Babcock & Wilcox

MASTER

POST IRRADIATION EXAMINATION OF THORIA-URANIA FUEL RODS

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March - August 1967

RESEARCH & DEVELOPMENT
DIVISION

NUCLEAR DEVELOPMENT
CENTER

Lynchburg, Virginia

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POST IRRADIATION EXAMINATION OF THORIA-URANIA FUEL RODS

— Quarterly Progress Report No. 1 —

March - August 1967

by

W. N. Bishop
S. F. Marsh
C. J. Baroch

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Lynchburg, Virginia

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1.0 INTRODUCTION

The objectives of this program are to determine the isotopic content of several heavy elements and fission products in irradiated thorium-uranium fuel and to perform metallurgical examinations on the fuel, stainless steel cladding, and Zircaloy fuel can. The specimens will be taken from Core A of the Consolidated Edison Indian Point Reactor.

A total of 13 fuel rods from six elements has been selected for analysis. The rods will be gamma scanned and punctured for fission gas release. The results of the gamma scans will guide the selection of sampling positions for isotopic and metallurgical analysis.

The isotopic content of the transactinium elements and certain fission products will be determined. The heavy elements for which analyses will be attempted are thorium, protactinium, uranium, neptunium, plutonium, and a combined fraction of americium-curium. Cesium¹³⁷, ¹⁴³Nd, ¹⁴⁸Nd, ¹⁴⁷Pm, ¹⁴⁸Sm, ¹⁴⁹Sm, and ¹⁵⁰Sm are the fission products which will be determined.

Metallographic examinations will be made on the fuel and cladding. Specimens of the stainless steel cladding and Zircaloy can will be tension tested under several conditions.

This is the first in a series of quarterly progress reports on this program and covers the period March 15, 1967 - August 31, 1967.

2.0 SUMMARY

Thirteen fuel rods contained in six fuel elements were selected for this program. Eleven of these rods have been removed from four of the elements.

A rapid sequential chemical separation procedure for uranium, plutonium, americium-curium, cesium, neodymium, promethium, and samarium has been adapted from a separation procedure used for burnup analysis of uranium fuels.

3.0 FUEL ROD SELECTION

The fuel rods to be examined under this program are being taken from Core A of the Consolidated Edison Indian Point Reactor. The fuel is composed of thorium and highly enriched uranium (93% ^{235}U) oxides originally in pellet form.

3.1 Core Description

Detailed descriptions of the Indian Point Reactor were reported during the design and construction of the reactor,^{1,2} and only a synopsis will be presented here.

The reactor core contained 120 fuel elements. Each element is 137.50 inches long and 5.711 inches square. A cross section of the element is shown in Figure 3.1 and an over-all view in Figure 3.2. The fuel element assembly consists of a fuel bundle, a fuel can, an upper transition piece, a fuel element spring, and an upper nozzle and seal assembly.

The fuel was fabricated as high density thoria-urania pellets of close dimensional tolerance, contained in tubing made of a modified Type 304 stainless steel containing 200-250 ppm of boron. Each tube was closed at the ends with suitable end caps. The outside diameter of the fuel rod was 0.304 inches and the clad thickness was 0.0205 inches. Between the fuel pellets and the clad there was an initial gap (at room temperature) nominally 0.0015 inches thick. This gap was filled with helium gas. Each bundle (subassembly of tubes) contained 195 tubes, spaced by ferrules (short, thin, tubular sections) in a 14 x 14 square array with one corner tube omitted. The ferrules were spaced axially 9 inches center-to-center in planes perpendicular to the longitudinal axis of the

bundle. A row of smaller ferrules was spaced around the outside of the bundle at each plane of ferrules to support the outer row of tubes, and to separate the fuel tubes from the can wall. The entire bundle was brazed as a unit.

The fuel element can was made of Zircaloy-2 envelope surrounding the tube bundle and fitted at each end with transition pieces. A spring in each fuel element, locked in place by the top transition piece assembly, held down the fuel bundle against hydraulic forces, while permitting thermal expansion of the bundles.

Twenty-one movable control rods were mounted between the fuel element cans. Fixed control rods and flux depressor plates were also located between fuel elements. The core and the positions of fuel elements and control members are shown in Figure 3.3.

The core contained three zones with different fuel loadings. The arrangement of the three zones is shown in Figure 3.3, where Zone I is composed of elements 1-32, Zone II of elements 33-76, and Zone III of elements 77-120.

