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PROCEDURE FOR THE PRECIPITATION OF THORIUM IODATE FROM HOMOGENEOUS SOLUTION

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A complete report of this work will be made available as an AEC-NYO report in the near future.

The solution may contain 2 to 150 mg. of thorium oxide. Thus far, the maximum limits of contaminants studied have been 1200 mg. of mixed trivalent rare earth oxides (lanthanum, cerium, praseodymium, neodymium and yttrium) and 350 mg. of phosphorus pentoxide (as phosphoric acid). In a single precipitation the total weight of rare earth and phosphoric oxides contaminating the iodate precipitate from 80 mg. of thorium oxide is of the order of 5 mg. A double precipitation will quantitatively separate thorium.

Concentrate the solution containing thorium to about 50 ml.. Add 36 ml. of concentrated nitric acid. Since the total acidity of the final solution must not be greater than 3 N (a value of 2.5 N is desirable), the original solution should not contain more than the equivalent of 8 ml. of 6N mineral acid. The latter may be perchloric, sulfuric, or nitric acid but not hydrochloric acid.

Add 14 grams of sodium metaperiodate previously dissolved in 100 ml. of water with any insoluble residue being filtered off. Finally, dilute the solution to 230 ml. and then cool to room temperature. Employ mechanical stirring throughout the entire period of precipitation. Add 2 ml. of distilled β-hydroxyethyl acetate; precipitation will begin in about 10 minutes when 50 to 100 mg. of thorium are present.

Thirty minutes after precipitation begins, add an additional 6 ml. of β-hydroxyethyl acetate. One hour after this addition, add a solution containing 2 grams of potassium iodate, 9 ml. of nitric acid, and 50 ml. of water. Thirty minutes following this, stop the stirring and allow the precipitate to settle.

Decant the supernatant liquid thru a Whatman No. 40 filter paper. Wash the precipitate by decantation ten times with a solution containing 8 grams of potassium iodate, 100 ml. of concentrated nitric acid, and 900 ml. of water. Dissolve the small amount of precipitate from the filter with a 10% hydrochloric acid solution saturated with sulfur dioxide and catch the filtrate in the original precipitation beaker containing the bulk of the thorium iodate.

Evaporate the solution to dryness on a low temperature hot plate and bake at 110°C for 1 hour. Dissolve the salts in 50 ml. of 10% hydrochloric acid and filter off any dehydrated silica. Wash well with 1:20 HCl solution.

If the quantity of rare earths and phosphate is large, thorium iodate should be reprecipitated.

Since thorium cannot be weighed as the iodate, the determination must be terminated either by conversion of the thorium to a suitable weighing form or by a volumetric method (cf. Gordon, Louis and Stine, C.R., ANAL. CHEM., 25, 192 (1953)).