

This table reveals several points regarding the action on metals of a hard water containing an excess of carbon dioxide with considerable magnesia, and little sulphate and no chlorine.

1. Such a water acts readily upon zinc, far more readily than does distilled water, and it seems to be able to dissolve out the zinc in brass, leaving the brass much corroded. This would explain the action of the water on brass faucets, etc., above mentioned. Such a water should not be conveyed through zinc-lined iron pipes.

2. The hardness of such a water is no protection against its action on lead, this water having more than half as much action as distilled water when protected from the air.

3. Protected from the air, the action upon iron is comparatively slight.

4. Aluminium is perfectly resistant against a water of this character.

Similar experiments were carried out with nickel, but in no case did the water have any action upon it.

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## NOTES ON THE ANALYSIS OF DYNAMITE AND GELATINE-DYNAMITE.

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### I.

THE analysis of dynamite is not often described in text-books and the following notes, gleaned from some years' experience, may be of interest to those who have occasion to investigate such products.

A simple case is that of a dynamite containing nitroglycerine, sodium nitrate, wood-pulp, and basic magnesium carbonate or kieselguhr. The following process of analysis is recommended as giving satisfactory results. Weigh out ten grams on a pair of watch-glasses, place in a desiccator over sulphuric acid and leave for at least five days. The loss in weight is called moisture. Weigh about the same amount into a Gooch crucible and extract with pure ether. A drop of the ether is allowed to evaporate on a piece of tissue paper from time to time, and the extraction continued until the paper no longer tastes of nitroglycerine. Dry the residue in an air-bath at about 80° C. for two to three hours

and weigh. The loss minus the moisture is called nitroglycerine. In the case of powders containing only nitroglycerine and kieselguhr, this extraction is considerably more difficult, and it is advisable to remove the dynamite from the Gooch crucible to a small beaker and stir up with ether, washing the residue back when free from nitroglycerine. After extracting the nitroglycerine, the niter is extracted with cold water, and after the water ceases to extract anything it is displaced by acetone with the object of drying the residue without affecting the starch if any be present. The loss in weight is called sodium nitrate, potassium nitrate being very rarely used. The residue from the water extraction is ignited until free from carbon. The loss is wood-pulp, flour, bran, etc. I have found no satisfactory method for separating these, and rely on testing with iodine for the presence of starch as an indication of the presence of flour, etc.

If no earthy absorbents have been used, the ash after ignition is not likely to be over one-tenth per cent. of the whole sample. If the ash is considerable it is extracted with hydrochloric acid and the residue dried. The loss, expressed in percentages, is subtracted from the percentage of loss on ignition. The loss on ignition minus the loss on extraction with hydrochloric acid is called wood-pulp, etc., while twice the latter is entered as basic magnesium carbonate. This method saves a quantitative determination of the magnesia in the extract and rests on the fact that the basic magnesium carbonate employed as an absorbent loses practically one-half its weight on ignition. The extract should, however, be examined for lime, etc.; a small amount of iron may come from the kieselguhr. If any residue be left after extracting with hydrochloric acid it is examined under the microscope for kieselguhr, mica, etc., or it may be a small amount of venetian red used for coloring the powder.

The following analysis will serve to illustrate the methods:

No. 515.	Per cent.	Per cent.
Moisture .....	1.3	....
Nitroglycerine .....	39.6	39.6
Sodium nitrate .....	46.8	47.1
Wood-pulp .....	9.5	....
Basic magnesium carbonate.....	1.8	....
Kieselguhr .....	1.0	....
	<hr/>	
	100.0	

The loss on ignition was 10.40 per cent. and on extraction with hydrochloric acid 0.90 per cent. A quantitative determination of the magnesia, calculated as  $\text{Mg}(\text{OH})_2 \cdot 3\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$ , gave one and eight-tenths per cent. The portion used for moisture determination may be used to check the extractions.

