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POST-IRRADIATION EXAMINATION OF THORIA-URANIA FUEL RODS

Quarterly Progress
Report No. 2

September - November 1967

RESEARCH & DEVELOPMENT
DIVISION

NUCLEAR DEVELOPMENT
CENTER

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THORIA-URANIA FUEL RODS

— Quarterly Progress Report No. 2 —

September - November 1967

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1. INTRODUCTION.

The objectives of this program are to perform isotopic and metallurgical analyses on thorium-uranium fuel; stainless steel cladding, and a Zircaloy fuel can taken from Core A of the Consolidated Edison Indian Point Reactor Number 1. The isotopic content of the several actinide and fission product elements in the fuel will be determined. The fuel and cladding will be examined metallographically. Specimens of the stainless steel cladding and Zircaloy can will be tensile tested under several conditions.

This second in a series of quarterly progress reports on this program covers the period September 1 - November 30, 1967. The reactor core and fuel rod selection are described in the first quarterly report.¹

2. SUMMARY

The fuel rods for this program have been removed from the fuel elements at Battelle Memorial Institute and transported to the Babcock & Wilcox Nuclear Development Center. Most of the rods have been gamma scanned.

A sequential chemical separation procedure for uranium, plutonium, americium-curium, cesium, neodymium, promethium, and samarium has been refined and is considered ready for use on thorium-uranium fuels. This procedure uses four columns; two extraction chromatography columns and two inorganic ion exchange columns, for the separation of the elements.

Two separation procedures for neptunium were evaluated and a solvent extraction procedure was selected for this program.

3. ISOTOPIC ANALYSIS OF THORIA-URANIA FUEL

The chemical separation procedure for uranium, plutonium, americium-curium, neodymium, promethium, samarium, and cesium reported in the previous quarterly report¹ has been modified slightly to eliminate an evaporation step and to improve ruthenium decontamination. This was achieved by replacing the silver paraperiodate column with a mixed lead dioxide-lead iodate column.

A procedure for separating neptunium from thoria-urania fuel has been devised. Neptunium(VI) is extracted as the tetraheptylammonium ion association complex from hydrochloric acid into methyl isobutyl ketone. After scrubbing the organic phase with acid deficient aluminum chloride for additional decontamination, neptunium is back extracted into a ferrous chloride-hydroxylamine strip solution.

3.1. Sequential Four-Column Procedure

The chemical separation procedure for uranium, plutonium, americium-curium, neodymium, promethium, samarium, and cesium, as previously reported¹ employs two extraction chromatography columns and two inorganic exchange columns in the sequential separation of these elements. The first extraction column, di-(2-ethylhexyl)phosphoric acid (D2EHP) on a Chromosorb support, retains thorium, uranium, plutonium, and zirconium from 1 N nitric acid. The effluent from this column subsequently passes through potassium cobaltoferrocyanide (KCFC) which retains cesium, and silver paraperiodate which retains cerium and ruthenium. The acidity of the effluent is reduced tenfold by evaporation prior to passage through the final column to ensure the retention of cerium-free lanthanides and trivalent actinides. Americium and curium are eluted with glycolic acid containing diethylenetriaminepentaacetic acid (DTPA), after which the individual lanthanides are successively eluted with dilute nitric acid.

The described procedure, outlined in Figure 3-1, uses an evaporation step between the third and final columns to reduce nitric acid to the proper level. This evaporation usually resulted in a solid residue which could not easily be dissolved. Excluding this residue from the final column resulted in low lanthanide-actinide recovery. If, on the other hand, the residue was transferred to the final column, the resulting slow leaching of lanthanides and actinides precluded a clear-cut separation of these elements.

Another technique to reduce the acidity was highly desirable. All attempts to adjust the acid concentration through buffering rather than evaporation, produced a bulky yellow-brown precipitate. The precipitate could be dissolved in an excess of ammonium hydroxide, indicating the presence of silver or cobalt, rather than iron. Since dissolution of the KCFC column would introduce both iron and cobalt into solution, it seemed more likely that silver paraperiodate was the progenitor of the precipitate. Various iodate and periodate salts had been investigated for cation retention in Company-sponsored work, and these were evaluated as a substitute for silver paraperiodate. Silver iodate and lead iodate produced effluents which could be buffered without a precipitate forming. Neither of these salts, however, completely removed cerium activity, presumably due to insufficient oxidizing potential. The addition of lead dioxide, a stronger oxidant, to either of these two salts resulted in complete cerium retention, superior ruthenium retention, and an effluent which could be buffered without precipitation. Since lead dioxide - lead iodate gave the most complete ruthenium retention, this mixture was adopted for this program.

