SOME NOTES ON THE ESTIMATION OF CHROMIUM AS CHROMIC OXIDE.

BY

WM. M. BLANCHARD AND PAULINE NORRIS.

Schirm (Chem.-Ztg., *33*, 877) states that chromium can be determined gravimetrically by precipitation from a chromic salt by ammonium nitrite solution and igniting the precipitate with the filter in a Rose crucible.

Schoeller and Schrauth (Chem.-Ztg., *33*, 1237) state that accurate gravimetric determinations of chromium may be made by precipitation from a boiling, dilute solution of the chromic salt by the addition of one cc. of aniline and boiling for five minutes. The precipitate is filtered, washed, and ignited.

Hanus and Lukas (8th. Intern. Congr. Appl. Chem., I, 209-12) state that chromic hydroxide can be precipitated quantitatively from neutral or alkaline solutions containing chromates by hydrazine hydrate and some of its derivatives.

Rothaug (Z. anorg. Chem., *84*, 165-89) states that a long series of estimations made by precipitating chromium hydroxide by means of ammonium hydroxide, ammonium sulphide, the iodide-iodate method, and by hydroxylamine, gave results running high from 0.17% to 1.60%, the error being due to the formation of chromic chromate during the ignition.

These statements are interesting compared with the assertions of Fresenius (Quant. Chem. Analysis, 6th. Ger. Edition, Cohn's Eng. Translation, page 281) that when chromium is precipitated in glass vessels, the results are high owing to contamination with silica; if the precipitation is carried out in porcelain vessels, the results are much more satisfactory, and if platinum vessels are used, the results are quite accurate.

With a view to verifying the statements of Fresenius or those of Rothaug, the following experiments were made: The purest obtainable potassium dichromate was carefully recrystallized and the chromium estimated by evaporating to dryness solutions to which were added ethyl alcohol and hydrochloric acid, redissolving, and precipitating the chromium from boiling dilute solutions with ammonium hydroxide. After washing, the precipitate was ignited with the filter, heated over a Meeker burner and afterwards with blast lamp, cooled and weighed. 0.1500 gram of the dichromate was taken each time; this quantity should give 0.0775 gram chromic oxide.

Five determinations in beakers of the best American glass gave 0.0791, 0.0797, 0.0791, 0.0794, 0.0795.

Five determinations with precipitation in a large porcelain casserole gave 0.0794, 0.0790, 0.0796, 0.0798, 0.0794.

Five determinations with precipitation in a large platinum dish gave 0.0798, 0.0797, 0.0791, 0.0792, 0.0794.

Three determinations with glass beakers, the precipitate being dissolved and reprecipitate, gave 0.0796, 0.0790, 0.0789.

One determination with precipitate ignited in a Rose crucible gave 0.0799.

Two blank determinations gave no precipitates.

From these results it would appear that the estimation of chromium by precipitation as chromium hydroxide and ignition in the ordinary way is not reliable and that the error is independent of the composition of the vessel in which the precipitation is effected.