Internally, each fuel element was composed of fuel rods of several different ^{235}U concentrations. The core contained fuel pellets of six different thorium to uranium ratios. Figure 3.4 shows the loading arrangement for each of the three different types of fuel elements.

Table 3.1 shows the loading of the various fuel rods in the core. The uranium was enriched to 93% ^{235}U .

The average fuel pellet density was 9.25 gm/cm³. Fuel pellet diameter was 0.260 inches. The pellets were stacked to an active fuel length of 98.5 inches at 68 F.

3.2 Core History

An extensive gamma scanning program was conducted at core midlife.³ One complete octant plus selected elements to show power symmetry were scanned. The elements for this program are being taken from this same octant which is shown in Figure 3.5. Elements which were adjacent to control rods which were not always out when the reactor was at power were not considered. Control rods 12, 13, and 17 were in this category and elements 21, 22, 57, 58, 27, 28, and 65 were therefore eliminated. From those remaining elements 20, 61, 62, 66, 105, and 106 were chosen.

It is impractical to remove rods from the inner rows of the elements by the techniques used, and if only one rod is to be taken, it is much simpler to remove a corner rod. Only type A and F rods are easily accessible and F rods have no ²³⁵U in the center portion. Thus, a type A rod will be taken from each element. It is possible to obtain a rod of each type from a Zone III element by taking the two rods (A and B) adjacent to the index strip and five rods (F, C, D, D, E) from the corner diagonally opposite. Since element 106 had a much lower burnup than element 105, the latter was chosen for obtaining one rod of each type. The fuel rod selection is summarized in Table 3.2.

The Zircaloy can will be taken from element 61. This element is predicted to have the highest burnup of the six elements selected for this program.

The date of final operation for Core A was October 20, 1965, after operating for 442 effective full power days at 585 megawatts thermal. The core average burnup was estimated to be 16 GWD/tonne.

Figure 3.1: Fuel Element Cross Section

Fuel Rod Detail

Figure 3.2: Fuel Element

Figure 3.3: Core Hardware Position Designations

Figure 3.4: Fuel Element Loading Patterns

ZONE I

(ALL UNMARKED RODS ARE ENRICHMENT C)

ZONE II

(ALL UNMARKED RODS ARE ENRICHMENT D)

ZONE III

(ALL UNMARKED RODS ARE ENRICHMENT E)

LEGEND:

- A — LOWEST ENRICHMENT
- B — SECOND LOWEST ENRICHMENT
- C — THIRD " "
- D — FOURTH " "
- E — HIGHEST ENRICHMENT
- F — $1/3 \text{ THO}_2 + 2/3 \text{ ENRICHMENT A}$

Figure 3.5: Core Octant for Fuel Rod Selection

TABLE 3.1. FUEL ROD LOADING

<u>Rod Type</u>	<u>Th U</u>	<u>Th/²³⁵U</u>	<u>²³⁵U Th+U</u>
A	31.1	33.4	.029
B	21.4	23.0	.041
C	15.9	17.1	.055
D	11.4	12.3	.075
E	9.0	9.6	.0935
F	Center 1/3 ThO ₂ only; Outer Two 1/3 Type A		

TABLE 3.2. FUEL ROD SELECTION

<u>Zone</u>	<u>Element No.</u>	<u>Rod Type</u>
I	20	A
II	61	A
II	62	A
II	66	A
III	105	A, B, C, D, D, E, F
III	106	A, B

4.0 ISOTOPIC ANALYSIS OF THORIA-URANIA FUEL

4.1 Chemical Separation Procedures

Chemical separation procedures for uranium, plutonium, americium-curium, neodymium, promethium, samarium, and cesium have been devised by modifying a procedure previously used in Company programs. The modified procedure employs two extraction chromatography columns and two inorganic ion exchange columns in the sequential separation of the above elements. The first extraction column, di-(2-ethylhexyl)-phosphoric acid (D2EHP) on a Chromosorb support, retains thorium, uranium, plutonium, and zirconium from 1N nitric acid. The effluent from this column flows directly onto a column of potassium cobaltoferrocyanide ($K_2CoFe(CN)_6$, also known as KCFC), which selectively retains cesium. The effluent from this column flows directly onto a column of silver paraperiodate (Ag_5IO_6), which retains cerium and ruthenium. The effluent from the third column, now sufficiently free of activity for safe handling in unshielded facilities, is adjusted to lower acidity and passed through a final D2EHP column which retains the lanthanides and trivalent actinides. The trivalent actinides, americium and curium, are first eluted with glycolic acid containing diethylenetriaminepentaacetic acid (DTPA), after which the individual lanthanides are successively removed with dilute nitric acid.

