

COMMUNICATIONS FROM THE LABORATORY OF THE SCHOOL OF
MINES, LEOBEN.

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1. DETECTION AND ESTIMATION OF SMALL QUANTITIES OF CHROMIC OXIDE.—The qualitative separation of very small quantities of chromic oxide from much ferric oxide alumina and manganous oxide presents many difficulties, and often fails, when the usual systematic course is pursued, viz., fusion of the oxides with potassic carbonate and potassic nitrate. The following process works much better:—The solution which may contain, besides the metals already mentioned, the salts of the alkaline earths, is poured into a boiling solution of sodic carbonate containing some potassic permanganate. After boiling for a few minutes longer, the excess of permanganate is reduced by addition of a few drops of alcohol and filtered. Ferric oxide, manganic peroxide, alumina, and the carbonates of the alkaline earths are precipitated, whilst the filtrate contains the chromium as sodic chromate. If present in not too small amount, the liquid is coloured more or less yellow. If colourless, the liquid is concentrated to a small bulk, and acidified with dilute sulphuric acid. A piece of starch which has been moistened with a solution of potassic iodide is now introduced, and if chromium is present, will turn violet. Or the liquid may be shaken with potassic iodide and carbon disulphide, when the latter will turn violet.

These reactions are more delicate than the test with hydrogen peroxide. For the quantitative estimation we proceed in similar manner. The solution is slowly added to the hot permanganate, and boiled for ten minutes. The filtrate is acidified with hydrochloric acid, boiled with alcohol, and finally precipitated in a porcelain dish with ammoniac sulphide. In a mixture of 50 c.c. solution of ferric chloride (1 c.c. containing ·00714 ferric oxide), 50 c.c. solution of alum (1 c.c. containing ·0068 alumina), and 1 c.c. chromic chloride, containing ·0018 chromic oxide, the latter was successfully quantitatively estimated.

2. VALUATION OF RICH COPPER ORES.—The usual plan is to first precipitate the copper by metallic zinc, then to redissolve precipitate after washing in nitric acid, and, after addition of ammonia, to titrate with potassic cyanide. The preliminary precipitation with zinc is advisable, as copper is but imperfectly separated from iron by ammonia. The following process will, however, be found more expeditious:—

The finely powdered sample is intimately mixed in a porcelain crucible, with double its volume of zinc dust. The lid is put on, and the whole ignited for ten minutes. The ore is by this treatment completely desulphurised, the zinc taking up all the sulphur. After cooling, the mass is boiled in a beaker, with dilute sulphuric acid (1·4), which dissolves the excess of zinc and the zinc sulphide, also the iron. The copper is left insoluble, but contains, besides small quantities of cupric sulphide, any metals precipitable by zinc which may have been present in the ore, also the gangue. After thorough washing with boiling water, the mass is, as usual, treated with nitric acid, filtered, diluted up to a definite bulk, and aliquot parts are titrated with potassic cyanide.

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3. CHANCEL'S PROCESS FOR THE SEPARATION OF ALUMINA FROM IRON.—The direct separation of alumina from iron is best effected by boiling the neutralised solution with sodic hyposulphite (Chancel). In practice, the process is but seldom employed, most chemists preferring to weigh the mixed oxides, and then to volumetrically estimate the iron. We have, however, made some experiments to test the accuracy of the method.

A very weak solution of alum, 30 c.c. of which yielded ·0768 alumina, was mixed with 25 c.c. of a neutral solution of ferric chloride (= ·1785 ferric oxide) diluted with water up to 400 c.c., and boiled down to about 100 c.c., after addition of excess of sodic hyposulphite. The precipitated alumina was then at once filtered off. The following results were obtained :—

Number of c.c. of solution of alum taken.			Containing Alumina	Alumina Found		
30	·0764	·0730
—	—	·0730
40	·1018	·0984
—	—	·0956
50	·1273	·1220

As will be noticed, the precipitation of the alumina was in no case complete. The filtrate and washings, of the two last experiments were boiled down to half the bulk, and further precipitates of alumina were obtained, amounting to ·0072 and ·0068 respectively. This, however, makes the results too high, which is explained by the difficulty of completely expelling the co-precipitated sulphur. The presence of sulphur may be detected by the yellowish colour the mass assumes on ignition. Alumina, free from sulphur, does not turn yellow. Chancel's process is, however, trustworthy, when the following rules are adhered to: strong dilution, and boiling down to at least half the volume, again boiling the filtrate, and collecting any further precipitate of alumina, and igniting precipitate before the blowpipe until yellow colour disappears.

4. ON THE USE OF HYDRIC PEROXIDE.—Hydric peroxide is frequently used in analytical work, for instance, to completely precipitate manganese from its ammoniacal solution. We have conducted some experiments, to see whether other metals would interfere with the accuracy of the process.

Fifty c.c. of a solution of manganous sulphate, containing ·1335 manganous manganic oxide, were mixed with a weak solution of zinc sulphate, mixed with excess of ammonia, and boiled with hydric peroxide. The precipitate was rapidly washed, and ignited before the blowpipe. Two other experiments were made, using nickelous sulphate, and calcic chloride. The following results were obtained:—The first precipitate weighed ·1540 gramme, the second ·1475 gramme, the third ·1376 gramme. As these results were too high, the precipitates were redissolved in hydrochloric acid, and again precipitated with ammonia and hydric peroxide, when the following weights were obtained: ·1334, ·1467, ·1341 gramme. From these experiments we may conclude the following:—Manganese may be completely separated from zinc by repeated treatment (about four times), with ammonia and hydric peroxide. Two precipitations will free it from nickel, but from lime the separation is incomplete.

Precipitated cobaltous oxide is quickly oxidised to the sesquioxide by ammoniacal hydric peroxide, but nickelous oxide does not alter. Ammoniacal solution of nickel does

not change on boiling with the reagent, but ammoniacal solution of cobalt colours deep brownish-red. On acidifying with hydrochloric acid and boiling, nearly all the cobalt precipitates as the roseo compound. This reaction is very useful for preparing pure cobalt compounds from cobalt ores containing nickel.
