

SILVER FROM COPPER, CADMIUM, ZINC, AND NICKEL.

Silver. Gram.	Copper. Gram.	Cadmium. Gram.	Zinc. Gram.	Nickel. Gram.	Potassium cy- anide. Grams.	Dilution. cc.	Current. N.D.100.	Volts.	Temperature.	Time. Hours.	Silver found. Gram.
0.1090	0.0526	0.0564	0.0622	0.0411	4.5	125	0.02 A	1.2	75 ^c	5 ³	0.1086
0.1090	0.0526	0.0564	0.0622	0.0411	3.5	125	0.02 A	1.2	75 ^d	5	0.1085

None of the other metals were found in the silver deposit. The addition of the nickel to the solution seems to retard the precipitation of silver to a slight degree.

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NOTES ON THE ANALYSIS OF EXPLOSIVES.

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DETERMINATION OF SULPHUR.

IN gelatine dynamite containing from 1 to 3 per cent. of sulphur, the following method is useful: Weigh out about 2 grams into a silver crucible of 100 cc. capacity; fill it two-thirds full with an alcoholic solution of caustic soda. Warm carefully on a water-bath until the nitroglycerine is decomposed and then evaporate to dryness. Add next about 40 grams of solid caustic potash and 5 grams of potassium nitrate. Fuse the contents of the crucible carefully over a blast-lamp until all organic matter is oxidized. Dissolve in dilute acetic acid, filter from a small amount of insoluble matter, and precipitate the sulphates with the usual precautions. The results check very closely.

AN INDIRECT METHOD FOR THE ESTIMATION OF NITROGLYCERINE IN GELATINE DYNAMITE, ETC.

About 15 grams of the sample are completely extracted with chloroform in a Soxhlet apparatus and the loss in weight noted. In another portion the moisture is determined by desiccation over sulphuric acid for five days, careful experiments having shown that nitroglycerine is not appreciably volatile in a desiccator. Another portion of about 2 grams is carefully extracted with pure ether by maceration in a small beaker. The ether is poured through a filter and the extraction repeated three or four times. The filtrate is now allowed to evaporate spontaneously or with the aid of a gentle current of air. When the

ether is evaporated add about 5 cc. of ammonium sulphide solution and 10 cc. of alcohol. Warm gently on a water-bath until the nitroglycerine is decomposed and then add about 250 cc. of water and enough hydrochloric acid to give a strongly acid reaction, filter, and wash the precipitate free from acid. Now wash the precipitate out with strong alcohol and chloroform, collecting this filtrate in a weighed platinum dish. Evaporate at a low temperature and dry to constant weight at 50° C. The contents of the dish are now transferred to a silver crucible and the sulphur determined as above described. The increase in weight of the dish less this amount of sulphur represents the chloroform-soluble substances in the original sample except the nitroglycerine, moisture, and sulphur. The percentage of the former substances plus the moisture and sulphur in the original sample gives, when subtracted from the total chloroform-soluble, the percentage of nitroglycerine.

In another portion, the residue in the platinum dish may be investigated for resins, paraffin, etc.

ON THE USE OF LUNGE'S NITROMETER.

All the glass stop-cocks should be tight. In reference to the practical testing of stop-cocks, see this Journal, 21, 430.

Standardizing the Nitrometer.—There are two methods applicable, the first of which I shall call the 'absolute method' and the second the 'empirical method.' The latter was devised by Dr. Clarence Quinan, of San Francisco.

The first consists in admitting to the reduction tube a quantity of air such that if reduced to 0° C. and 760 mm. pressure, it would occupy a volume of 100 cc. The analysis is then made by weighing out a convenient quantity of nitrate, decomposing it in the usual way, introducing the nitric oxide into the measuring tube and regulating the pressure, so as to bring the volume of air in the reduction tube to 100 cc., while the surface of the mercury in the measuring tube is at the same level as the surface of the mercury in the reduction tube. If deemed preferable the level of the mercury in the measuring tube may be made to coincide with the level of the mercury in the reservoir tube so as to make the pressure in the measuring tube equal to the atmospheric pressure. The volume of nitric oxide is then read off and reduced to normal conditions by the usual calculations.

The correctness of the results obtained in this method, aside from errors of manipulation, is affected by the following factors:

- (A) The accuracy of the barometer.
- (B) The accuracy of the thermometer.
- (C) The accuracy of the graduation of the reduction tube.
- (D) The accuracy of the graduation of the measuring tube.
- (E) The accuracy of the weights employed.

It is evident that the work of calibrating all the instruments employed would be very considerable and it is not safe to assume the correctness of such instruments as they occur in trade.