With the precipitation problem eliminated, a suitable buffer was needed. Glycolic acid in the pH 2.5 to 3.5 range allows complete extraction of the lanthanides into D2EHP.² A 5 M ammonium glycolate solution was adjusted to pH 5.0 with ammonium hydroxide and the buffering behavior was evaluated as shown in Table 1.

This buffer is shown to be satisfactory for 1 N nitric acid over a wide range of acid-to-buffer volume ratios. A ratio of 3:1 was selected as a desirable midpoint value for the procedure.

The modified procedure is outlined in Figure 3-2. Distribution of fission product activity in this procedure is shown in Figure 3-3.

Table 1. Final pH of Various Ammonium Glycolate
Buffer - 1 N Nitric Acid Mixtures

<u>1 <u>N</u> HNO₃: buffer volume ratio</u>	<u>Final pH</u>
1.0	4.2
1.5	3.9
2.0	3.7
2.5	3.5
3.0	3.3
3.5	3.1
4.0	2.8
4.5	2.4

Figure 3-1. Outline of Previous Separation Procedure
Adapted for Thoria-Urania Fuel

Figure 3-2. Outline of Modified Separation Procedure Adapted for Thoria-Urania Fuel

Figure 3-3. Distribution of Fission Product Activity in Four-Column Procedure

3.2. Chemical Separation of Neptunium

Neptunium-237 is produced during the irradiation of uranium-containing fuels by the fast neutron induced $(n, 2n)$ reaction on ^{238}U , and by the (n, γ) reaction on ^{236}U followed by beta decay of the 6.75 day ^{237}U product. Because the ^{236}U concentration initially may be low compared to the ^{236}U produced by (n, γ) on ^{235}U , production by two successive (n, γ) reactions on ^{235}U may be the major production mode for ^{237}Np in highly enriched fuels. All of these processes are of sufficiently low cross section to make ^{237}Np a minor constituent of the irradiated fuel; preliminary estimates indicate a Th/Np ratio of 2×10^5 and a U/Np ratio of 10^4 . The high concentration of major fuel constituents relative to neptunium impose stringent decontamination requirements on the neptunium separation procedure.

3.2.1. Ion Exchange

Thorium is known to be strongly retained on cation exchange resin from dilute nitric acid.³ Zolotov and Nishanov⁴ report the separation of neptunium from uranium and plutonium with 1 N nitric acid on KU-2 cationite. These authors claim completion reduction of Np(VI) to Np(V) by the resin. Pentavalent neptunium is less strongly retained than hexavalent or tetravalent actinides and is therefore the first eluted from a mixture.

Since KU-2 resin is not commercially available, a study of neptunium-uranium separation was undertaken using Dowex 50-X4 with 1 N nitric acid as the eluant. The variables were resin mesh size and column height. The results are shown in Figure 3-4. Uranium-235 and ^{239}Np were used as tracers and the gamma activity in 1-ml fractions was measured to determine the elution curves. Gamma spectra indicated whether the activity was due to ^{235}U or ^{239}Np . Neptunium-239 had been prepared by irradiating depleted uranium in the BAWTR and separating ^{239}Np by the same cation exchange procedure being tested. Gamma spectra taken after the separation showed no impurities.

A 15-cm column of Dowex 50-X4 (100-200 mesh) gave adequate separation in a reasonable time and was selected to test the reproducibility and completeness of neptunium recovery. Five columns of this type were loaded with replicate samples of ^{239}Np in a simulated

thoria-urania dissolver fuel solution. Three-ml fractions of the eluate were collected. The ^{239}Np content of each fraction, determined by gamma counting, was compared to an aliquot identical to that loaded onto each column. The results are shown in Table 2.

The low recovery of ^{239}Np , ranging from 93.5 to 97.8% was thought due to the presence of some tetravalent neptunium, which would be retained strongly by the resin. To eliminate this possibility, excess potassium permanganate was added to the nitric acid solution of neptunium tracer. A portion of this hexavalent neptunium was then made 0.1 M with sodium nitrite and heated to form pentavalent neptunium.⁴ The recovery of neptunium in six replicate cation exchange separations with permanganate and permanganate-nitrite treatment is shown in Table 3.

