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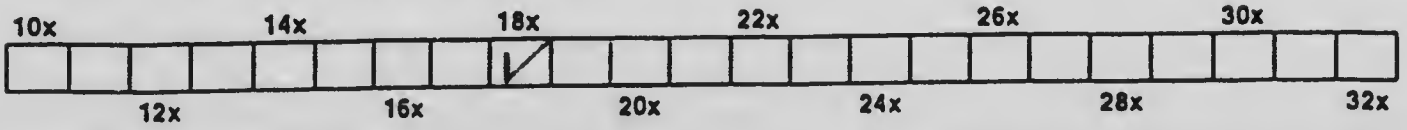

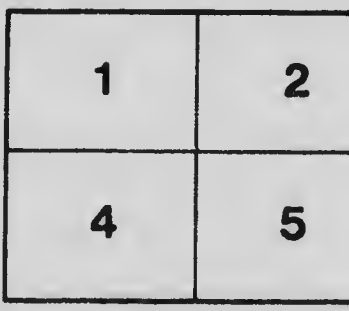

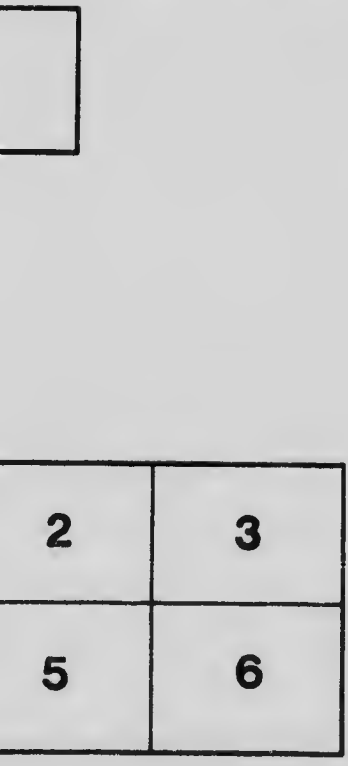

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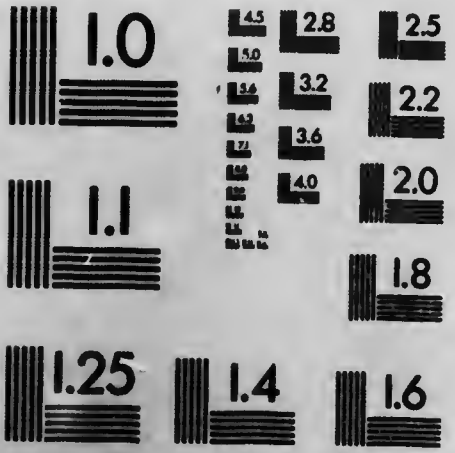

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PAPERS

FROM THE

CHEMICAL LABORATORY.

No. 22.—Solubility of the Sulphides of Arsenic, Antimony and Tin.

BY

W. R. LANG and C. M. CARSON.

Reprinted from the Journal of the Society of Chemical Industry, August 15, 1902.

TORONTO, 1902.

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EYRE AND SPOTTISWOODE, EAST HARDING STREET, E.C.
1902.

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SOLUBILITY OF THE SULPHIDES OF ARSENIC, ANTIMONY, AND TIN.

BY W. R. LANG AND C. M. CARSON.

In a former paper (this Journal, June 16, 1902, 748), an account was given of a short investigation into the accuracy of the methods recommended for the separation of the sulphides of arsenic, antimony, and tin. The following are the results of further experiments with reference to the solubility of the three sulphides in hydrochloric acid:—

I. *Arsenic Sulphide in Hydrochloric Acid.*—One gramme of arsenious oxide was dissolved in hydrochloric acid, and the solution made up to 250 c.c. Into each of several flasks 25 c.c. of the solution were measured, the arsenic precipitated as the sulphide, and filtered.

(*a*) The precipitates were treated with equal volumes of hydrochloric acid of different concentrations and allowed to stand, at the room temperature, for four hours, when they were again filtered, the filtrates diluted with water, and tested by passing in hydrogen sulphide.

