# DEPARTMENT OF THE INTERIOR, BUREAU OF MINES, BULLETIN 51. THE ANALYSIS OF BLACK POWDER AND DYNAMITE

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JOSEPH A. HOLMES, DIRECTOR

# THE ANALYSIS OF BLACK POWDER AND DYNAMITE

BY

WALTER O. SNELLING AND C. G. STORM

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# THE ANALYSIS OF BLACK POWDER AND DYNAMITE.

By WALTER O. SNELLING and C. G. STORM.

## INTRODUCTION.

Although descriptions of the methods of analysis of explosives are to be found in many books on explosives, and in works on engineering chemistry or chemical analysis, most of these descriptions are incomplete and lacking in details. The methods of analysis employed in the laboratories of most explosives factories are frequently treated as trade secrets, and very little information is published from such laboratories.

This bulletin outlines the methods of analysis that are used by the Bureau of Mines in the examination of certain classes of explosives. The present form of most of these methods has been worked out in the bureau's explosives laboratory. The methods employed by Prof. C. E. Munroe were taken as a basis, and were elaborated to meet the demands incident to the treatment of complicated mixtures and to the development of the explosives art. A subsequent bulletin will discuss the methods of analysis of "permissible" explosives, many of the latter being of decidedly complicated character and requiring special treatment. This bulletin presents the methods of analysis of "ordinary" dynamite, and the ammonia, gelatin, low-freezing, and granular dynamites, and the common grades of black gunpowder and black blasting powder. The bulletin is published by the bureau for the information of all persons interested in explosives and their safe and efficient use in mining work.

As the term "ordinary" dynamite, though much used, has no conventional meaning, and may be used to cover a wide variety of compositions of matter, it may be noted that the standard dynamite used at the Pittsburgh testing station is a good example of the "ordinary" dynamite known in this country. This testing station dynamite has the following composition:

*Composition of Pittsburgh testing station dynamite.*

| | Per cent. |
|---|---|
| Nitroglycerin | 40 |
| Sodium nitrate | 44 |
| Wood pulp | 15 |
| Calcium carbonate | 1 |

As most permissible explosives contain only the constituents found generally in the various types of ordinary dynamite, the chemist will usually find it possible to analyze such explosives either wholly or partly by following the general methods of analysis here given for the type of explosive that seems most closely related to the one under examination. The methods of extraction with ether, with water, etc., here outlined are general methods which are applied with equal success to all classes of explosives, and therefore by the use of these general methods, following a thorough qualitative examination, little difficulty should be met except with those classes of permissible explosives that contain large amounts of salts holding water of crystallization, such as alum and magnesium sulphate, or those containing an unusual number of uncommon constituents. Even with such explosives, however, if the information desired is principally in regard to the percentages of explosive ingredients (nitroglycerin, ammonium nitrate, etc.), the methods outlined in this bulletin may be satisfactorily followed.

## DYNAMITE.

"Ordinary" dynamite consists essentially of nitroglycerin absorbed in some porous material. Owing to its physical condition and its extreme sensitiveness to shock, liquid nitroglycerin is not suitable for use as an explosive in mining and quarrying, but when nitroglycerin is absorbed in a porous material a more or less plastic mass is obtained which is far less sensitive to shock than liquid nitroglycerin, although, when properly fired by means of a detonator, it retains most of the explosive properties of nitroglycerin. Among the many substances that have been used as absorbents for nitroglycerin are sawdust, wood pulp, ground mica, and infusorial earth (kieselguhr), or mixtures of these substances with alkaline nitrates and other substances.

It is usual to classify absorbents for nitroglycerin as active and inactive. Pulverized gunpowder, for example, or mixtures of wood pulp with sodium nitrate or other oxidizing agents, represent "active" absorbents, whereas mica, kieselguhr and similar materials, which play no part in the explosive reactions and which are employed merely to absorb or retain the liquid nitroglycerin, form the so-called "inactive" absorbents.

The type of dynamite most generally used to-day consists of nitroglycerin absorbed in a mixture of wood pulp and sodium nitrate, and to this mixture is usually added a small amount of some antacid such as calcium carbonate, magnesium carbonate, or zinc oxide. This antacid is added in the belief that it increases the stability of the resulting explosive by neutralizing such small amounts of free acid as may be produced by the decomposition of the nitroglycerin during long storage.