Table 2. Distribution of ^{239}Np in Cation Exchange Column Effluent (%) in Five Replicate Separations

<u>1-3 ml</u>	<u>4-6 ml</u>	<u>7-9 ml</u>	<u>10-12 ml</u>	<u>13-15 ml</u>	<u>15-18 ml</u>	<u>Total</u>
0.14	92.4	5.2	0.06	0.02	0.01	97.8
0.03	90.1	3.5	0.01	-0.01	-0.03	93.6
0.22	86.0	10.2	0.07	-0.05	0.02	96.5
0.06	88.4	5.1	0.01	-0.05	-0.05	93.5
0.06	87.7	7.6	-0.03	0.02	-0.04	95.4

Table 3. Reproducibility of the Recovery of Pretreated ^{239}Np From Cation Exchange Separation (%)

<u>Pretreated with KMnO_4</u>	<u>Pretreated with KMnO_4, then NaNO_2</u>
97.7	96.6
96.6	95.2
96.3	95.9
95.2	95.5
92.2	96.1
97.2	95.5

Figure 3-4. Cation Exchange Separation of Neptunium and Uranium (Dowex 50-X4)

Recovery of neptunium seemed somewhat more consistent with the combined permanganate-nitrite pretreatment; however, it was still considerably less than quantitative recovery. Nelson and Michelson⁵ have studied the cation exchange behavior of neptunium in hydrobromic acid systems and found that neptunium is reduced to (IV) on the resin, even in the presence of bromine. Neptunium(IV) is undoubtedly stabilized by the high affinity of the resin for tetravalent cations. It is likely that a portion of the neptunium could be reduced to (IV) even in 1 N nitric acid, resulting in the observed low recovery.

It is worth emphasizing that cation exchange is satisfactory for those applications where radiochemically pure neptunium is more important than quantitative recovery. However, for this work another approach was desirable.

3.2.2. Solvent Extraction

Of the more versatile solvent extraction systems for the actinides are those of Maeck⁶⁻⁸ which involve tetraalkylammonium complexes of these elements. The procedure for neptunium⁷ is based on extraction of the tetrapropylammonium nitrate neptunium(VI) ion association complex into methyl isobutyl ketone (MIBK) from acid deficient aluminum nitrate salting solution. Tetravalent neptunium is then back extracted as (IV) into a ferrous chloride-hydroxylamine reducing solution* and re-extracted into thenoyltrifluoroacetone (TTA)-xylene. Uranium, which accompanies neptunium in the first extraction, neither back extracts into the aqueous strip solution nor re-extracts into TTA and is thus separated. However, this procedure cannot tolerate large amounts of thorium, which extracts well even from unsalted nitric acid.⁹

In a comprehensive study of 57 metal-tetraalkylammonium complexes from various acids, Maeck⁹ reported little or no extraction of thorium from hydrochloric acid. Although little neptunium(VI) extracted from this media as the tetrapropylammonium or tetrabutylammonium complex, nearly 75% extraction was reported with the tetrahexylammonium salt. It seemed likely that further lengthening of the

-----
* 0.25 M FeCl₂ - 0.5 M NH₂OH · HCl - 1.0 M HCl.

alkyl chain would enhance neptunium extraction, without significantly increasing the extraction of thorium from a hydrochloric acid system.

Solutions of tetraheptylammonium chloride (Eastman Organic Chemicals) were prepared in MIBK and nitropropane. Some of the common oxidants such as permanganate and argentic oxide are not useful in a hydrochloric acid system. Three oxidants which seemed compatible with chloride were evaluated. With cerium(IV), added as ceric ammonium nitrate, neptunium extraction did not exceed 90%. When the sample was fumed with perchloric acid, before adding the hydrochloric acid, neptunium extraction remained below 12%. Chlorate ion, added as potassium chlorate, reacted with chloride to form chlorine, a satisfactory oxidant. Extraction behavior of neptunium with chlorate oxidation, determined by measuring the ^{239}Np gamma activity in each phase, is shown in Table 4.

Neptunium extraction was found to exceed 99.5% from all of these systems at 5 N or greater hydrochloric acid concentration. Higher than 6 N was not studied with MIBK due to high mutual solubility of the two phases. Although neptunium is more extractable in nitropropane, it is also more difficult to back extract. The recovery of neptunium in a ten minute contact of the nitropropane phase with the reducing-strip solution was only about 95%. With MIBK as the diluent, back extraction exceeded 99%. Therefore, MIBK was selected as the diluent.