Lunge's nitrometer may be used to advantage as a check on the analysis. About one-half gram of the dynamite is weighed into a small weighing bottle and about ten cc. of pure sulphuric acid poured over it. It is then allowed to stand from twelve to eighteen hours. In this time the whole usually goes into solution. In the cold the reaction is perfectly calm and no nitrogen is lost. It is then introduced into the nitrometer and decomposed as usual. The result is most conveniently expressed in cubic centimeters of nitric oxide per gram. On the sample mentioned above three determinations in the nitrometer gave: 237.0, 234.5, and 236.5 cc. of nitric oxide per gram. 39.6 per cent. of nitroglycerine gave 116.9 cc., 46.8 per cent. of sodium nitrate gave 122.9 cc. of nitric oxide per gram respectively, total 239.8 cc. The sodium nitrate used is generally ninety-six to ninety-eight per cent. pure; assuming ninety-seven per cent., reduces the calculated cubic centimeters to 236.1. The nitrometer work shows that in this case the ether-soluble portion contains nothing but nitroglycerine, the water-soluble portion nothing but sodium nitrate and its usual slight impurities. Eight minutes agitation in the nitrometer is sufficient for almost all cases.

## II. GELATINE-DYNAMITE.

The gelatine-dynamites are considerably more difficult to analyze. The following analysis may be selected as typical:

No. 478.	Per cent.
Moisture .....	0.4
Nitroglycerine.....	33.7
Sulphur .....	1.9
Resin .....	7.0
Sodium nitrate .....	54.0
Guncotton .....	1.1
Wood-pulp .....	1.9
	<hr/>
	100.0

The sample for moisture is weighed out as before. A filter cone (C. S. & S.) is dried and weighed in a weighing bottle and

about fifteen grams of the powder weighed into it. This is extracted in a Soxhlet apparatus with chloroform. The loss includes nitroglycerine, sulphur and resin. Sometimes the powder melts together in the hot chloroform, and in that case it must be taken from the filter and broken up in a beaker with cold chloroform until the bulk of the nitroglycerine is dissolved. After extracting with chloroform the residue is extracted with cold water to remove the sodium nitrate, dried and weighed, then extracted with acetone and the loss on extraction with acetone called guncotton. The still remaining portion consists of wood-pulp and earthy absorbents which are treated as above described. Sodium nitrate is appreciably soluble in acetone and should, on that account, be removed before the guncotton. It is often recommended to extract with acetone first and precipitate the guncotton with chloroform. This method gives results which are too high because the sodium nitrate dissolved by the acetone is also precipitated along with the guncotton. Allowance for this can, however, be made by incinerating the guncotton and calculating the ash as sodium nitrate. The second and following extractions can conveniently be made in the filter cone, setting it in a test-tube with perforated bottom. The filter is finally incinerated in a platinum dish with the residue, allowance being made for its ash content which in one case was 11.4 milligrams.

The chloroform solution is evaporated almost to complete absence of chloroform and taken up with glacial acetic acid. The precipitated sulphur is washed into a weighed Gooch crucible and washed further with a little strong alcohol to remove the resin. The nitroglycerine is determined by weighing a suitable portion of the powder (in this case one and two-tenths grams) into a small beaker. Ether is then poured on, and the powder worked over with a platinum spatula. The ether is several times renewed, being poured on a filter and collected in a glass evaporating dish. The ethereal solution is allowed to evaporate spontaneously in a cool place, the dish being covered with tissue paper. After twelve hours the residue can be washed into the nitrometer with acetic acid, care being taken not to use too much acetic acid which retards the reaction of decomposition, while if too little be used the reaction may be

extremely violent. Even with the greatest care in evaporation some nitroglycerine is lost and the results are usually about one per cent. low.

The calculated cubic centimeters of nitric oxide per gram on this analysis, assuming the guncotton to contain 11.9 per cent. of nitrogen, are 99.5 for the nitroglycerine, 141.7 for the sodium nitrate, and two and one-tenth for the guncotton; total 243.3. Two determinations in the nitrometer gave 243.8 and 242.7. As the sodium nitrate was certainly not pure, this result shows that the figure entered for nitroglycerine must be too low. Assuming the sodium nitrate to be ninety-eight per cent. pure gives 138.9 cc. and this, plus 2.10 cc. for the guncotton, subtracted from 243.2, total by analysis, gives 102.2 cc. for the nitroglycerine, corresponding to 34.6 per cent. Of course if necessary the water-soluble portion could be isolated and estimated in the nitrometer. The amount given in the analysis, 33.7 per cent., was determined by analyzing the ether-soluble portion in the nitrometer and calculating the nitric oxide obtained as  $C_3H_5N_3O_9$ . The resin in this case was recognized by its odor, and estimated by difference. An attempt to determine it in alcoholic solution, in presence of nitroglycerine, by titration with alcoholic potash, failed because nitroglycerine by itself is decomposed by alcoholic potash, decolorizing phenolphthalein solution. Some so-called flameless dynamites contain ammonium nitrate. The ammonia is estimated by distillation from aqueous solution and calculated as nitrate.