The empirical method is as follows :

A sample of potassium nitrate is purified until it shows the absence of impurities by the ordinary tests. It is then dried and the nitric oxide derived from a known quantity, is passed into the measuring tube. The quantity of air in the reduction tube is then varied until the volume of the nitric oxide is approximately that calculated from the amount of nitrate taken. The reduction tube is now sealed and a series of analyses of potassium nitrate made with slight variations in the amount taken, and in each case a correction factor determined which shows 100 per cent. in purity in the nitrate taken (see example below). The extreme variations in the determinations should not exceed 0.05 per cent. From this series of determinations a correction factor is calculated which is applied to all determinations.

EXAMPLE.

0.5078 gram KNO_3 gave 111.7 cc. nitric oxide.	
Log. 111.7	0.04805
Log. 0.5078	<u>0.70569</u>
Log. of cc. per gram	0.34236
0.5057 gram KNO_3 gave 111.2 cc. nitric oxide.	
Log. cc.	0.04610
Log. s.	<u>0.70389</u>
	0.34221
0.5058 gram KNO_3 gave 111.2 cc. nitric oxide.	
Log. cc.	0.04610
Log. s.	<u>0.70398</u>
	0.34212

The average of these logarithms gives : Log. cc. per gram 0.34223, corresponding to 219.9. The correction is obtained by the proportion 221.0 : 219.9, the first term being the theoretical cubic centimeters per gram for potassium nitrate.

Log. 221.0.....	0.34428
Log. 219.9.....	0.34223
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	0.00205

In other words in any analysis the mantissa of the logarithm representing the cubic centimeters per gram or the percentage of nitrogen must be increased by 0.00205.

EXAMPLE.

0.5913 gram guncotton gave 119.1 cc. of nitric oxide.	
Log. cc.....	0.07591
Log. s.....	0.77181
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	0.30410
	0.00205
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	0.30615

This corresponds to 202.3 cc. per gram or 12.71 per cent. nitrogen.

The advantage of this method lies in the fact that all the sources of constant errors A to E are eliminated, no thermometer nor barometer being needed. The weights may be inexact if only consistent. The graduation of the tubes may be inexact and inconsistent. In fact they could be grossly inaccurate without affecting the accuracy of the results if separate corrections were made for each point on the measuring tubes.

The obvious objection to the method lies in the difficulty of preparing a pure potassium nitrate which shall not have a low content of nitrogen by reason of the presence of foreign matter nor a high content of nitrogen from the presence of sodium nitrate. These objections may be met by preparing samples of potassium nitrate from different sources. All should show the same percentage of nitrogen. Secondly, a sample of nitrate of soda should be purified and the correction factor determined from it independently. Within the limits of experimental error it should be the same as the correction factor derived from potassium nitrate.

EXAMPLE.

0.4302 gram sodium nitrate gave 112.5 cc. nitric oxide.	
Log. cc.....	0.05115
Log. s.....	0.63367
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	0.41748
Log. theoretical cc. per gram for sodium nitrate	0.41954
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	0.00206

As a third check a sample of pure dry nitroglycerine may be used.

At one time in my work the reduction tube was set by aid of a barometer, since shown to be faulty. A sample of potassium nitrate carefully purified showed in this case about 99 per cent. purity and a sample of sodium nitrate also carefully purified showed the same percentage of purity within the limits of error. The barometer setting of the reduction tube was therefore discarded and the correction factor introduced since it was manifest that, while either of the nitrates might be impure even after the most careful purification, it was in a high degree improbable that both should be impure and of exactly the same degree of impurity.

During the hot weather of summer there is a distinct advantage in having a large subtractive correction factor. In this way the gas may be measured nearly at the pressure of the atmosphere, thus avoiding the considerable strain on the stop-cock and consequent tendency to leakage.

The nitric oxide may be measured either dry or moist, more conveniently, however, in the latter condition. If so measured a few drops of water should be left in each tube. As the tension of the aqueous vapor is the same in each tube, it may be neglected in the calculations.

The sulphuric acid used in nitrometer work should be free from oxides of nitrogen and iron. It should be of 94 to 95 per cent. strength. In acid of 98 per cent. strength nitric oxide is quite freely soluble.

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AN AUTOMATIC FILTER-WASHER.

BY J. M. PICKEL.

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THE apparatus shown in the accompanying cut, consists of a battery of ten washers. The parts of each washer are: A reservoir, 1, to contain the liquid with which the washing is done. A rubber tube, 2, provided with two thumb-screw clamps, 3, leading from the reservoir to the delivery vessel. A delivery vessel, 5, provided interiorly with a small siphon, which delivers the washing liquid intermittently in small portions on the substance being washed. A larger funnel, 7, containing the filter. A smaller inverted funnel, 6, covering the substance. The object of this funnel is, while it prevents spattering, to throw the liquid around