ON THE ANALYSIS OF SULPHO-CARBONATES.

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SOLUTIONS of carbon bisulphide in potassium sulphide—so-called sulpho-carbonate, K_2CS_3 —occur at present in commerce, and are valued in proportion to the percentage of carbon bisulphide contained in them. Several chemists have lately published their experience in regard to the analysis of these solutions, but the processes they adopted appear to us defective on the score of either accuracy or convenience. The principle of all the methods is identical; solution of some metallic salt is added, and the precipitated metallic sulpho-carbonate is decomposed by heating into metallic sulphide and carbon disulphide. Thus, Guyot Dennecey (*J. Pharm.* [5] 6, 336, abstract in *Chem. Soc. Journ.*, Feb. 1883) adds the sulpho-carbonate drop by drop to a hot solution of 100 grms. of zinc chloride, contained in a 2 litre flask, distils and measures the carbon disulphide. E. L. de Bouquet (*Mon. Scient.*, 1882, 994, abstract in *Berl. Ber.* 1882, 2933) decomposes with copper sulphate, distils, passing the vapour through alcoholic potash, olive oil and bromine water, estimating ultimately the sulphur as $BaSO_4$.

We find that the process may be very much simplified as follows:—To three to five grms. of the solution, strong cold lead acetate (or other metallic solution) is added, until the liquor in which the precipitate is suspended is colourless, the whole being contained in a small tubulated retort, capable of holding about 6 to 8 ounces. The retort is connected with two nitrogen bulb tubes, filled with *strong* alcoholic potash solution, and kept cool by immersion in water. The contents of the retort are heated to boiling and kept so for about five minutes. The whole of the carbon bisulphide is absorbed by the alcoholic potash, the second bulb tube rarely containing more than traces. The clear contents of the tubes are then washed into a beaker, rendered slightly acid with acetic acid, and the xanthate which has resulted from the combination of the bisulphide with the alcohol, is titrated with copper

sulphate solution, containing per litre 12.47 grms. of crystallised sulphate, 1 c.c. corresponding to .0076 grms. CS_2 (Macagno's solution). The yellow copper xanthate readily conglomerates on agitation, leaving the liquor practically clear. When, on addition of the copper solution, a further precipitate can no longer be observed, a drop of the liquor is taken out with a glass rod, placed on a double piece of filter paper, and the spot on the lower paper is touched with a little ferro-cyanide solution. On the appearance of the faintest pink tint, the amount of copper solution used is read off. The total volume of liquor is then measured, and for every 100 c.c., 1 c.c. of standard solution is subtracted, that amount being necessary to produce, when diluted with 100 c.c. of distilled water, a pink tint with ferro-cyanide on filter paper. The number of c.c. multiplied with .0076 gives the amount of carbon bisulphide obtained. The whole process takes barely 10 minutes.

The following figures will show that the method is sufficiently accurate for practical purposes :—

1.0435 grms. of CS_2 (weighed in a thin glass bulb) were dissolved in alcoholic potash. Copper solution used, 137.2 c.c., minus 2.5 = 134.7 c.c. corresponding to 1.0237 grms. or 98.1 per cent. CS_2 .

.9660 grms. CS_2 used 131.3 c.c. — 2.5 = 128.8 c.c. = .9789 grms. or 101.8 per cent. CS_2 .

.7141 grms. used 94.1 c.c. — 2 c.c. = 92.1 corresponding to .6999 grms. or 98.0 per cent.

.7990 grms. took 108 c.c. — 2.5 = 105.5 c.c., equal to .8018 grms. or 100.3 per cent.

Average 99.4 per cent. CS_2 .

We also made a number of experiments to convert a weighed quantity of CS_2 into sulpho-carbonate by treatment with potassium sulphide, but were not able to dissolve the whole of the quantity taken. Sulphur separated, and obstinately retained some of the carbon disulphide.

33.78 grms. of a commercial sample of sulpho-carbonate were diluted to 500 c.c.

50 c.c., heated as described, yielded an amount of xanthate which used 55.5 c.c. copper solution = .4218 grms. or 12.49 per cent. CS_2 .

50 c.c., ditto, used 57.0 c.c. = .4332 grms., or 12.82 per cent.

The following results show the importance of adding the metallic solution *cold* to that of the xanthate :—

50 c.c. of the above fluid were heated nearly to boiling, and after cooling, mixed with lead acetate. Copper solution used 54.5 c.c. = .4142 grms. or 12.26 per cent. CS_2 .

50 c.c. boiled for 5 minutes previous to the addition of the lead. Copper solution used, 42.5 c.c. = .3230 grms., or 9.56 per cent. CS_2 .

50 c.c. boiled for 8 minutes. Copper solution used, 33.0 c.c. = .2508 grms., or 7.42 per cent. CS_2 .

50 c.c. boiled for 20 minutes, used 16.8 c.c. copper solution = .1277 grms. or 3.78 per cent. CS_2 .

The sulpho-carbonate, by boiling, is well known to be converted into carbonate, $\text{K}_2\text{CS}_3 + 3\text{H}_2\text{O} = \text{K}_2\text{CO}_3 + 3\text{H}_2\text{S}$.

We add a few analyses of commercial samples :—

	Sp. Gr.	Sp. Gr.	Sp. Gr.
	1.413	1.422	1.429
CS_2 11.78 by weight.	10.63	10.84	
K_2O 24.68 per cent.	24.39	24.71	
Na_2O .61	1.29	.74	