

The time factor is satisfactory on considering that three elements are determined in something over 15 hours, with a minimum of attention, and very simple apparatus.

It appears from the experience here given that the method of constant potential separation can be simply applied in commercial analyses when the voltage margin is not too narrow.

By arranging to stir the solution and employing larger surfaces for deposition the time factor can be reduced, but this complicates the apparatus.

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NOTES.

On the Gravimetric Determination of Zinc as Pyrophosphate.—In the original method, Tamm¹ used disodium phosphate as precipitant, adding a large excess to the slightly acid solution containing zinc and excess of ammonium chloride. Later, Dakin² used ammonium phosphate, and washed the precipitate with a 1 per cent. solution of it, finally washing with 95 per cent. alcohol.

In order to determine whether ammonium phosphate is superior to sodium phosphate as precipitant, and whether water could be used for washing as well as ammonium phosphate solution, a few experiments were made as follows:

To the hot solution containing the zinc as sulphate in a Jena beaker, was added ten times as much ammonium phosphate in 10 per cent. solution as there was zinc present (total vol. 150 cc.). The precipitate was allowed to settle on the steam bath until small in bulk (required about 30 mins.), and filtered on a porcelain gooch crucible, washed five times with cold water, and ignited to constant weight over a Bunsen burner.

Grams zinc taken: 0.2240, 0.1841. Found: 0.2242, 0.2242, 0.2242, 0.1839, 0.1844, 0.1841.

The above procedure was repeated, but to the zinc solution before precipitating were added 10 cc. of 30 per cent. acetic acid, 20 cc. of nitric acid (1 cc. = 0.47 g. HNO₃), and the solution made neutral to litmus with ammonium hydroxide. If an excess of ammonia is present, it either keeps some zinc in solution, or if evaporated off, causes the precipitate to adhere firmly to the glass.

Grams zinc taken: 0.1841. Found: 0.1847, 0.1836, 0.1836, 0.1836, 0.1839.

The first procedure was next varied by washing with 1 per cent. ammonium phosphate and 95 per cent. alcohol.

Grams zinc taken: 0.2240. Found: 0.2250, 0.2245, 0.2242.

¹ *Chem. News*, 24, 148.

² *Z. anal. Chem.* 39, 273.

Also sodium phosphate (5 g.) in 10 per cent. solution and 2 g. ammonium chloride were substituted for the ammonium phosphate in the first procedure above.

Grams zinc taken: 0.1841. Found: 0.1844, 0.1840, 0.1851. Grams zinc taken: 0.2240. Found: 0.2242, 0.2250, 0.2242.

The precipitate in this case did not settle as readily as where ammonium phosphate was used. The large excess of sodium phosphate is necessary, as two grams gave very low results.

It appears that the results are not improved by washing with 1 per cent. ammonium phosphate and alcohol, and that ammonium phosphate is more satisfactory than sodium phosphate. The presence of an excess of ammonium salts does not interfere. RALPH W. LANGLEY.

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Constant-level Reservoir.—The arrangement shown in the figure has been used by us for some time for purposes similar to the one described by Mr. Fitzgerald.¹ We have found it especially useful during the filtration of solutions of bitumen in carbon tetrachloride containing large amounts of carbenes, when it is essential that the filter be not allowed to become dry.

As seen in the sketch, the Gooch filter is connected with the Erlenmeyer filtering flask in the usual manner, and a globe-shaped, stoppered separatory funnel is adjusted till the lower end of the funnel stem is about a half inch from the felt of the filter. It is then filled with the solution, and the stopper tightly inserted. When the stopcock is opened the filtration commences, and continues until all the solution has left the funnel stem. The stopcock is then closed, and the stopper removed, when the inside of the funnel can be washed with clean solvent. The stopper is inserted and the stopcock opened, allowing the operation to continue, washing the precipitated carbenes.

The parts of this apparatus are all glass, and therefore are not attacked by the vapors from the carbon tetrachloride.

If it is desired to make the operation continuous to the end without the necessity of watching for the emptying of the reservoir, another funnel containing fresh solvent may have its stem inserted in the tubular

¹ THIS JOURNAL, 31, 839.

