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ANALYSIS OF IRRADIATED THORIUM OXIDE
(SERIES I - DEVELOPMENT TEST IF-588-I)

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(SERIES I - DEVELOPMENT TEST IP-588-D)

AUTHORS:

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ANALYSIS OF IRRADIATED THORIUM OXIDE
(SERIES I - DEVELOPMENT TEST IP-588-D)

I. INTRODUCTION

An analytical chemistry program was undertaken to determine total U-233 and the U-232 to U-233 isotopic ratio in samples of thorium oxide irradiated in Development Test IP-588-D (HW-77619). That test was carried out to measure the effect of Th-230 content on the formation of U-232 in U-233 during thorium irradiation. In June 1963, irradiations were initiated on four samples of thorium oxide of known Th-230 content. Three samples were of low Th-230 content, ranging in value from 0.2 to 1.2 ppm, whereas the fourth sample had a Th-230 content of 80 ppm. Exposures of approximately one, two and four months were scheduled. This report summarizes the analytical chemistry results obtained on the samples exposed for one month and briefly describes the analytical procedures which were used.

II. SUMMARY

The analytical results of main interest are shown below. The detailed results are given on page 114. The total U-233 is shown at 200 days after sample discharge. Thorium-230 values are those measured prior to irradiation.

<u>Sample No.</u>	<u>Th-230, ppm</u>	<u>^{U-233} ppm U-233</u>	<u>U-233 g/T Th</u>
1	0.2	2.8	1810
2	0.4	2.7	1860
3	1.2	3.0	1850
4	80	20	1870

III. EXPERIMENTAL AND DISCUSSION

A. Sample History

Samples of the four thorium oxides of known Th-230 content were encapsulated in quartz ampoules. Duplicate ampoules were prepared for each of the four oxides and for each of the three irradiations proposed. Accurately weighed amounts of thorium oxide were placed in the quartz ampoules. Sample weights were approximately one gram.

The first set of samples was irradiated in a PCCF tube of DR Reactor for a 27.7 day exposure. Irradiation was initiated on June 13 and the samples were discharged on July 17. On September 9, analytical separations were initiated. Uranium-233 was separated from its parent Pa-233, thorium, and other constituents. At time of separation, the U-233 had reached approximately 84 percent of its maximum value.

B. Analytical Procedure

Initial analytical operations were carried out in cell #5 of the Shielded Analytical Laboratory. The quartz ampoules were opened by crushing mechanically in a closed lusteroid test tube and transferred quantitatively to a dissolving flask. The thorium was dissolved in hot 12N HCl-0.04 N HF. Maintaining the HF concentration below that which would cause

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HW-78959

the insoluble ThF_4 to be formed gave complete dissolution in 10-15 minutes.

The dissolver solution was loaded directly onto a 1 cm x 12 cm column of Dowex 1 x 8, 100-200 mesh anion exchange resin in the chloride form. Under these conditions, the uranium is quantitatively adsorbed and the thorium passes through the column. The dissolution flask was rinsed with two - 12 ml portions of 10 N HCl and the rinses added to the column. Six to seven column volumes (30-35 ml) of 9 N HCl-1 N HF were passed through the column to remove Pa-233. Five column volumes of 10 N HCl were used to remove fluoride ion and the uranium was eluted with four column volumes of 0.1 N HCl. The uranium product was removed from the shielded cell, converted to the nitrate form by evaporating to dryness in HNO_3 , and brought to a calibrated 10 ml volume in 2 N HNO_3 .

The U-232 to U-233 isotopic ratio was determined by alpha energy analysis using a silicon diode detector and a 400 channel pulse height analyzer. Sample mounts free from residue were prepared by electrodeposition on platinum disks from aliquots of the 10 ml nitrate solutions which had been further purified by ether extraction. Electrodeposition was carried out from a nitrate solution. Electrolysis was started at pH 1.5 and continued until the solution was basic. Deposition takes place at the cathode which is basic with respect to solution. Dissolution of the plated hydroxide is prevented by a gradual increase in the solution pH brought on by the reduction of nitrate at the cathode.

The uranium product solutions were assayed by three independent methods - alpha counting, isotopic dilution, and fluorimetrically. Alpha counting was done with an Alpha Simpson Proportional Counter (ASP) of the Chemical Analysis Unit. Isotopic measurements were done under the direction of R. C. Lagergren, Nuclear Physics Research, on a thermal ionization mass spectrometer. Fluorimetric measurements were done by the Radiological Analysis Unit.

C. Reliability

The estimated accuracy of the total U-233 values reported is ± 5 percent. Uranium recoveries for the separation procedure were determined by running two dummy samples, which had been spiked with known amounts of U-233, through the entire procedure in the shielded cell. Recoveries were 92.3 percent and 94.6 percent. The average of these two values was used to adjust the actual sample values to 100 percent recovery. The other main source of uncertainty in total U-233 values is in calculating the fraction of U-233 present from Pa-233 decay at time of separation. Calculations based on the exposure record, time after discharge, and burn-up are estimated to be accurate to ± 4 percent. Actual assays of the product solutions by alpha counting and isotopic dilution are estimated to be accurate to ± 3 percent or better. A comparison of results obtained by those two methods is shown in the detailed results. The total values reported are the average of those two methods.

As further verification, total uranium values were also obtained by fluorimetric measurement. Although inherently less precise than values obtained by isotopic dilution and alpha counting, the fluorimetric values were in good agreement.

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HW-78959

The values reported for the U-232 to U-233 isotopic ratio at 200 days after sample discharge include a 16 percent adjustment for further decay of Pa-233 to pure U-233. These values are estimated to be accurate to ± 5 percent. However, the actual measurement of the ratio of U-232 to U-233 by alpha energy analysis was better than ± 5 percent. The sample disks were completely free of other alpha emitters and the two energy peaks were completely resolved. Standard sources of alpha emitters of known energy were used to calibrate and verify the alpha energy measurements. The high quality of such measurements is due to the electrodeposition techniques used and to the availability of the latest in refined radiochemical instrumentation.

DETAILED ANALYTICAL RESULTS

<u>Sample Identification</u>	<u>Techmanix</u>	<u>Lindsay</u>	<u>Vitro</u>	<u>Dow</u>
Th-230 (ppm) (1)				
U-233 $\mu\text{g/g}$ Th (2)	0.17 ± 0.05	0.37 ± 0.08	1.24 ± 0.14	82.8 ± 6.3
	(3) 1698	1744	1714	1702
	(4) 1657	1704	1703	1753
Rounded Avg.	<u>1680</u>	<u>1720</u>	<u>1710</u>	<u>1730</u>
U-232 ppm U-233 (5)	3.4	3.3	3.5	24
U-232 ppm U-233 (6)	2.8	2.7	3.0	20
U-233 g/ton Th (7)	1810	1860	1850	1870

- (1) Preirradiation values.
- (2) Separated approximately 55 days after end of irradiation.
- (3) Results from alpha counting
- (4) Result from mass spectrometry - isotopic dilution
- (5) 55 days after end of irradiation
- (6) Projected to 200 plus days after irradiation
- (7) Projected to 200 plus days after irradiation

Note: U-233 present 55 days after end of irradiation was approximately 84 percent of the total at 200 plus days.

The estimated accuracy of all analytical results except the Th-230 values is ± 5 percent.

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