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DISSOLUTION OF THORIUM OXIDE

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W. E. PROUT
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Chemical Separations Processes
For Plutonium and Uranium
(TID-4500)

DISSOLUTION OF THORIUM OXIDE

by

M. Lee Hyder
William E. Prout
Edwin R. Russell

Approved by
L. H. Meyer, Research Manager
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July 1966

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CONTRACT AT(07-2)-1 WITH THE
UNITED STATES ATOMIC ENERGY COMMISSION

ABSTRACT

The factors that influence the rate of dissolution of aluminum-canned irradiated ThO_2 were quantitatively assessed. Conditions were established that provide dissolution rates acceptable for a plant-scale process.

CONTENTS

	<u>Page</u>
LIST OF TABLES AND FIGURES	iv
INTRODUCTION	1
SUMMARY	1
EXPERIMENTAL PROCEDURES	2
RESULTS	3
Effect of Dissolvent Composition	3
Effect of Particle Size	5
Effect of Agitation	6
Tests on Different Types of Thoria	8
Effect of Reactor Irradiation	9
Chemical Removal of Aluminum Cans	10
BIBLIOGRAPHY	11

LIST OF TABLES AND FIGURES

<u>Table</u>		<u>Page</u>
I	Particle Size Distribution of Sol-Gel Thoria	3
II	Effect of HNO ₃ and HF Concentrations on the Dissolution of ThO ₂	4
III	Dissolution of Sol-Gel Thoria	8
<u>Figure</u>		
1	Effect of HNO ₃ Concentration on Dissolution Rate	4
2	Effect of Particle Size on the Dissolution of Sol-Gel ThO ₂	5
3	Rate of Dissolution of Sol-Gel ThO ₂ under Various Conditions of Agitation	6
4	Effect of Agitation on Dissolution of Sol-Gel ThO ₂ with Side Heating	7
5	Variation of Dissolution Rate with Type of ThO ₂	8
6	Dissolution of Irradiated and Unirradiated ThO ₂	9

DISSOLUTION OF THORIUM OXIDE

INTRODUCTION

Thorium dioxide (ThO_2) is a useful reactor target material for the production of ^{233}U . Desirable properties of ThO_2 are its radiation stability, high density, and high melting temperature. In addition, reactor-grade ThO_2 can be produced at relatively low cost by several processes. A disadvantage to the use of ThO_2 , however, is its low dissolving rate in conventional acidic dissolvents.

The purpose of this investigation was to determine suitable dissolving conditions for processing irradiated ThO_2 targets in existing Savannah River Plant equipment. The effects of reactor irradiation, particle size, and agitation on dissolving rate required evaluation. In addition, a choice had to be made between chemical removal of the aluminum cladding before dissolving the ThO_2 , and codissolution of the cans and ThO_2 . Rates of dissolution were determined for reactor-grade ThO_2 from several different commercial sources.

The tests performed in this investigation were made on a small scale in the laboratory. Subsequent dissolving tests on a semiworks scale are to be reported in a separate document. (1)

SUMMARY

Laboratory studies were carried out to develop a method for the dissolution of irradiated ThO_2 targets as the first step in separation of the ^{233}U product by a solvent extraction process. The most satisfactory dissolvent composition for plant use was determined to be 13M HNO_3 -0.025M HF -0.1M $\text{Al}(\text{NO}_3)_3$. The dissolution rate of thoria was affected by the particle size, the method of thoria preparation, and the degree of agitation, but not by reactor irradiation. Moderate agitation of the thoria-dissolvent mixture, such as that obtained by heating the dissolver vessel at the bottom, produced dissolving rates as high as those obtained with vigorous mechanical stirring.

Codissolution of the aluminum can with the ThO_2 was more satisfactory for plant operation than separate removal of the can by chemical decladding.

EXPERIMENTAL PROCEDURES

In general the laboratory-scale tests simulated plant conditions as closely as possible. Dissolutions were made in stainless steel vessels, from one to four liters in volume, that were fitted with water-cooled reflux condensers to minimize loss of dissolvent by evaporation. The vessels were heated to the boiling temperature of the dissolvent by electric hot plates or heating mantles, or with electric heating tapes wrapped around the vessels. Heating the vessels from the bottom with hot plates gave reproducible and rapid dissolving rates because of the considerable agitation produced throughout the solution by boiling; this method was used for most studies. Heating the vessels from the side with heating tape more closely resembled conditions in conventional plant dissolvers; but less agitation was produced and dissolving was consequently slower.

