ART. XXXVII.—*On the Estimation of Cadmium as the Oxide*; by PHILIP E. BROWNING and LOUIS C. JONES.

[Contributions from the Kent Chemical Laboratory of Yale University—LVII.]

IN an article entitled "The Estimation of Cadmium" * Max Muspratt discusses critically some of the methods in use for the determination of that element. Muspratt finds that the method which involves the precipitation as carbonate, ignition and weighing as oxide gives low results, and he accounts for these results by the well known tendency of cadmium to reduction, especially in the presence of organic matter. To avoid this reduction he dissolved the precipitated carbonate in nitric acid, evaporated to dryness on a water bath, and gently ignited the nitrate to the condition of the oxide. This treatment also gave low results, although the oxide obtained from the ignition of the nitrate was found to contain traces of sulphate from the solution of the cadmium sulphate used. A second method of treatment was to filter and dry the precipitated carbonate and remove as much of it as possible to a weighed porcelain crucible, ignite and weigh by itself. This oxide was found to be free from traces of sulphate.†

The remainder of the cadmium oxide adhering to the paper was dissolved in pure nitric acid and the solution and rinsings evaporated to dryness and ignited in a weighed crucible and weighed. Here also low results were obtained which, after the extended process of manipulation, would scarcely seem surprising. If, however, Muspratt adds, the oxide obtained from the ignition of the carbonate be taken as Cd_2O the results are satisfactory.

A third modification gives more satisfactory results. The method of treatment is the same as the last mentioned, except that the oxide obtained from the ignition of the carbonate is ignited in a stream of oxygen until no further increase in weight results.

In a former paper from this laboratory‡ one of us made use of the carbonate method for the determination of cadmium after having separated that metal from copper. The filtration was made on asbestos in a Gooch crucible and the results were most satisfactory. The object of the work to be described is to show that when the carbonate is filtered upon an asbestos felt previously ignited the dangers of reduction are obviated and the carbonate process is both simplified and placed among good analytical methods. The solution used for the work was one of cadmium sulphate, and the standard was determined by evaporating measured and weighed portions to dryness in the

* Jour. Sci. Chem. Industry, vol. xiii, 211.

† Muspratt's theory is that in the ignition of the carbonate the sulphate is dissociated, while the ignition of the nitrate does not effect this result.

‡ Browning, this Journal, vol. xlv, 280.

presence of a few drops of sulphuric acid, igniting at low redness, and weighing as the anhydrous sulphate. The average of several closely agreeing results was taken as the standard. Measured and weighed portions of this solution were diluted to about 300 cm³ with hot water and a solution of potassium carbonate, 10 per cent, added drop by drop with constant stirring until no further precipitate was obtained. The precipitate was then boiled for about fifteen minutes, when it became granular and settled quickly. It was then filtered upon asbestos, washed thoroughly, dried, and ignited at red heat until a constant weight was obtained. In several instances the weighed oxide was treated with a drop of nitric acid, again ignited and weighed, but in no case was there a perceptible change in weight. In the following table the results are tabulated.

No.	CdO taken. gram.	CdO found. gram.	Error. gram.
(1)	0·1140	0·1143	0·0003 +
(2)	0·1142	0·1137	0·0005 —
(3)	0·1141	0·1148	0·0007 +
(4)	0·1141	0·1148	0·0007 +
(5)	0·1142	0·1146	0·0004 +
(6)	0·1143	0·1147	0·0004 +
(7)	0·1143	0·1144	0·0001 +
(8)	0·1139	0·1146	0·0007 +
(9)	0·1270	0·1272	0·0002 +
(10)	0·1279	0·1283	0·0004 +
(11)	0·1272	0·1281	0·0009 +
(12)	0·1278	0·1281	0·0003 +
(13)	0·2556	0·2561	0·0005 +
(14)	0·2550	0·2547	0·0003 —
(15)	0·1272	0·1279	0·0007 +
(16)	0·1281	0·1288	0·0007 +
(17)	0·1274	0·1278	0·0004 +
(18)	0·1284	0·1290	0·0006 +
(19)	0·1271	0·1277	0·0006 +
(20)	0·1278	0·1285	0·0007 +
(21)	0·2555	0·2555	0·0000 ±

As will be noticed, the results show a plus error which might naturally be due to a slight inclusion of the alkali carbonate. To test the truth of this hypothesis, a portion of the oxide which gave a plus error of 0·0007 gram. was dissolved and tested for potassium by the perchloric acid method* and an amount of that element was found equal to 0·0006 gram. of the carbonate. Another portion of the oxide which showed no error was similarly treated and only 0·0002 gram. on the carbonate was found. The results show, as we think, that the carbonate method can be successfully applied to the quantitative estimation of cadmium without recourse to the tedious process of manipulation recommended by Muspratt.

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* Kreider, this Journal, vol. xlix, 443.