Table 4. Extraction of ^{239}Np From Hydrochloric Acid Into Various Tetraheptylammonium Chloride-Diluent Mixtures

Diluent tetraheptylammonium chloride concentration	MIBK, %		Nitropropane, %	
	0.25 <u>M</u>	0.5 <u>M</u>	0.25 <u>M</u>	0.5 <u>M</u>
4 <u>N</u> HCl	76	89	98.9	99.6
5 <u>N</u> HCl	99.6	99.8	--	--
6 <u>N</u> HCl	99.9	99.8	99.8	99.9
8 <u>N</u> HCl	--	--	99.9	99.7

Decontamination from extraneous activity was evaluated by processing mixed fission products in a simulated thoria-urania dissolver fuel solution.* Most of the ruthenium and about 10% of the zirconium extracted from 5 N hydrochloric acid. Repeated contacts with 5 N hydrochloric removed almost none of this activity; however, essentially all of the zirconium activity back extracted into the reducing-strip solution.

Scrubbing the organic phase with acid deficient aluminum chloride,** analogous to the aluminum nitrate used in nitrate systems,⁷ successfully removed the zirconium activity, leaving only ruthenium as shown by gamma spectral analysis. A portion of the ruthenium does back extract into the reducing-strip solution with neptunium (Figure 3-5), but is of little consequence as ruthenium volatilizes during the preparation of neptunium counting sources.

Due to the improved decontamination from fission products with an aluminum chloride scrub, it seemed reasonable that extracting initially from aluminum chloride would result in even better decontamination. This indeed was the case. However, extraction of neptunium was always low in aluminum chloride, presumably due to incomplete oxidation to Np(VI). The initial extraction from 5 N hydrochloric acid was therefore retained.

Thorium decontamination was evaluated by adding ²³⁴Th tracer to the simulated thoria-urania dissolver fuel solution. The ²³⁴Th was scavenged with iron carrier from a normal uranium carbonate solution. The iron and small amounts of uranium which were occluded were then retained on Dowex 1-X8 from 9 N hydrochloric acid, while carrier-free ²³⁴Th eluted.

Thorium decontamination was found to exceed 99.8% per contact from either 5 N hydrochloric acid or acid deficient aluminum

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* A simulated thoria-urania dissolver solution was prepared by adding thorium, uranium, HF and H₃BO₃ to a uranium-fission product solution. The resulting solution was 1 M HNO₃, 0.02 M HF, 0.09 M H₃BO₃ and contained 10 mg/ml of thorium and 1 mg/ml of uranium in addition to mixed fission products. An aliquot of this solution was evaporated to dryness and taken up to the original volume in 5 N HCl.

** 2.8 M AlCl₃, 2.0 M NH₄OH.

chloride salting solution. As the proposed separation procedure combines one contact with each of these media, the overall thorium decontamination factor is $(0.998/0.002)^2$ or 2×10^5 . This approximates the expected Th/Np ratio. When the respective specific activities and alpha decay energies of thorium and neptunium are considered, this decontamination is seen to be completely adequate.

The overall recovery of neptunium in this procedure is shown in Table 5. A very consistent recovery of 98.8% is seen, with the largest loss encountered in incomplete back extraction from the organic phase. The possibility of decreasing the loss at this step by a double contact with strip solution will be explored. In any event, the loss has been shown to be small enough and consistent enough that a bias correction can be applied with confidence.

The final step in the neptunium separation will be the extraction of neptunium(IV) from the reducing-strip solution into TTA. This organic phase, as previously reported,⁷ can be used directly to prepare the neptunium alpha counting plate for radiometric assay.

Table 5. Distribution of ^{239}Np in the Solvent Extraction Procedure(a)

Not extracted from 5 <u>N</u> HCl-KClO ₃	0.33 ± 0.05%
Removed in AlCl ₃ scrub	0.12 ± 0.04%
Remaining in organic phase after FeCl ₂ -NH ₂ OH strip	0.72 ± 0.03%
Recovery in final strip phase	98.88 ± 0.05%

(a) Based on six replicates.

Figure 3-5. Distribution of Fission Product Activity in Solvent Extraction Procedure for Neptunium

4. METALLURGICAL ANALYSIS

Before destructive analysis of the fuel rods, they will be gamma scanned and visually examined. The results of gamma scanning will guide the final selection of sampling locations for both isotopic and metallurgical analysis. Most of the rods have been gamma scanned, but data processing is still in progress. The scanning procedure and results will be described in the next quarterly report.

5. FUEL ROD PROCUREMENT

The fuel element design and the rod removal procedure were described in a previous report.¹ All the rods for this program have been removed from the respective elements at Battelle Memorial Institute and transported to the B&W Nuclear Development Center.

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