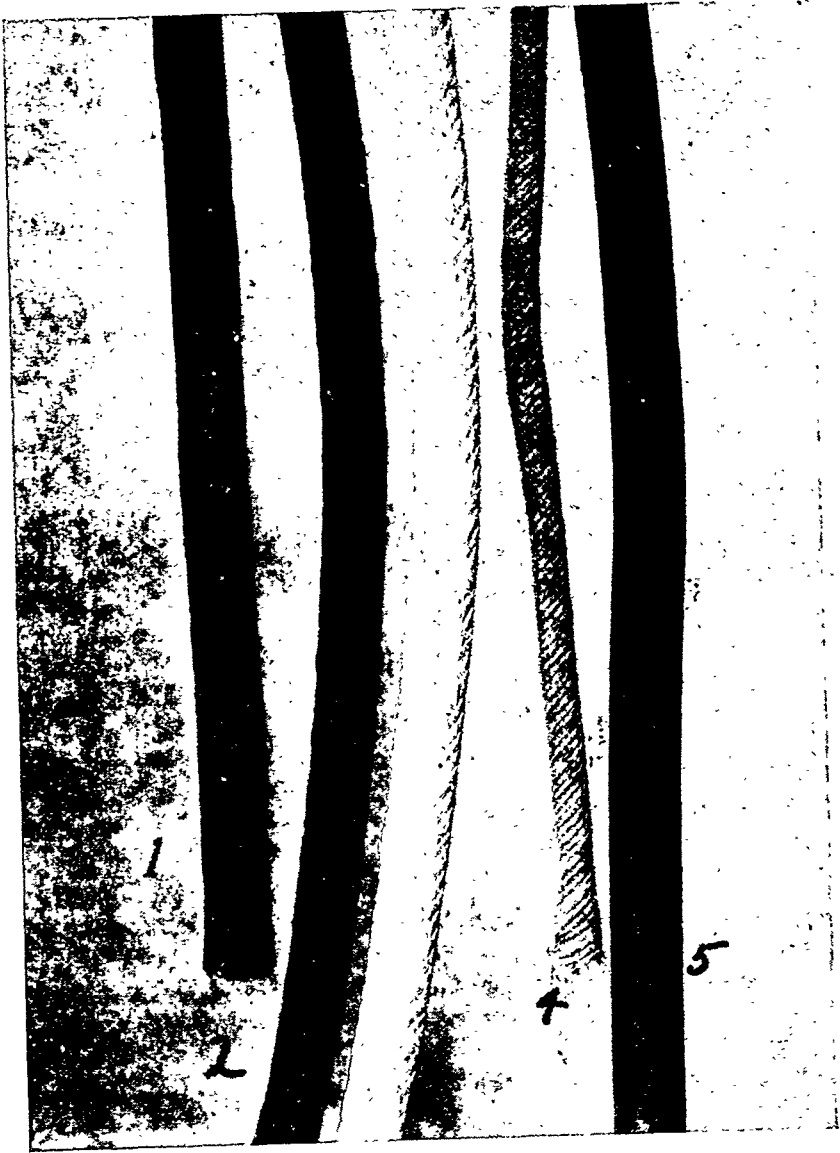


FIGURE 14

Types of Fuse: (1) Single tape safety fuse; (2) Black corded safety fuse ("Black Aztec"); (3) White corded safety fuse ("Bear Brand"); (4) Fireworks fuse; (5) Cordeau detonating fuse.

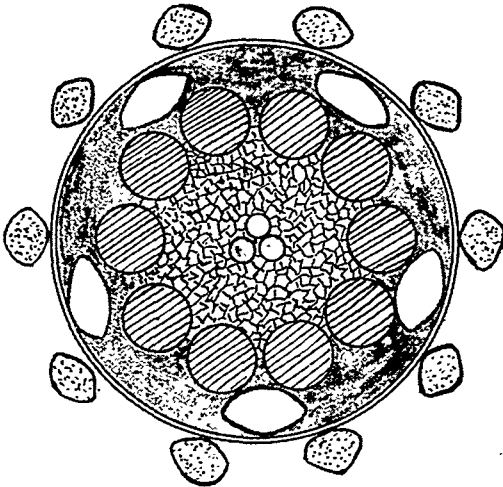


FIGURE 15

CROSS SECTION OF SAFETY FUSE.