ThO₂ used in these studies included: (a) material prepared by the Sol-Gel process⁽²⁾ by Mallinckrodt Chemical Works (MCW) and by Nuclear Fuels Services (NFS), (b) material prepared by arc-fusion at Oak Ridge National Laboratory (ORNL), and (c) pellets prepared by an extrusion and sintering process by the General Electric Company (GE). MCW samples were used in most studies. Reactor-grade aluminum tubing was used for decladding studies; chemicals were reagent grade.

In the dissolving tests dry ThO₂ and cold dissolvent solution were added to the dissolver, in the ratio of one mole of ThO₂ per liter of dissolvent. Usually the thoria test batch was dissolved almost completely within four to seven hours, during which time four to six one-milliliter analytical samples were removed. After complete dissolution, the final concentration of thorium found by analysis was 0.98 - 1.05M, depending upon evaporation loss. Loss of dissolvent by evaporation was generally less than 5%.

During a series of tests all conditions were reproduced except the variable being investigated. Thorium analyses were made by the thoron colorimetric method.

Dissolution tests of irradiated ThO₂ were made in shielded cells with samples irradiated to 1200 and to 2000 g ²³³U/ton. The experimental equipment was similar to that used in the other studies.

RESULTS

EFFECT OF DISSOLVENT COMPOSITION

Previous work at ORNL, ^(3,4) with ThO₂-UO₂ targets containing 4% uranium, showed that the most effective dissolvent was concentrated nitric acid (~13M) containing HF. In the present studies solutions of this type only were tested for the dissolution of ThO₂.

The ThO₂ used in these tests was MCW Sol-Gel type, with the particle size distribution shown in Table I.

TABLE I

Particle Size Distribution of Sol-Gel Thoria
(MCW Lot T-2)

<u>U.S. Std. Screen</u>	<u>Weight %</u>
+16	17.8
-16 +20	7.7
-20 +40	14.4
-40 +50	3.1
-50 +60	3.1
-60 +80	6.2
-80 +100	8.3
-100	39.4

Samples of the test oxide were dissolved to determine the optimum concentrations of HNO₃ and fluoride, and the optimum ratio of oxide to dissolvent. In each test 26.4 g of oxide was dissolved with 80, 100, or 120 ml of dissolvent. The samples were heated on an electric hot plate.

The results in Table II show the effects of concentrations of nitric acid and fluoride, and of dissolvent volume on the rate of dissolution. In general, the dissolution is characterized by a relatively rapid rate in which 80 to 90% of the oxide is dissolved in about 6 hours, followed by a significantly reduced rate for dissolution of the remainder, as illustrated in Figure 1. This behavior is attributed to rapid dissolution of the relatively large fraction of fine particles, and much slower attack on the coarser particles as the acid strength is reduced and the fluoride catalyst is complexed more completely by Th⁴⁺.

TABLE II

Effect of HNO₃ and HF Concentrations
on the Dissolution of ThO₂

26.4 g samples of ThO₂ from Lot T-2
Dissolvent boiled under reflux

Dissolvent (a)			ThO ₂ Dissolved, %			Th(NO ₃) ₄ , M		
HNO ₃ , M	HF, M	Volume, ml	4 hr	6 hr	8 hr	4 hr	6 hr	8 hr
13	0.1	80	80.3	89.0	93.5	1.00	1.11	1.17
13	0.1	100	85.0	93.5	96.0	0.85	0.94	0.96
13	0.1	120	92.0	97.3	98.8	0.77	0.81	0.82
13	0.05	120	86.5	93.8	96.4	0.72	0.78	0.80
16	0.05	120	84.3	92.5	95.5	0.70	0.77	0.80
10	0.05	120	73.0	82.1	86.0	0.61	0.68	0.72
10	0.05	80	64.3	77.0	82.5	0.80	0.96	1.03
10	0.1	80	70.8	84.0	89.0	0.88	1.05	1.11
10	0.1	100	75.8	87.2	92.1	0.76	0.87	0.92
10	0.1	120	82.8	91.3	95.2	0.69	0.76	0.79

(a) Each dissolvent also contains 0.1M Al(NO₃)₃.