Hydrochloric Acid (Sp. Gr., 1·16).	Water.	Filtrate after passage of Hydrogen Sulphide.
c.c.	c.c.	
60	0	Slightly coloured.
50	10	" "
40	20	" "
30	30	Colourless.
20	40	"
10	50	"
0	60	"

(*b*) The sulphides were treated with hydrochloric acid as in (*a*), but, before filtering, hydrogen sulphide was passed

into each. The filtrates were tested as before with hydrogen sulphide

Hydrochloric Acid.	Water.	Filtrate after passage of Hydrogen Sulphide.
c.c.	c.c.	
60	0	Showed no colouration.
40	20	" "
30	30	" "
20	40	" "
6	69	" "

(*c*) The sulphides were obtained as in (*a*), and were again treated with hydrochloric acid, but, instead of being allowed to stand at the room temperature, were boiled for 15 minutes. The mixtures were then filtered, and the filtrates tested.

Hydrochloric Acid.	Water.	Filtrate with Hydrogen Sulphide.
c.c.	c.c.	
60	0	Sulphide had almost completely dissolved.
40	20	Filtrate gave precipitate with hydrogen sulphide.
30	30	" "
20	40	" "
0	60	" "

(*d*) The procedure was the same as in (*c*), except that, before filtering, the mixture was cooled and then saturated with hydrogen sulphide.

Hydrochloric Acid.	Water.	Filtrate when tested with Hydrogen Sulphide.
c.c.	c.c.	
60	0	No colouration.
40	20	"
30	30	"
20	40	"
0	60	"

Arsenious sulphide may, then, be treated with hydrochloric acid (sp. gr., 1·16) without danger of solution, if hydrogen sulphide be passed into the mixture to saturation.

II. *The Solubility of Antimony Trisulphide in Hydrochloric Acid.*—A solution was made containing 1 grm. of antimony tetroxide in 250 c.c.; 25 c.c. of the solution were measured out, the antimony precipitated as the sulphide, and the precipitate filtered. The sulphide was then washed into a flask by means of 8 c.c. of concentrated hydrochloric acid (sp. gr. 1·16) being completely dissolved, but giving an orange coloration to the solution. On addition of another 2 c.c. of acid the coloration disappeared.

The sulphide of antimony was again obtained by precipitation of 25 c.c. of the original solution. This was filtered and dissolved in 50 c.c. of concentrated hydrochloric acid. Into this solution hydrogen sulphide was passed continuously, whilst water was added from a burette. When 18 c.c. of water had been added, a slight colouration was visible, and when the amount of water had reached 21 c.c., the sulphide began to precipitate. To again obtain a clear solution, 8 c.c. of hydrochloric acid were required.

The sulphide of antimony is soluble, then, in a mixture of hydrochloric acid (sp. gr., 1·16) and water in the proportion of 50 to 18, even when hydrogen sulphide is passed in to complete saturation.

III. *Stannous Sulphide* is soluble in hydrochloric acid slightly more dilute than that which is required to dissolve antimony trisulphide.

In order to separate the sulphides of arsenic from those of antimony and tin, the mixture should be treated with hydrochloric acid diluted with not more than one-third of its volume of water, and then completely saturated with hydrogen sulphide. The arsenious sulphide will be undissolved, and on filtering, will be left free from antimony and tin. The precipitate may be washed with a mixture of hydrochloric acid and water in the proportion of 50 to 18, or with hydrochloric acid (sp. gr., 1·16), if hydrogen sulphide be passed into the acid before washing.

This method, which is substantially the same as that given by Pattinson and Pattinson (this Journal, 1898, 211, 214), was employed to estimate the arsenic in 25 c.c. of a solution containing 1 grm. of arsenious oxide, 1 grm.

of antimony tetroxide, and 1 grm. of tin oxide in 250 c.c. The sulphide of arsenic, after separation, was dissolved in nitric acid, the arsenic precipitated by magnesia mixture, and weighed as magnesium pyro-arseniate :—

—	As_2O_3 (calculated).	As_2O_3 (estimated).
(1)	0·1000	0·0981
(2)	0·1000	0·0983

In qualitative work, the solution of the sulphides in yellow ammonium sulphide may first be precipitated by dilute hydrochloric acid. The mixture of sulphides, on being filtered and washed, may then be separated as described above. This is preferable to the method of pouring the solution into concentrated hydrochloric acid.