The analysis of dynamite is best carried out by first separating, with ether or some other appropriate solvent, the nitroglycerin from the dope in which it is absorbed. After the nitroglycerin has been thus removed, the soluble nitrate in the dope may be removed by dissolving in water; the antacid may then be dissolved in dilute acid, and the residue insoluble in ether, water, dilute acid, etc., may be directly determined by weight.

In its simplest form, therefore, the analysis of dynamite consists in the removal of the constituent materials, one by one, through the use of appropriate solvents. Dynamites of the most complicated composition may usually be analyzed in this way, through selective solution. In the present paper the methods of analyzing ordinary types of dynamite are discussed, and those that have been found best in an experience covering several thousand analyses are stated.

## PHYSICAL EXAMINATION.

Upon receiving a sample of explosive for analysis it is desirable to record full information in regard to the size and weight of each cartridge, with a complete copy of any lettering that may appear on the wrapper. It is also advisable to record the nature of the outer wrapping paper (such as ordinary paper, parchmentized paper, or paper coated with paraffin), and whether the cartridge has been redipped; that is, placed in a paraffin bath after being filled. Whether a cartridge has been redipped can usually be determined by carefully opening the wrapper. If there is a greater thickness of paraffin near the edge where the sheet overlaps, or if the overlapping edge is attached to the adjacent portions of the paper by means of an adhering deposit of paraffin, it may be assumed that the cartridge has been redipped.

### DETERMINATION OF GRAVIMETRIC DENSITY.

It is possible to determine approximately the gravimetric density or apparent specific gravity of a cartridge of explosive by measuring carefully the length and circumference of the cartridge, calculating from these figures the volume in cubic centimeters, and then dividing the weight in grams of the cartridge by this figure. However, experiments made at the bureau's explosives laboratory have shown that even with the most careful measurements the figures thus obtained are liable to be in error by as much as 10 to 20 per cent, a difference entirely too great to make the method permissible for exact work. With some redipped cartridges weighing in water has given satisfactory results, but cartridges seldom have a coating of paraffin so complete as to permit the use of this method. Accordingly a method was sought that would at all times give satisfactory results even with cartridges that had not been redipped.

The volume of the cartridge can be determined conveniently by using sand instead of water as the measuring material. A weighed glass cylinder about 30 cm. high and 5 cm. in inside diameter is filled with fine sand (preferably sea sand) that has been sifted through a 60-mesh sieve. A straight edge is drawn across the top of the cylinder, the level of the sand being left flush with the top edge, and the weight of the cylinder and contained sand is determined. From this weight the weight of the cylinder is subtracted and the result is the weight of the sand, which, divided by the weight of water required to fill the cylinder, gives the apparent specific gravity of the sand used. All the sand except enough to fill the cylinder to a depth of about 1 inch is now poured out, a weighed cartridge of the explosive is placed in the cylinder, and sand added until the cylinder is filled flush to the top as before, when it is struck with the straight edge and then the weight of cylinder and sand and cartridge is noted. From these figures the weight of sand displaced by the cartridge is found. This weight divided by the apparent specific gravity of the sand gives the volume of the cartridge. The weight of the cartridge divided by its volume gives its apparent specific gravity or gravimetric density. This determination leaves the cartridge in condition for use in sampling, if desired.

In making this determination care should be taken that the cylinder is filled each time in exactly the same manner, the sand being poured in slowly and not packed by jolting, shaking or otherwise. Repeated determinations of the weight of sand required to just fill the cylinder will prove that with proper care uniform results may be obtained; in practice this method has been found to be both rapid and exact.

### TEST FOR LIABILITY OF EXUDATION.

To determine whether there is liability of leakage of nitroglycerin from cartridges containing this explosive, it is always advisable to make an exudation test, which indicates the amount of nitroglycerin that may be lost by the explosive tested under prescribed conditions. The tests most commonly used for this purpose are the 40° test, the pressure test, and the centrifugal test.

#### 40° TEST FOR EXUDATION.

In the 40° test a cartridge of the explosive under examination is placed in a vertical position in an oven heated to 40° C. Some small perforations are made in the wrapper at the ends of the cartridge, and the cartridge is then placed on end on a small wire tripod in a