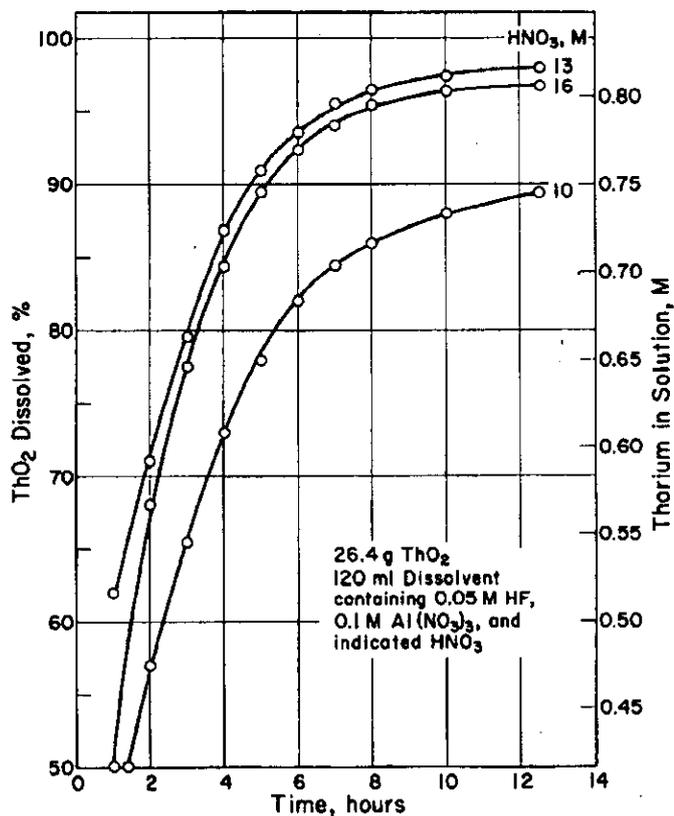


FIG. 1 EFFECT OF HNO₃ CONCENTRATION ON DISSOLUTION RATE

The laboratory tests showed that the optimum concentration of nitric acid is ~13M. Concentrations of fluoride >0.05M increase the dissolving rate, but as dissolution proceeds ThF₄ will precipitate for concentrations of F⁻ >0.1M. In subsequent work an HF concentration of 0.025M provided adequate dissolution rates and no ThF₄ precipitation. 0.1M Al(NO₃)₃ was added to the dissolvent to complex the fluoride during the early stages of dissolution and to limit corrosion of the dissolver vessel; no adverse effect of aluminum ion on the dissolving rate was observed.

EFFECT OF PARTICLE SIZE

As expected, the rate of dissolution of ThO₂ was highly dependent on particle size; this is illustrated in Figure 2. In