A schematic diagram showing structural elements: (1) Outer covering of ten white 2-strand cotton threads S wound and covered with white paint; (2) Light tan paper strip, $\frac{5}{8}$ inch wide wound laterally; (3) Five white 2-strand cotton threads Z wound and covered with pitch; (4) Ten jute cords S wound; (5) Crushed black gunpowder; (6) Two white and one red cotton 3-strand center threads.

with pitch and cannot be peeled off because of its adhesive action, the pitch is dissolved out with carbon tetrachloride. In this fashion the various layers of the fuse may be separated. (See Figure 15.) The thread, cord, tape or paper strip constituents should each be measured and described as to material, color, width, size³⁵ and strand composition (number of strands per thread). Finally, the center thread or threads are similarly examined. The color, number and strand composition of the center threads are of great importance in identifying the source of the fuse. With this information concerning the structural characteristics of the fuse, one may identify it by means of the following outline, providing the fuse was manufactured in the United States:

KEY TO IDENTIFICATION OF SAFETY FUSE MANUFACTURED
IN THE UNITED STATES

I. *Fuse with Outer Tape Covering*

A. Contains a *Single Tape Coat*

1. Contains single 2-strand white center thread = *Comet Brand (C)*

³⁵ There is no really standard method for designating thread sizes. Probably the best recognized standard is the U. S. Federal Standard Stock Catalog Specification V-T-276A, in which the size (ticket number) is based upon the number of strands and the weight per unit length.

2. Contains single 3-strand or 4-strand white center thread = *Single Tape* (E)
 - B. Contains *Two Tape Coats*
 1. Contains single 2x2* strand white center thread = *Double Tape* (N)
 2. Contains single 3-strand or 4-strand white center thread = *Double Tape* (E)
 - C. Contains *Three Tape Coats*
 1. Contains single 4-strand white center thread = *Triple Tape* (N)
 2. Contains single 2-strand white center thread = *Triple Tape* (C)
- II. *Fuse with Black Corded Outer Covering*
- A. Contains a Paper Layer
 1. Outer covering is ten 3-strand threads = *Black Aztec* (N)
 2. Outer covering is ten 2-strand threads = *Black Sequoia* (C)
 - B. Contains no Paper Layer
 1. Has one layer of jute cords (single countered)
 - (a) Contains single 2-strand white center thread = *Dreadnaught* (C)
 - (b) Contains single 4-strand white center thread = *Black Monarch* (N)
 2. Has two layers of jute cords (double countered)
 - (a) Outer jute layer has 5 cords = *Sylvanite* (N)
 - (b) Outer jute layer has 6 cords = *Blue Label* (C)
- III. *Fuse with White or Grey Corded Outer Covering*
- A. Contains no Tape or Paper Layer
 1. Has single layer of jute cords = *White Finished Monarch* (N)
 2. Has two layers of jute cords = *Charter Oak and Grey Charter Oak* (E) depending upon color of outer coating
 - B. Contains a Paper Layer
 1. Has three white center threads = *White Sequoia* (C) and *Victor* (C) Brands

*2x2 strand thread is thread composed of two strands, each consisting of two strands twisted together. It is different from four-strand thread.

2. Has two white and one red center threads = *Clover Brand* (E)
- C. Contains a Tape Layer
1. Outer covering has ten 3-strand threads = *Bear Brand* (N)
 2. Outer covering has ten 2-strand threads
 - (a) Third layer is five 2-strand threads = *Crescent Brand* (E)
 - (b) Third layer is 3-strand threads and has two white cotton 2-strand center threads = *Eagle Brand* (B)
 - (c) Third layer is six 2-strand threads = *Bear Brand* (C)

IV. Fuse with Colored Outer Covering

- A. Orange outer covering = *Orange Clover Brand* (E)
- B. Yellow outer covering = *Beaver Brand* (E)

Key—(E) = Fuse manufactured by Ensign-Bickford Company, with factories at Simsbury, Connecticut, and Avon, Connecticut.

(N) = Fuse manufactured by National Fuse and Powder Company, Denver, Colorado.

(C) = Fuse manufactured by Coast Manufacturing and Supply Company, Livermore, California.

(B) = Fuse manufactured by Blight's Son and Company, Eagle River, Michigan.