4.1.1 Existing Chemical Separations

Inasmuch as the techniques used in burnup analysis at the Babcock & Wilcox Nuclear Development Center encompass some parts of the work program for this project, an effort was made to limit development work to modifications of

this basic procedure. In the existing approach to burnup analysis the number of fission events, based on radioactive ^{137}Cs and stable ^{148}Nd fission product concentrations, are compared to the concentration and isotopic composition of the residual fuel. From these data atom percent burnup, fuel depletion, and alpha (the capture to fission ratio for the fissioning isotope) may be calculated.

The separation procedure for cesium, neodymium, and uranium (Figure 4.1) employs three columns for the sequential separation of these elements. The first is an inorganic exchange column of silver paraperiodate, on which a large part of the unwanted activity is removed. The second column contains potassium cobaltoferrocyanide, which has a high affinity for cesium. The final column is of the extraction type for separating neodymium and uranium.

First Column

Silver paraperiodate, Ag_5IO_6 , efficiently and selectively removes certain fission product elements.⁴ In addition to zirconium, ruthenium, and tetravalent plutonium, this inorganic exchange material also removes cerium through oxidation to the highly insoluble ceric salt. The removal of ^{144}Ce and its 17-minute ^{144}Pr daughter is quite important since this eliminates a large part of the radiation hazard in the remainder of the procedure. The mechanism is presumably the replacement of silver ions with ions forming less soluble iodate-periodate salts; however, ruthenium is not usually considered part of this group. No satisfactory explanation for the observed retention of ruthenium is presently available.

Second Column

The effluent of the silver paraperiodate column flows directly onto a column of potassium cobaltoferrocyanide (KCFC). This material, recently proposed for cesium removal by Prout⁵ and Boni⁶, selectively removes the heavy alkali metal ions which form less soluble cobaltoferrocyanide salts than potassium. Due to the lack of other alkali metal activity in fission products cooled greater than six months, this material is specific under these conditions for ^{137}Cs .

Third Column

The final column consists of di-(2-ethylhexyl)-phosphoric acid (D2EHP) on a support of siliconized Chromosorb particles. This extraction chromatographic technique produces the equivalent of multistage solvent extraction for resolving such chemically similar elements as the lanthanides. The high distribution values of tetravalent and trivalent cations into D2EHP (Figure 4.2) is well known. Less well known, however, is the usefulness of D2EHP in separating the lanthanide group from the trivalent actinides. Weaver and Kappelmann⁷ have reported a solvent extraction process for recovering trivalent actinides in which large group separation factors were observed. This separation was due to the preferential extraction of the lanthanides into the diluted organophosphate while the trivalent actinides were preferentially complexed in the aqueous phase by a suitable simple carboxylic acid-amino polyacetic acid mixture. In our case the aqueous phase is 1M glycolic acid-0.016M diethylenetriaminepentaacetic acid (DTPA). The pronounced effect of DTPA on separating the lanthanides from americium-curium is shown in Figure 4.3. Subsequent washing of this column with dilute nitric acid elutes neodymium, free of interfering elements.

An interesting observation, quite important to this chemical separation scheme, is that uranyl ion (UO_2^{++}) is retained by D2EHP from even strong nitric acid (Figure 4.2), in contrast to its typical divalent cation behavior in hydrochloric acid. The mechanism of the higher-than-expected extraction from nitric acid may involve an oxonium-type extraction of neutral uranyl nitrate into the oxygenated organic solvent. In any event, the much lower extraction from hydrochloric acid provides the basis for eluting uranium as the final step of the procedure.

4.1.2 Adaptation to Thoria-Urania Fuel

Complete sample dissolution and representative sampling are essential to any successful analysis. In the post irradiation analysis of the Consolidated Edison Indian Point Reactor core, the samples are a mixed thoria-uranium, with the thoria content ranging from 90 percent to 100 percent. Because of the high thoria content and the chemical inertness of refractory thoria, the problem of complete dissolution and representative sampling is formidable.