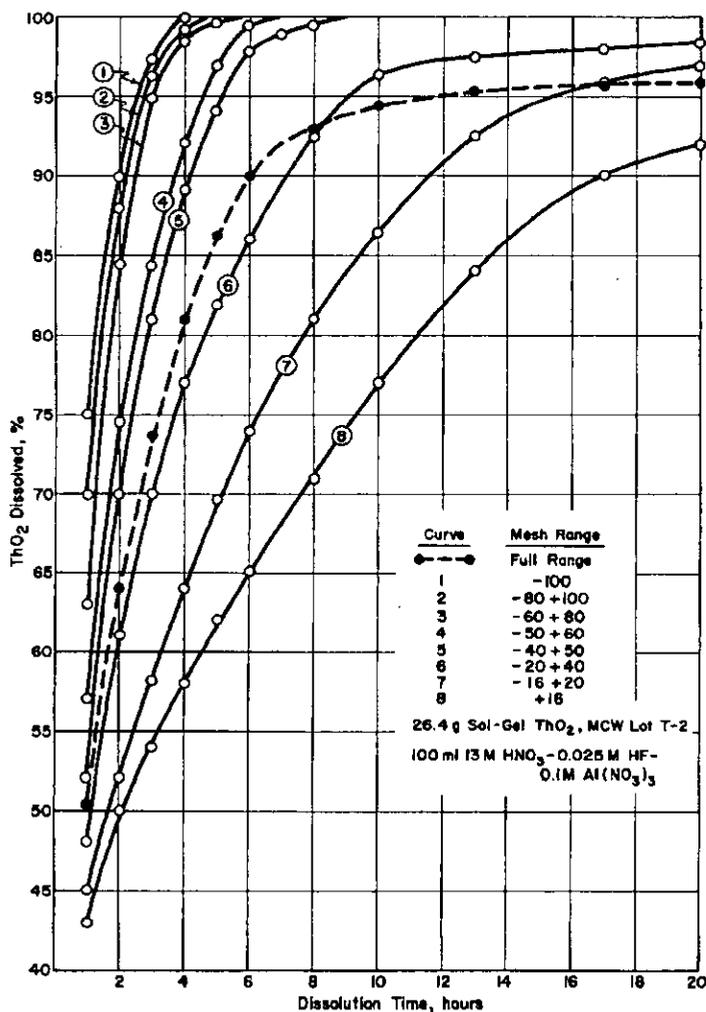
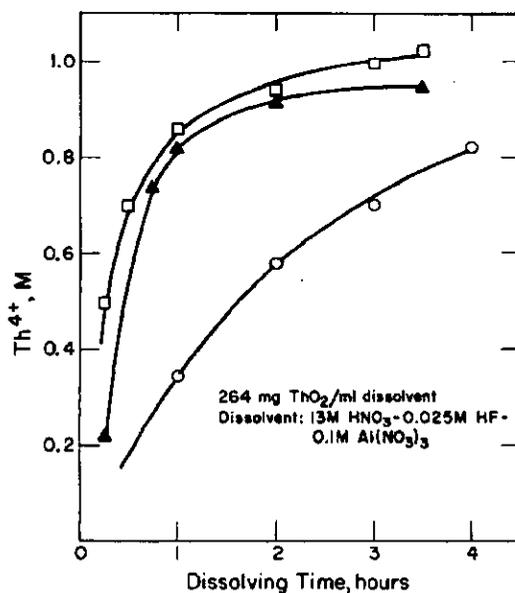


FIG. 2 EFFECT OF PARTICLE SIZE ON THE DISSOLUTION OF SOL-GEL ThO₂

these experiments, MCW Sol-Gel thoria from a single batch was sieved into fractions using standard screens, and samples from each fraction were dissolved under good agitation, with other conditions reproduced as closely as possible. The data show that very fine subdivision is desirable for rapid dissolution, but a lower limit to particle size is imposed by handling and packing considerations in target fabrication. The correlations in Figure 2 emphasize the necessity for fixing particle size range in any test series designed to compare dissolving rates for different types of thoria.

EFFECT OF AGITATION

The results of early tests suggested that agitation adequate for dissolving the ThO_2 at experimentally reproducible rates could be obtained in the laboratory through boiling the heterogeneous mixtures by heating the bottom of the dissolving vessel. Later tests showed that violent mechanical stirring during boiling by this type of heating caused no increase in rate; typical data are shown in Figure 3. Normal boiling induced by bottom-heating does not greatly disturb the bed of thoria itself; it therefore appears that the relatively rapid dissolution rates are due to efficient circulation of the dissolvent through the thoria, rather than by suspension of the thoria in the liquid.



- Vessel heated from bottom, vigorous mechanical stirring
- ▲ Vessel heated from bottom, no stirring
- Vessel heated from side, no stirring

FIG. 3 RATE OF DISSOLUTION OF SOL-GEL ThO_2 UNDER VARIOUS CONDITIONS OF AGITATION

In conventional plant dissolvers, heat is supplied by steam coils arranged in vertical rows inside the dissolvers near the walls; these conditions were simulated in the laboratory by heating the outer walls of the dissolving vessel with electrical tape. Dissolving rates obtained in these experiments, shown in Figure 3, were much lower than those obtained by heating from the bottom; boiling took place near the walls of the vessel, and apparently circulation of the dissolvent to the thoria in the center of the vessel was inefficient. Under the condition of side-heating, a small amount of additional agitation produced a considerable increase in dissolving rate; for example, the addition of small amounts of aluminum metal to the thoria greatly accelerated dissolution of the ThO_2 (Figure 4). Codissolution of aluminum produced no significant effect in well-agitated solutions, thereby indicating that the effect of adding aluminum derived solely from the increased agitation produced by gases evolved from the slowly dissolving metal.

