

IX. TREATMENT OF THE FILTRATE FROM PRECIPITATES FORMED BY HYDROGEN SULPHIDE IN PRESENCE OF ACID.

The filtrate from the sulphides precipitated by hydrogen sulphide in the acidified solution from which copper had been removed as cuprous sulphocyanide was neutralized carefully with ammonium hydroxide (no precipitation taking place in the presence of tartaric acid) and treated with hydrogen sulphide. The precipitate should contain the sulphides of iron, nickel, cobalt, and possibly copper in traces, if compounds of these elements are present. The filtrate was rejected. The precipitate was treated on the filter with dilute hydrochloric acid and the residue washed. The filtrate was reserved for the estimation of iron. The insoluble precipitate was ignited with the filter and the residue dissolved in aqua regia, the solution evaporated to dryness, the residue taken up in hydrochloric acid, the solution treated with hydrogen sulphide, and any precipitate filtered off, ignited, and weighed as copper oxide.

The filtrate from the copper sulphide, or the solution in which hydrogen sulphide gave no precipitate in presence of hydrochloric acid, was carefully made alkaline with ammonium hydroxide, and hydrogen sulphide was passed in. The precipitate, filtered off and ignited, was weighed as nickelous oxide and cobaltic oxide. Any considerable residue obtained at this point should be treated for the separation of nickel and cobalt by the double nitrite process; in the case of the very small residues actually obtained in this work, the simple test for cobalt with the borax bead was all that could be made.

The filtrate reserved after treatment of the sulphides of iron, nickel, cobalt, and copper with hydrochloric acid, was boiled, treated with bromine water, and made alkaline with ammonium hydroxide. The precipitate was filtered off, washed, ignited, and weighed as ferric oxide.

X. SPECIAL TESTS FOR TIN.

In the special tests for tin alone, it was usual to attack the material with nitric acid, desiccate, take up into nitric acid and water, boil, heat with ammonium hydroxide, and then with nitric acid, filter off the insoluble material, ignite and weigh. By this treatment all the tin originally present was gathered in the residue. The residue was fused with a mixture of sodium carbonate and sulphur, the melt extracted with water. The extract was acidified, and the precipitate thus obtained collected, washed with ammonium nitrate, ignited and weighed. In the ignition any arsenic present would be volatilized, so that the residue should consist only of stannic oxide and possibly antimony oxide. If the residue was considerable, the separation of antimony and tin was effected by the method previously described in section IV. If the residue was inconsiderable and no indication of antimony had been noted in the color of the sulphide, the qualitative confirmatory test for tin was made at once.

In the main, the procedure outlined above was followed closely; but great care was taken in the scrutiny of final products, which were often subjected to special analysis to make sure that the separations had been as nearly complete as possible. The analyses were made, almost entirely, by Dr. Martha Austin Phelps and Dr. Charles Paxson Flora.