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SUBJECT: Examination of Irradiated ThO₂ Slugs
for the U233/Th Ratio

TO: J. R. Johnson

FROM: R. P. Wischow

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DATE	FEB 14 1957 <i>AL</i>
For The Atomic Energy Commission	
<i>H. F. Canale</i>	
Chief, Declassification Branch	

Two thorium oxide slugs were received from the Solid State Division for evaluation of the U^{233} /thorium ratio as a function of the slug radius.⁽¹⁾ The slug history and physical measurements have been given in CF-55-4-162.

EXPERIMENTAL

Dissolution of the slugs was done in a glass dissolver fitted with reflux condensers, an off-gas outlet, and a heat source. To obtain data on the ease of dissolution, dissolving time, etc., a non-irradiated ThO_2 slug was dissolved (see Table 1). From these data, it was determined that about one-third of the irradiated ThO_2 slug would be dissolved by a seven hour reflux period in the dissolvent. Also, the dissolution time was not found to be prohibitive with respect to fluoride attack upon the glass dissolver.

The irradiated ThO_2 slugs, as received, were broken in several pieces, each about $1 - 1\frac{1}{2}$ inches long. The larger diameter slug fragments were chosen for this study to utilize the greater radius as well as the higher irradiation dosage, i.e., 2.435×10^{20} nvt.⁽²⁾ The slugs were a deep blue-black color, confirming the previous observation that ThO_2 , present in metal slugs as an impurity, changed color upon neutron irradiation.

A complete slug fragment of slug #2⁽²⁾ was dissolved (see Table 2). Fractional dissolution of another fragment of the same slug was done with the data reported in Table 3. No difficulty was found in dissolving these slugs except the long refluxing period necessary to affect final slug dissolution. The presence of impurities, presumably silica or the bonding matrix, was observed in the final solutions; these impurities tend to decrease the dissolution rate.

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Table 1

Dissolution of Non-Irradiated ThO₂ Slug

Dissolvent: 13 M HNO₃; 0.04 M HF; 100% stoichiometric excess
of HNO₃ added

Date	Refluxing Time (hr)	Weight of ⁽¹⁾ ThO ₂ Slug (g)	ThO ₂ Dissolved (g)	Thorium ⁽²⁾ (mg/ml)	HNO ₃ ⁽²⁾ (M) ³
3-15-55	---	360	---	---	13.00
3-15-55	6	254	106	74.9	11.57
3-16-55	7	166	88	123.8	10.64
3-17-55	7	98	68	171.0	9.89
3-18-55	7	54	44	202.4	9.40
3-21-55	7	25	29	225.6	9.05
3-22-55	7	4	25	228.5	8.89
3-24-55	7	---	4	237.5	8.86

1. Obtained by weighing the ThO₂ slug after each dissolution period.
2. Obtained by analysis.

Note: ThO₂ slug does not dissolve uniformly; pieces broke off and deep pits appeared on the slug surface.

Table 2

Dissolution of Irradiated ThO₂ Slug Fragment

Date	March 16/18, 1955
Dissolvent	13 M HNO ₃ , 0.04 M HF; 100% stoichiometric excess HNO ₃ added
Slug weight	212.7 g
Theoretical thorium weight	187.2 g
Dissolution time, reflux	35 hr
Final solution volume	1060 ml
Analysis:	
Thorium	180.6 mg/ml
U ²³³	2.35 x 10 ⁶ α c/m/ml at 52% geometry
U ²³³ /thorium	(1182 g U ²³³ /ton thorium)
Material balance:	
Total g thorium (theoretical)	187.2
Total g thorium (analyzed)	191.4
Material balance	102%

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Table 3

Determination of U^{233} Content
as a Function of ThO_2 Slug Radius

Conditions: Dissolvent 13 M HNO_3 ; 0.04 M HF
After each refluxing period, the solution was removed from slug; the dissolver was flushed with water; the slug removed, weighed and measured. Measurements were taken with vernier micrometer

Sample ⁽¹⁾	Weight (g)	Diameter (in.)	Length (in.)	Refluxing Time (hr)	Thorium (mg/ml)	$U^{233} \alpha$ (c/m/ml) ⁽⁴⁾	$\frac{g U^{233}}{Ton Thorium}$
1	169.0	1.125	1.188	7	119.5	1.29×10^6	981
2	104.5	0.938	1.00	7	91.5	1.13×10^6	1123
3	54.5	0.75 ⁽²⁾	--- ⁽³⁾	21	104.5	1.25×10^6	1087

1. Sample number refers to portion of slug dissolved.
2. Cone shaped; measurement only approximate.
3. Pitted surface at end of slug; no measurement taken.
4. Counted at 52% geometry.

CONCLUSIONS

Since the slugs were broken into fragments, only tentative conclusions can be drawn from these data. Thorium oxide slugs do not dissolve uniformly; therefore, diameter and length measurements during dissolution are not completely valid. Deep pits were observed on the slug surfaces with somewhat greater and irregular penetration of the slug at the broken ends than at the machined end.

Table 2 shows that slug #2 ⁽²⁾ contains 1182 g U^{233} /ton thorium; Table

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3 indicates an average g/T level of 1064 g U²³³/ton thorium for another fragment of the same slug. The fragment used to obtain the data in Table 2 had one machined end, whereas in Table 3, the slug fragment had two broken ends. This latter fragment was chosen for the U²³³/Th ratio evaluation because it was assumed that any discrepancy caused by irregular dissolution would be minimized in that the broken ends should dissolve at the same rate. More optimum conditions would have been to use an unbroken slug whereby more valid data could be obtained.

The solutions from these experiments are available for additional testing and/or analyses if this becomes necessary.

R. P. Wischow

RPW/jr

REFERENCES

1. ORNL-CF-55-2-104, Johnson, J. R., "Thorium Oxide Slugs."
2. ORNL-CF-55-4-162, Carroll, R. M., "Thorium Oxide Slugs."