The beneficial effect of boiling the dissolvent by heating from the bottom of the vessel was substantiated by large-scale tests in a dissolver with a diameter of 6 feet. These tests, to be described in a separate report,⁽¹⁾ showed that improvement by a factor of three or more could be obtained by adding steam coils across the bottom of the dissolver.

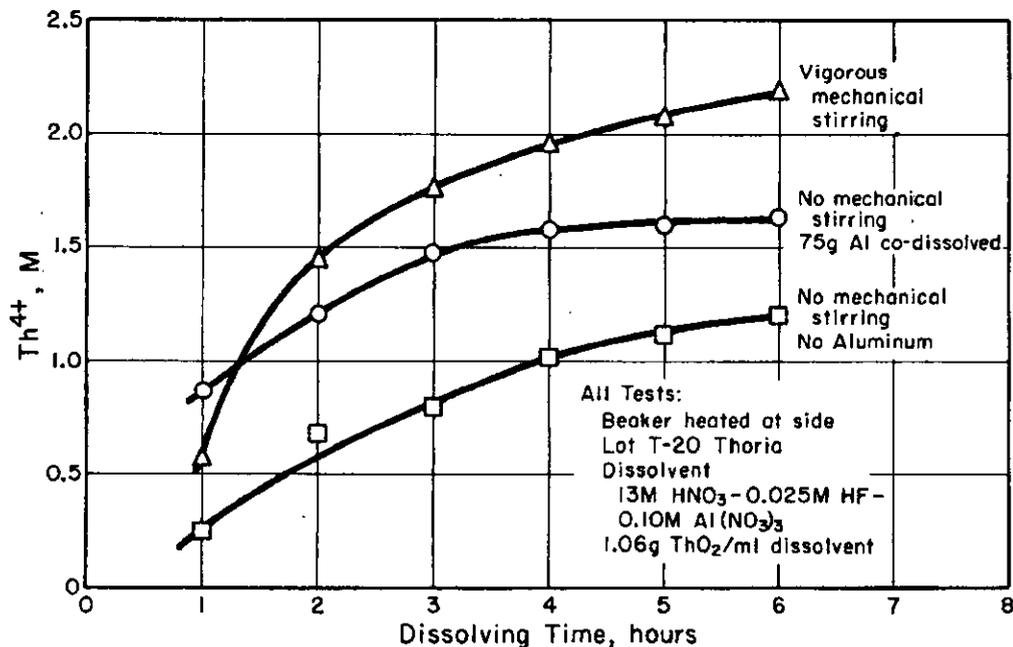


FIG. 4 EFFECT OF AGITATION ON DISSOLUTION OF SOL-GEL ThO_2 WITH SIDE HEATING

TESTS ON DIFFERENT TYPES OF THORIA

Dissolution rates of thoria of various types and from various sources were compared. Laboratory dissolving tests were run on the following materials: MCW Sol-Gel thoria (several different lots); NFS Sol-Gel thoria; ORNL arc-fused thoria; and GE extruded, sintered thoria. For all materials, a size fraction of -16 +20 mesh (U. S. Standard Sieve) was used. Typical results are shown in Figure 5, with additional data in Table III.

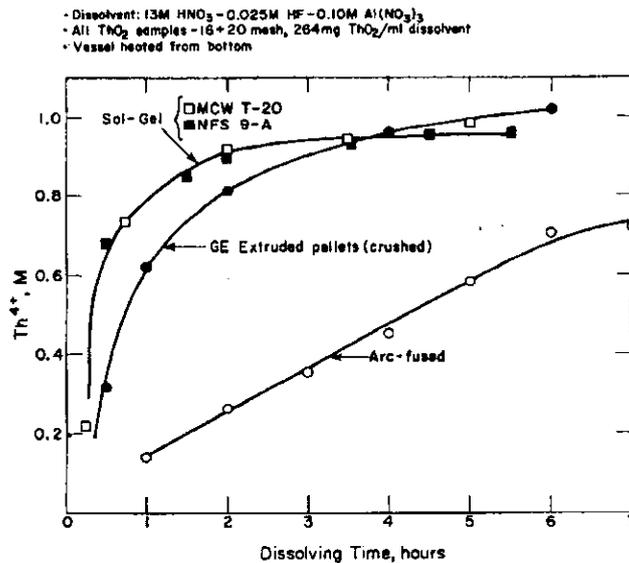


FIG. 5 VARIATION OF DISSOLUTION RATE WITH TYPE OF ThO₂

TABLE III

Dissolution of Sol-Gel Thoria

Time required for 95% dissolution of Sol-Gel ThO₂ (-16 +20 mesh). Dissolving conditions as described in Figure 5.

