

A METHOD FOR THE SIMULTANEOUS DETERMINATION OF ARSENIC AND SULPHUR IN ORGANO-ARSENICALS

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During the course of the synthesis of aryl sulphonyl esters of hydroxy phenyl arsonic acids,¹ a new and general method for simultaneous estimation of sulphur and arsenic in sulphur containing organo-arsenicals, by oxidation with alkaline permanganate, has been evolved. The analytical results obtained by the new method compare very well with those obtained by the application of previously known methods for arsenic and sulphur. Uptil now, separate estimations have to be made for sulphur and arsenic in a sulphur containing organo-arsenical compound necessitating larger quantities of sample and also involving longer period. When the reagents and apparatus are ready, a simultaneous determination of arsenic and sulphur by the new method can be completed within 8 hours. As long as necessary precautionary measures are taken, excess of potassium permanganate (even upto 10 g.) does not affect the accuracy of the result of arsenic estimation. The end point is usually very sharp.

EXPERIMENTAL

The sample (25–35 mg.) of material was weighed accurately into a 300 c.c. pyrex conical flask provided with a vertical condenser, and oxidised by refluxing for 2 hours with 5 g. of potassium permanganate and 10 c.c. of 30% solution of potassium hydroxide. It was cooled to room temperature and 5 c.c. of hydrochloric acid (1 : 1) were added drop by drop under shaking. After the addition of hydrochloric acid was over the flask was slowly heated on the wire-gauze and a fresh quantity of concentrated hydrochloric acid was added drop by drop through the top of the condenser until the solution became pale yellow or almost colourless. The sides of the flask were then washed with 50 c.c. of distilled water.

The mixture was heated to boiling and a 10% solution of barium chloride was added in a fine stream through a funnel with a capillary stem or from a burette, at the rate of 10 c.c. in 2 to 10 minutes' time. The reagent was added in slight excess (10 c.c. of 10% barium chloride—with 2 mols.

of water of crystallization—will precipitate 0.13 g. of sulphur). The beaker was placed on a steambath and the precipitate was allowed to settle for an hour. The solution was filtered through a fine grade of filter-paper or through a Gooch crucible. Since a portion of the precipitate frequently passed through the filter-paper, the filtrate was passed through the same filter for a second time. The precipitate was washed 10 times with hot water, then dried and ignited gently over a Bunsen burner for half-an-hour. The barium sulphate was cooled in a desiccator and then weighed. The percentage of sulphur was calculated from the weight of barium sulphate so obtained.

Concentrated hydrochloric acid (15 c.c.) and glacial acetic acid (15 c.c.) were added to the filtrate. After the addition of 3 g. of potassium iodide, the flask was stoppered and kept in a dark place for 15 minutes. The liberated iodine was finally titrated against standard thiosulphate. From the amount of the thiosulphate consumed, the percentage of arsenic was calculated.

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REFERENCE

1. Roy, *et al.* .. *Current Science*, 1948, 17, 89.