The most satisfactory dissolution medium for refractory thorium dioxide is fluoride-catalyzed, concentrated nitric acid. Thoria which has been compacted to high density and then irradiated is the most difficult to dissolve, as the "blue oxide" is formed.⁸ Prolonged heating with fluoride-catalyzed nitric acid will eventually dissolve even this material, according to the literature.

The presence of micro amounts of fluoride and macro amounts of thorium in the dissolved sample were expected to interfere in some of the previously described separation techniques. Synthetic mixtures containing the anticipated levels of thorium, uranium, fission products, and fluoride were

therefore prepared and investigated. As expected, the retention of fission product zirconium and cerium on silver paraperiodate was significantly reduced by competing fluoride complexation. The fluoride interference was overcome by adding a sufficiently large excess of boric acid to preferentially complex the fluoride. However, thorium was still incompatible with the original three-column procedure.

The separation procedure has been modified (Figure 4.4) by preceding the three-column procedure, as previously described, with a shorter extraction column of D2EHP on Chromosorb. By maintaining 1N nitric acid through this column, only thorium, uranium, plutonium, and zirconium are retained. The effluent passes through a KCFC column for cesium removal, and a silver paraperiodate column for removal of cerium and ruthenium, as before. The acidity then is reduced tenfold for loading the final D2EHP volume prior to (A) elution of americium and curium with glycolic acid-DTPA, and (B) elution of neodymium with dilute nitric acid. A continuation of this elution with slightly more concentrated nitric acid also isolates promethium and samarium, free of interferences.

Uranium, retained on the initial extraction column in the modified procedure, is selectively eluted with 6N hydrochloric acid while the tetravalent cations remain on the column. Further washing with 6N hydrochloric acid-1% hydroiodic acid reduces plutonium to the non-retained trivalent state which then elutes. Thorium and zirconium, which are only tetravalent in solution, remain on the column. Their removal, while possible, is impractical in this scheme.

Modifications have thus resulted in a four-column procedure which sequentially separates uranium, plutonium,

cesium, americium-curium, neodymium, promethium, and samarium in forms suitable for their respective measurement. Uranium, plutonium, neodymium, and samarium are measured by isotope dilution mass spectrometry. Cesium¹³⁷, ¹⁴⁷Pm, and ²⁴¹Am are measured by alpha, beta, and gamma spectrometry. Alpha spectrometry will be used to measure important isotopes whose low abundances are expected to preclude accurate mass spectrometric analysis. These isotopes include ²³²U and ²³⁸Pu.

4.2 Mass Spectrometric Standards

The high specific activity of spent nuclear fuels and the accompanying hazards to personnel make it desirable to work with the smallest sample aliquot consistent with the required accuracy and the capabilities of the measurement technique. The preferred technique for high accuracy measurement of individual stable and long-lived isotopes is isotope dilution mass spectrometry, in which microgram samples are adequate for many elements. However, when using an enriched isotope of the same element, the concentration of the enriched isotope should approximate that of the sample and it must be accurately known. Because chemical methods of analysis are not sufficiently reliable at these very low concentrations, it is necessary to prepare standardized solutions of the natural element at the milligram per gram level from high purity material. The standard solutions are diluted accurately by weight to the microgram per gram level. The enriched isotope solutions, at the microgram per gram level, are then calibrated against the diluted natural element solutions by mass spectrometry.

Enriched isotope spikes have been prepared as follows. (The same neodymium spike prepared earlier for Company-sponsored work is being used for this project.)

Uranium

A standard solution of the natural element was prepared from NBS 950a U_3O_8 after repeated ignition at 900 C to insure stoichiometry. This material is used to calibrate a solution of 98% ^{233}U which, in turn, was used to calibrate the 99.96% depleted ^{238}U to be used in this work.

Neodymium

A standard solution was prepared from 99.8% natural element oxide after repeated ignition at 900 C to insure stoichiometry. This solution was additionally standardized by ethylenediaminetetraacetic acid (EDTA) titrimetry. The diluted natural element solution is being used to calibrate the 96.0% enriched ^{150}Nd to be used in this work.

Samarium

A standard solution was prepared from the 99.99% natural element oxide after repeated ignition at 900 C to insure stoichiometry. The diluted natural element solution is being used to calibrate the 94.5% enriched ^{144}Sm to be used in this work.

Plutonium

A standard solution has been prepared from NBS 949a plutonium metal standard. The dilution of this solution will be used to calibrate the 99.88% enriched ^{242}Pu obtained for this work.