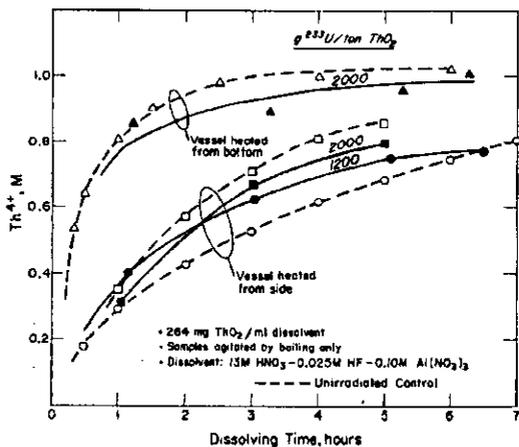
<u>Batch</u>	<u>Time, hours</u>
MCW	
T-2	15
T-18	2.3
T-20	3.5
T-21	2.1
T-28	8.0
T-34	>10
T-38	>10
NFS	
9-A	4.0
62	1.5

Sol-Gel thorium dissolved much more readily than arc-fused material, as illustrated in Figure 5. Crushed GE pellets dissolved slightly slower than Sol-Gel thorium.

In general the widest variations observed between different lots of Sol-Gel thorium were due to differences in particle size distribution; but a number of batches of Sol-Gel material were found to have intrinsically poor dissolving properties, as shown in Table III. These batches (MWC T-28, T-34, and T-38) appear to have contained about 25% of poorly dissolving thorium, and about 75% of more rapidly dissolving material resembling lots T-18, T-20, and T-21. Batch T-2 also dissolved relatively slowly. The variations in manufacturing conditions were not known well enough to allow correlation with dissolution rates, but tests of samples of thorium prepared in the laboratory by the Sol-Gel process showed that an important parameter that affected dissolution is the sintering temperature. The NFS thorium samples that were tested dissolved at rates comparable to the better lots of MCW material (T-18, T-20, and T-21).

EFFECT OF REACTOR IRRADIATION

Two dissolving tests were carried out with reactor-irradiated thorium to determine whether irradiation has a significant effect on dissolving properties. The thorium used in these studies was irradiated to either 1200 or 2000 g $^{233}\text{U}/\text{ton}$. Unirradiated samples from the same lots of oxide were dissolved in control tests; the thorium irradiated to 1200 g $^{233}\text{U}/\text{ton}$ was dissolved in the same vessel as that used for the control. This vessel consisted of a four-liter stainless steel beaker, fitted with a Pyrex water-cooled condenser on top and wrapped with heating tape to simulate conventional (side-heating) dissolver conditions. Control dissolvings on the 2000 g/ton ThO_2 were made in 1-liter laboratory beakers.



The results are shown in Figure 6. The differences in dissolving rates between irradiated and unirradiated samples were not significant. The data indicate that dissolving rates of oxide irradiated to <2000 g $^{233}\text{U}/\text{ton}$ can be determined by tests with unirradiated material.

FIG. 6 DISSOLUTION OF IRRADIATED AND UNIRRADIATED ThO_2

CHEMICAL REMOVAL OF ALUMINUM CANS

Three methods were tested for chemically dissolving the aluminum cans used to contain thoria for irradiation. The methods were (1) mercury-catalyzed nitric acid decanning; (2) decanning with NaOH-NaNO₃ solution; and (3) codissolution with the thoria. The first two are standard procedures developed for other types of fuel elements and were intended to allow the rejection of the aluminum (in solution) from undissolved ThO₂. The third produces a solution containing both the aluminum and the thorium; this method had not been previously investigated.