Another length of fuse (one foot if available) should be carefully slit with a sharp knife or razor blade and the powder contents collected for examination. The weight of the powder in a measured length of fuse is determined, and this powder is then placed in a desiccator over concentrated sulphuric acid for 24 to 48 hours. The loss in weight represents the moisture content of the sample. The specimen is then transferred to a series of 3-inch diameter sieves (20, 40, 60, and 80 mesh) and shaken. The amount remaining on each screen is weighed and the size distribution noted. The remaining material is used for chemical analysis.

Chemical Analysis of Fuse Powder:

The analysis of black powder is very simple and involves an extraction with water to obtain the water-soluble salts (potassium nitrate), drying the residue at room temperature and finally in a desiccator, then extracting with pure carbon disulphide to extract the sulphur. The evaporation of the solvent in each instance will give the extracted material which may then be weighed and the percentage calculated. The residue insoluble in water and carbon disulphide is the charcoal used in the manufacture of the powder. A typical result of the examination of fuse powder is given for the contents of fuse represented in Figure 15:

Weight of powder per linear foot of fuse is 1.31 grams; burning speed of fuse observed is 40.7 seconds per foot. The powder composition:

<i>Physical</i>		<i>Chemical</i>	
On 20-mesh screen.....	1.0%	Potassium Nitrate	71%
On 40-mesh screen.....	32.7%	Sulphur	19%
On 60-mesh screen.....	34.2%	Charcoal	10%
On 80-mesh screen.....	19.0%		
Through 80-mesh screen.	13.1%		
			100%
	100.0%		

Oftentimes unexploded bombs will be found where the failure to ignite is traceable to the faulty use of fuse. After lengths of fuse have been cut for some time, the exposed powder absorbs moisture and may fail to burn. If such a fuse is crimped into a blasting cap, it may fizzle out before exploding the cap due to the damp powder at the end. If the fuse has been bent many times at a sharp angle it may break the powder core so badly as to cause misfire. Likewise, contact of the fuse with oil or grease may cause the powder to become comparatively non-inflammable because of this contamination.

THE SAFE DISPOSAL OF EXPLOSIVES

Where a hazardous explosive such as nitroglycerine is involved as evidence in a case, it is safe practice to dilute the material (after analyzing it) with a definite amount of *pure* methyl (wood) alcohol. Nitroglycerine dissolved in ten times its volume of pure methyl alcohol is entirely safe from the explosive standpoint. Such

a solution will burn vigorously but will not explode. If necessary, one can always evaporate off the alcohol to obtain the sample again.

Where one has to dispose of explosives, this may be safely done in two ways. The explosive may be spread in a very thin layer in an open field away from all buildings and burned, or it may be destroyed chemically. The latter is oftentimes the more time-consuming operation, but its results are certain and there is less opportunity for mistakes of judgment. For destruction of the explosive, it is placed in an appropriate sized container (a bucket or metal wash-tub) and treated with at least ten times its volume of alcohol (this may be denatured or commercial grade but should not contain more than 5 to 8% water). Any lumps of explosive are broken up with a heavy broomstick.

In another tub or bucket, an alcoholic solution of lye (commercial sodium hydroxide) is made, using the same weight of lye as the estimated weight of nitroglycerine in the explosive to be destroyed. This alcoholic solution is made to about 10% strength (9 pounds of alcohol per pound of lye). When the lye has dissolved and the solution cooled to room temperature, the alcoholic explosive mixture is poured into it slowly and with constant stirring. Care should be taken not to splash caustic soda (lye) solution on the skin, clothing or especially into the eyes, for it is exceedingly corrosive to textiles and skin and can easily cause permanent blindness. For really safe work, water-tight goggles should be worn during the manipulation of the caustic soda. Likewise, spills of nitroglycerine or nitroglycerine solutions on the skin give rise to the very distressing and possibly dangerous physiological effects of its skin absorption. Stirring of the solutions may be done with a stick or barrel stave. The addition of the alcohol solution of the explosive should be stopped if the heat of the chemical reaction becomes too great. As soon as the mixture cools down, more nitroglycerine solution may then be added. In this way one may safely destroy a quart of nitroglycerine in less than an hour. The reaction mixture which results, is no longer explosive and can safely be dumped down the drain into the sewer.

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