tory of the first series of experiments, the results of which have been already stated.

Until some other explanation can be found to show why this relation should hold, it seems necessary to conclude that a sulfide of tellurium, having the formula TeS, exists; that it is formed by passing hydrogen sulfide gas into a solution of tellurous acid, according to the reaction

$$2H_2S + H_2TeO_3 = TeS + S + 3H_2O,$$
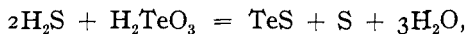
and that this compound decomposes very readily at any temperature higher than $0°$, and that it is unstable even at $0°$, decomposing in about four hours to tellurium and sulfur.

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# PRECIPITATION OF THE COPPER-ARSENIC GROUP AND THE SEPARATION OF ITS DIVISIONS.

By J. I. D. HINDS.

The analysis of solutions containing quinquevalent arsenic and bivalent tin is simplified and expedited by the method here outlined. The facts upon which the method is based are mostly well known, though I have added some definit information, especially as to the loss of arsenous chloride on boiling its acid solution and the solubility of copper sulfide in colorless ammonium sulfide. These facts have been confirmed by careful experiments and are as follows:

1. For the ready precipitation of quinquevalent arsenic by hydrogen sulfide a hydrochloric acid concentration above $2 N$ is required; for the complete precipitation of other members of the group, notably cadmium, antimony and tin, the acid concentration must be below $0.5 N$; to hold in solution the metals of succeeding groups the concentration must be above $0.125 N$. These different concentrations are secured first by evaporation and then by proper dilution.

2. The quantity of arsenous ion lost on long boiling with dilute hydrochloric acid is a *function of the quantity present*, and when a solution normal in HCl is boiled half way, the loss is *less than one thousandth of the quantity present*. I have established this fact by many determinations, an account of which will be given in a subsequent paper. The investigation covered solutions varying in concentration from $0.01 N$ to normal in arsenous ion. The solutions were distilled half way and the quantity of arsenic in the distillate determined.

3. The sulfides of arsenic, antimony and stannic tin are easily soluble in colorless ammonium sulfide. Stannous sulfide is not thus soluble and its presence is avoided.

4. Copper sulfide is only slightly soluble in colorless ammonium sulfide and in this method the quantity dissolved is inconsiderable, though a

little polysulfide may be formed because of separation of sulfur or action of ferric ion.

The process is carried out as follows: To a given quantity of the solution (say 45 cc.) add one-ninth of its volume (5 cc.) of concentrated hydrochloric acid and a few drops (0.5 cc.) of nitric acid. The laboratory hydrochloric acid is usually about 10 $N$ and the solution is thus made about normal. If much acid is already present allowance must be made for it. Transfer the mixture to an Erlenmeyer flask and boil it half away under the hood. The residual liquid is about 2 $N$ in HCl since but little acid is lost on boiling at this concentration. The nitric acid has oxidized stannous to stannic ion, has prevented separation of metals by reduction, and has probably raised the valence of a portion of the arsenic. Pass a rapid stream of hydrogen sulfide through the hot liquid, heating again to boiling once or twice and shaking vigorously. The precipitation of arsenic begins quite promptly and proceeds rapidly. When no more precipitate forms (5 to 10 minutes), add enough water (80 cc.) to make the volume a little more than twice the original (100 cc.). This reduces the HCl concentration below 0.5 $N$. Continue to pass the gas until the liquid is cold and until no more precipitate falls (10 to 15 minutes), filter and wash. For a somewhat similar process, except as to the boiling, see Noyes and Bray.[1]

Transfer the precipitate to a beaker, cover it with concentrated ammonium hydroxide, pass a rapid stream of hydrogen sulfide for one or two minutes, warm gently, shake well, filter and wash. The sulfides of arsenic, antimony and tin dissolve very promptly, carrying with them generally only a trace of copper. Filtrate and residue are treated in the usual way.

The advantages of this method are as follows: (1) A definit acid concentration is secured; (2) the precipitation of arsenic is certain, prompt and complete in a few minutes; (3) after dilution the other metals are thrown down with similar ease and certainty; (4) the use of yellow ammonium sulfide is avoided; (5) the time is much shortened, 30 to 45 minutes being sufficient for the whole process.

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## THE QUANTITATIVE DETERMINATION OF PERCHLORATES.[2]

BY ARTHUR B. LAMB AND JOHN W. MARDEN.

In the course of a study of certain cobaltammines it became necessary

[1] THIS JOURNAL, 29, 167; Proc. 21.

[2] Read before the Section of Inorganic and Physical Chemistry of the American Chemical Society at the Washington Meeting, December 28, 1911.