

insoluble in dilute sulphuric acid, so that this reagent is not suitable for the estimation of ferrous salts in the presence of phenolic compounds. Sodium vanadate has, however, been found to give excellent results as shown in the following table.

Composition of solution		Amount of ferrous iron found by ceric sulphate titration	Amount of ferrous iron found by sodium vanadate titration
Amount of ferrous iron taken	Amount of phenolic compound		
millimols.		millimols.	millimols.
0.7191	nil	0.7200	0.7201
0.7191	0.5008 phenol	0.8230	0.7201
0.7191	0.5090 paracresol	0.8245	0.7201
0.7101	0.4634 ortho cresol	0.8212	0.7086
0.7101	0.4666 meta cresol	0.8194	0.7086
0.7139	0.4562 resorcinol	0.8945	0.7139

Sodium vanadate will thus be found to have some advantages even over ceric sulphate. Detailed results will be published elsewhere.

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1. Gopala Rao and Ramanjaneyulu, *Curr. Sci.*, 1949, 18, 72. 2. Gopala Rao and Brahmaji Rao, *Ibid.*, 1949, 18, 3. Viswanadham and Gopala Rao, *Ibid.*, 1943, 12, 327. 4. Lyons and Appleyard, *Quart. J. Pharm. Pharmacol.*, 1937, 10, 348. 5. Ferrey, *Ibid.*, 1937, 10, 351.

THORIUM PERIODATE AND ITS USE IN THORIUM ESTIMATIONS

RAY CHAUDHURY¹ reports that thorium nitrate in dilute nitric acid solution yields on prolonged heating over a water-bath with excess of sodium paraperiodate a gelatinous precipitate of the composition $\text{ThHIO}_6 \cdot 5\text{H}_2\text{O}$. This substance is further reported to be stable upto 600°C . The strength of the acid solution has not been specifically mentioned, but our experiments with 2N acid using potassium periodate in place of the sodium salt have not been successful. We have therefore undertaken a more systematic investigation and our experiments show that precipitation occurs only at 1N or lower acid concentration. Even under these conditions significant quantities of thorium remain in solution though a ten-

fold excess of periodate is used. If, however, hot neutral potassium periodate is added in slight excess to a hot neutral solution of thorium nitrate, there results immediately a white gelatinous precipitate, which may conveniently be filtered and washed through a sintered glass crucible. This, on drying at 105 to 110°C . for 3 to 4 hours, changes to a semi-transparent mass of constant weight, and composition as reported by Ray Chaudhury.¹ The time of drying is not critical, but the temperature should not rise much above 120° (even at 180° iodine vapours are observed). When the above procedure is followed, the precipitation of thorium is quantitative as can be gathered from the following typical results.

Wt. in g. of $\text{ThHIO}_6 \cdot 5\text{H}_2\text{O}$ obtd.	Wt. in g. of thorium calculated	Wt. in g. of thorium taken
0.2471	0.1051	0.1055
0.2479	0.1054	0.1055
0.2368	0.1007	0.1008
0.2357	0.1002	0.1008

Thorium periodate precipitate is soluble in dilute mineral acids and can accurately be estimated volumetrically in the following way:—the washed precipitate is dissolved in dilute hydrochloric acid and a slight excess of potassium iodide is added. The iodine liberated is titrated against standard thiosulphate. One atom of thorium corresponds to one periodate group or eight equivalents of thiosulphate. The following example will illustrate the accuracy of the volumetric estimation 0.1055g. of thorium was taken. The estimated values were 0.1047, 0.1048, 0.1050 and 0.1052.

An interesting property of the periodate precipitate is that while it ordinarily retains five molecules of water of hydration, on prolonged desiccation (3 weeks) in vacuum over caustic potash, a part of this water is lost and its final composition corresponds to $\text{ThHIO}_6 \cdot 4\text{H}_2\text{O}$.

Work on the use of periodates in the separation of thorium from cerium earths is in progress.

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1. Ray Chaudhury, *J.I.C.S.*, 1941, 18, 335.