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Chemistry

AEC Research and Development Report

# "VERSENE" TITRATION OF THORIUM AND ALUMINUM

by
W. R. Cornman
Analytical Chemistry Division

August 1956



E. I. du Pont de Nemours & Co.
Explosives Department - Atomic Energy Division
Technical Division - Savannah River Laboratory

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ABSTRACT

A method was developed for the determination of thorium and aluminum in nitric acid solutions containing small amounts of fluoride ion. Titration with "Versene" (disodium ethylenediaminetetraacetic acid) was employed with Alizarin Red S as an internal indicator.

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## RESULTS

### Analysis of Samples

To establish the method, preliminary analyses were carried out on ten-microliter aliquots of a synthetic solution containing no fluoride. The results are given in the following table:

RESULTS OF ELEVEN ANALYSES OF A SYNTHETIC SOLUTION

| | |
|---|---|
| Thorium present, micromoles | 15.36 |
| Thorium found, micromoles | 15.37 |
| Standard Deviation, per cent | 0.5 |
| Aluminum present, micromoles | 6.30 |
| Aluminum found, micromoles | 6.10 |
| Standard Deviation, per cent | 1.3 |

### Effect of Fluoride

The effect of fluoride on the thorium and aluminum determinations was studied. Samples of solution, which were 0.14M in fluoride, were analyzed for thorium and aluminum. The results are given in the following table:

EFFECT OF FLUORIDE ON DETERMINATION OF ALUMINUM AND THORIUM

| | |
|---|---|
| Number of determinations | 13 |
| Thorium present, micromoles | 14.44 |
| Thorium found, micromoles | 14.41 |
| Standard Deviation, per cent | 0.5 |
| Aluminum present, micromoles | 6.30 |
| Aluminum found, micromoles | 5.98 |
| Standard Deviation, per cent | 2.6 |

The presence of 0.14M fluoride decreased the recovery of aluminum by about three per cent.

A ten-microliter sample of a solution of irradiated thorium was analyzed with "Versene". This analysis yielded a molarity of 1.24 for thorium and a molarity of 0.22 for aluminum. A colorimetric determination yielded a thorium molarity of 1.25. The expected aluminum concentration calculated from the amount of aluminum added to the thorium solution was 0.2M. No independent determination for aluminum was made.

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## APPENDIX

### EQUIPMENT AND CHEMICALS

One-tenth-milliliter and one-milliliter Gilmont ultramicroburets equipped with ruby plungers were used for all titrations.

A stock solution of 0.6M aluminum nitrate was prepared from Merck reagent grade aluminum nitrate and was standardized by ignition to the oxide with a precision of 0.0004M.

A stock solution of 0.1M thorium nitrate was prepared from Baker reagent grade thorium nitrate and was standardized by ignition to the oxide with a precision of 0.0008M.

A 0.1M solution of "Versene" was prepared from Bersworth analytical reagent grade disodium "Versenate". The solution was standardized to 0.0003M against ferric iron according to the method of Milner and Woodhead[4].

A one-tenth weight per cent aqueous solution of Alizarin Red S was used as an indicator solution.

### ANALYTICAL PROCEDURE

#### Determination of Indicator Blank

1. Add three drops of Alizarin Red S indicator to 30 milliliters of HCl solution of pH 3.5.

2. Titrate with standard thorium solution to the pink color of the thorium-indicator complex. Record this volume in microliters as the indicator blank.

#### Analysis of Samples

1. Add a 10-microliter sample to 30 milliliters of HCl solution (pH 3.5) contained in the titration vessel.

2. Add three drops of Alizarin Red S indicator.

3. Titrate with standard 0.1M "Versene" solution to the appearance of the yellow color of the indicator. (High aluminum-to-thorium ratios interfere with the thorium end point. The maximum permissible mole ratio of aluminum to thorium is about two.) Record this volume in microliters as the amount of "Versene" needed for thorium.

4. Add standard "Versene" in about 20 per cent excess and allow to stand ten minutes. ("Versene" and aluminum react slowly at the pH employed. A ten-minute waiting period is necessary to ensure completeness of reaction.)

5. Titrate with standard 0.1M thorium solution to the appearance of the pink color of the thorium indicator complex. Record this volume in microliters as the amount of thorium needed for back titration.

CALCULATIONS

$$\text{Thorium Molarity} = \frac{M_1 V_1}{V_S}$$

$M_1$ = "Versene" molarity

$V_1$ = "Versene" required for thorium titration, microliters (Step 3)

$V_S$ = Sample size, microliters

$$\text{Aluminum Molarity} = \frac{M_1 V_2 - M_2(V_3 - V_4)}{V_S}$$

$M_1$ = "Versene" molarity

$M_2$ = Standard thorium molarity

$V_S$ = Sample size, microliters

$V_2$ = Excess "Versene" added, microliters (Step 4)

$V_3$ = Volume thorium required in back titration, microliters (Step 5)

$V_4$ = Indicator blank, microliters

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