Figure 4.1: Outline of Separation Used for B&W
Burnup Program (UO₂ Fuel)

Figure 4.2: Extraction of Various Metal Ions into
Di-(2-ethylhexyl) phosphoric acid (D2EHP)^{9, 10}

Figure 4.3: Separation of Lanthanides From Am-Cm⁷

Figure 4.4: Outline of Separation Procedure Adapted for Thoria-Urania Fuel

5.0 METALLURGICAL EXAMINATION

The major effort on this task does not begin until the fuel rods and Zircaloy-2 can are in the Babcock & Wilcox hot cells. However, some preliminary preparations for these examinations were completed.

Sketches of the tensile specimens to be obtained from the cladding and Zircaloy-2 can are shown in Figures 5.1 and 5.2. The exact specimen design for the cladding has not been determined as it is not known how difficult it will be to remove the fuel. If the fuel can be easily removed, then the tubular design will most likely be used. Templates for cutting out the tensile specimens have been ordered.

Thermal aging of the cladding from a prototype fuel bundle was initiated. Specimens will be aged at 600 F for 3000 and 6000 hours to determine whether time at temperature has any significant effect on the tensile properties of the cladding.

A meeting was held with representatives of Battelle Memorial Institute and Ohio State University to discuss their phase of the metallurgical examination. It was agreed that the examination should be directed towards a better understanding of the mechanisms involved in the effects of irradiation on the behavior of stainless steel. With this background, OSU and BMI agreed to prepare a detailed description of their program for review by B&W. The description should be received early in the next quarter.

Figure 5.1: Dimensions of Tensile Specimens for
Stainless Steel Cladding

Tubular Specimen

Split Tubular Specimen

Dimensions in Inches

Figure 5.2: Dimensions of Tensile Specimens for Zircaloy-2 Can

Dimensions in Inches

6.0 FUEL ROD PROCUREMENT

Individual fuel rods are removed from the bundles in the hot cells at Battelle Memorial Institute. The spent fuel is stored at the Nuclear Fuel Services reprocessing plant at West Valley, New York. Pairs of the elements are shipped to BMI where the elements are disassembled, the fuel rods are removed, the elements are reassembled and returned to NFS.

6.1 Fuel Element Disassembly

The fuel element is described briefly in section 3.1 and is shown in Figure 3.2. Disassembly of the element is accomplished by compressing the spring, rotating the locking pin 45 degrees and slowly releasing the tension on the spring. The upper nozzle and transition assembly are removed and the fuel bundle can be pushed out of the can. Reassembly of the element is the reverse of the disassembly procedure.

6.2 Fuel Rod Removal

The fuel rods are held together by a series of stainless steel ferrules brazed to the rods at 9-inch intervals along the rod length. To remove a peripheral rod from the bundle, the ferrules are slit by a circular saw driven by a slow-speed gear motor on a two-way track.

6.3 Rod Procurement Progress

To date the rods have been removed from elements 62, 66, 105, and 106. Eleven of the thirteen rods have been removed from the elements.

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Attn: T. Stern (2)
R. Allio
-

19. Westinghouse Electric Corporation (5)
Bettis Atomic Power Laboratory
P.O. Box 79
West Mifflin, Pennsylvania 15122

Attn: E. J. Kreigh
R. H. Fillnow
B. Lustman
W. J. Babyak
J. J. Taylor

20. Idaho Nuclear Corporation (2)
P.O. Box 1845
Idaho Falls, Idaho 83401

Attn: J. M. Beeston
W. C. Francis

21. The Babcock & Wilcox Company (91)

Arthur, VD	Kosiancic, EJ/Carlson, RV
Ball, RM	Lewis, RH
Baroch, CJ (2)	Library (2)
Barringer, HS	Littrell, LW (2)
Bishop, WN (45)	Lynch, ED
Breazeale, WM	Marsh, SF (2)
Central Files (6)	McDaniel, GT
Clark, RH	Moncrief, EC
Collings, DM	Moore, WT
Deuster, RW	Munim, AV
Dewell, EH	North, DC
Engelder, TC	Poor, HH/Alliance
Ewing, JW	Prince, LK
Gibson, WR	Probert, PB/Barberton
Grant, SP	Rosenberger, GG
Harbinson, EN	Schmitt, DH
Holt, CD	Travis, CC/TRG
Hostetler, DR	Vorres, KS/Rubright, MM/Alliance
Johnson, CR	Weissert, LR