The two examples given cover the great bulk of dynamites and gelatine-dynamites used in the United States but many other mixtures, often of a very fanciful nature, are occasionally encountered, some of them exceedingly difficult if not impossible to analyze. The methods of analysis given in Guttman's *Manufacture of Explosives*, Vol. II, are mainly reliable as far as they go, while some of Sanford's methods are likely to lead to disappointment. His separation of nitroglycerine from camphor is especially difficult to understand since nitroglycerine and carbon disulphide mix completely on the addition of camphor.

It is hoped that this paper will provoke discussion which may lead to the adoption of uniform methods of analysis by the chemists who have to deal with this class of products.

## III. VARIOUS NOTES.

The nitrometer is conveniently set by the use of pure potassium nitrate. Exactly one-half gram is weighed into a weighing bottle, covered with sulphuric acid and allowed to stand until the solution becomes clear. The nitric oxide from this is brought into the measuring tube, and the amount of air in the reduction tube, which should also contain a few drops of water, is varied until the gas in the measuring tube shows 110.5 cc. with the air in the reduction tube at 100 cc. The use of potassium nitrate eliminates a large number of errors, such as those of reading the barometers, thermometers, etc. The potassium nitrate may be purified by dissolving it in the least possible quantity of cold water and precipitating with an equal volume of pure alcohol. Three times is generally sufficient, and the absence of chlorides and sulphates may be taken as a criterion of purity. Sodium nitrate, made by dissolving sodium in alcohol and neutralizing with dilute pure nitric acid, may be used as a check on the potassium nitrate. Leaky stop-cocks are a frequent source of annoyance in working with the nitrometer. The stop-cock of the measuring tube may be tested each time by allowing it to stay open for an instant after connecting it to the decomposing bulb by means of a thick-walled rubber tube and allowing the mercury reservoir to be at its lowest position. The mercury should not sink in the measuring tube. This assures the tightness of everything from the lower side of the stop-cock in the measuring tube to the upper side of the stop-cock in the decomposing bulb. The stop-cock in the decomposing bulb may be tested by analyzing the standard potassium nitrate, first with the level of the mercury in the reservoir about six inches above that in the decomposing bulb, and secondly about six inches below. If the first analysis shows too low a result, and the second too high, it points conclusively to a leak. A person having much nitrometer work to do must familiarize himself with the grinding of stop-cocks as they rarely leave the manufacturers' hands in a perfect condition. Grease for the stop-cocks is made from vaseline with the addition of Japan wax and rosin, the proportions being varied in summer and winter to get a product of suitable hardness and consistency.

The refraction method for the valuation of Chili niter gives results from one to one and one-half per cent. higher than those obtained by using the nitrometer and calculating the nitric oxide obtained as sodium nitrate. This is due to the almost unvarying presence of potassium nitrate, and the occasional presence of potassium perchlorate. The latter may be determined by fusing at a low temperature in the presence of powdered cupric oxide. The increase of chlorides after fusion is calculated as potassium perchlorate. The following is a complete analysis of one sample:

No. 471.	Per cent.
Moisture .....	2.2
Insoluble.....	0.1
Magnesium sulphate .....	0.2
Magnesium chloride.....	0.1
Sodium chloride.....	0.4
Potassium nitrate.....	3.6
Sodium nitrate .....	93.4
Potassium perchlorate.....	0.0
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The refraction method gave 96.8 per cent. of sodium nitrate by difference.

THE CALIFORNIA POWDER WORKS,  
 PINOLE, CONTRA COSTA COUNTY,  
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## A MODIFICATION OF PIERCE'S METHOD FOR THE DETERMINATION OF ARSENIC IN ORES.

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**T**HERE are several methods for the estimation of arsenic in ores in current use, many of which, however, are defective or do not approach the accuracy required even in technical work.

Pierce's method<sup>1</sup> (one of the best of those in general use), with or without Canby's modification, on account of its simplicity, is largely employed in metallurgical works notwithstanding the fact that it is subject to a range of inaccuracy which renders it inadmissible where accurate results are required, and which can be reduced to a minimum only by the most careful execution.

It is my purpose, after having pointed out the defective points

<sup>1</sup> Proceedings of the Colorado Scientific Society, Vol. I.