The procedures were simulated in laboratory tests by adding pieces of reactor-grade aluminum tubing to the ThO₂ in the dissolver vessel at an Al/Th ratio of 1.4. In the case of the mercury-catalyzed acid and the alkaline dissolutions all of the aluminum was dissolved, then the decanning solution was removed and analyzed, after which the thoria dissolvent was added. In the test of codissolution the dissolvent was added to the vessel containing Al and Th and the concentration of each in solution was determined from periodic samplings. An evaluation of the results showed that

- (1) Neither the acid decanning solution [6M HNO₃, 10⁻³M Hg(NO₃)₂] nor the alkaline decanning solution (4.3M NaOH, 3M NaNO₃) dissolved thorium oxide appreciably. However, in one test in which irradiated ThO₂ was boiled for 24 hours in the acid decanning solution, about 20% of the ²³³U was leached from the ThO₂, indicating that acid decanning is probably unsuitable when the decanning solution is to be discarded.
- (2) The alkaline decanning solution was fairly viscous and tended to entrain fine ThO₂ particles. The subsequent dissolution of ThO₂ was retarded after the exposure to the alkaline decanning solution. This result may have been the result of compaction of the finely divided thoria or of aggregation of the grains by aluminates.
- (3) Codissolution of the aluminum and the ThO₂ appeared to be the most satisfactory method for plant use. In laboratory tests, aluminum metal present with the ThO₂ dissolved in the standard dissolvent [13M HNO₃-0.025M HF-0.1M Al(NO₃)₃] before the thoria dissolution was complete. In the case of poor agitation, the dissolution of the aluminum metal accelerated the ThO₂ dissolution; the rate of dissolution of the aluminum was controlled by addition of Hg(NO₃)₂.

The codissolution method was developed further in semiworks tests.⁽¹⁾ In that work, a cyclic decanning and dissolving procedure was developed in which thoria is dissolved in fresh dissolvent to ~1M $\text{Th}(\text{NO}_3)_4$, then Al-canned thoria slugs are added to the dissolver together with a small amount of $\text{Hg}(\text{NO}_3)_2$ to promote dissolution of the aluminum. The resulting solution is removed, leaving a "heel" of ThO_2 for the next cycle. In this procedure the dissolution of the aluminum decreases the nitric acid concentration to ~1M, which is desirable for subsequent processing.

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TIS File

Mr. R. G. Erdley, Chief
Patent Branch
Savannah River Operations Office
U. S. Atomic Energy Commission
Post Office Box A
Aiken, South Carolina

August 9, 1966

Dear Mr. Erdley:

REQUEST FOR PATENT REVIEW

Please review for patent matter:

DP-1044, Dissolution of Thorium Oxide, by M. L. Hyder, W. E. Prout,
and E. R. Russell

If any technical clarification is needed please call J. E. Beach whose document review is attached.

Please telephone your comments to the TIS Office (Ext. 3402) and notify me by signing and returning to TIS the original of this letter. A copy is provided for your file.

If you decide to pursue a patent on any development covered, I shall be happy to supply additional information required such as appropriate references and the names of persons responsible for the development.

The above item is approved
for release.

Very truly yours,

for J. A. Koch 8/17/66
R. G. Erdley, Chief Date
Patent Branch
SROO, USAEC

C. W. J. Wende, Director
Technical Division

By: J. E. Beach
J. E. Beach

EXTERNAL RELEASE OF TECHNICAL INFORMATION

Description of Material

No. DP-1044

Date: 8/9/66

Title: Dissolution of Thorium Oxide

Author: M.L. Hyder, W.E. Prout, E.R. Russell

Type of Material

Classified DP Report

Classified Paper

Unclassified DP Report

Unclassified Paper

Letter

Technical Content

Approved by /s/ L. H. Meyer

Date: 3/10/66

Classification

Approved by

S. W. O'Rear /ASA
S. W. O'Rear

Date: 8/9/66

Authority:

Topic 151.1.3 SROO Classification Guide

SROO Classification Notices 37 and 39

Category if DP Report

Approved by

S. W. O'Rear /ASA
S. W. O'Rear

Date: 8/9/66

Final Du Pont Release

Approved by

[Signature]
Coordinating Organization Director

Date: 8/27/66

Released by

R. G. Erdley:

8/27/66