GEL-ADDITION PROCESS CHEMICAL STUDIES

— Quarterly Progress Report No. 18 —

November 1970 - January 1971

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Research and Development Division
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R. V. Carlson

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ABSTRACT

The development of a sol-gel process and related processes for preparing (U,Pu)O₂ fast reactor fuels has continued at the Babcock & Wilcox Lynchburg Research Center. Progress for the 18th quarter, ending January 31, 1971, is reported. The program is sponsored by the U.S. Atomic Energy Commission under Contract No. AT(30-1)-3714.

Eighteen fuel pins containing ²³⁸UO₂-20% PuO₂ in the form of pellets, shards, and sol-gel microspheres have been shipped to the EBR-II for irradiation in an unencapsulated bundle. Fuel was fabricated and loaded in 16 additional pins during this quarter. Most of these will replace pins in the EBR-II bundle during scheduled outages.

Nine capsules irradiated in the BAWTR were disassembled previously. Gas analyses and detailed metallographic examinations of the 27 fuel pins contained in these capsules are in progress in B&W's hot cell facilities. Photomicrographs of "as-ground" cross sections of sphere-pac and pellet pins exposed at 6, 10, 15, and 20 kW/ft are shown.

CONTENTS

	Page
1. INTRODUCTION	1-1
2. RESULTS AND CONCLUSIONS	2-1
2.1. Fuel Preparation	2-1
2.2. Fuel Fabrication	2-1
2.3. Irradiation Testing	2-1
2.4. Analytical Support	2-2
3. FUEL PREPARATION	3-1
4. FUEL FABRICATION	4-1
4.1. Fabrication of UO ₂ Insulator Pellets	4-1
4.2. Fabrication of EBR-II Fuel	4-2
4.3. Loading EBR-II Pellet Pins	4-2
4.4. Loading EBR-II Vipac Pins	4-3
4.5. Loading EBR-II Sphere-Pac Pins	4-5
5. IRRADIATION TESTING	5-1
5.1. Time Required for Inpile Restructuring	5-1
5.2. EBR-II Irradiations	5-3
6. ANALYTICAL SUPPORT	6-1
6.1. Characterization of EBR-II Fuel	6-1
6.2. Ceramographic Examination of EBR-II Fuel	6-1
7. REFERENCES	7-1
DISTRIBUTION	A-1

List of Tables

Table

4-1. FY-1971 EBR-II Pellet Pin Data	4-3
4-2. FY-1971 EBR-II Vipac Pin Data	4-4
4-3. Smeared Density of Each Segment of FY-1971 Vipac Pins	4-4

Tables (Cont'd)

Table		Page
4-4.	FY-1971 EBR-II Sphere-Pac Pin Data.	4-5
5-1.	Description of Irradiated BAWTR Fuel Pins Being Examined	5-2
6-1.	FY-1971 EBR-II Fuel Characterization	6-2
6-2.	Trace Metallic Impurities in FY-1971 EBR-II Fuel	6-3

List of Figures

Figure		
5-1.	As-Ground Cross Section of Sphere-Pac Pin C-69 (Irradiated 4 days at 6 kW/ft)	5-4
5-2.	As-Ground Cross Section of Sphere-Pac Pin C-78 (Irradiated 4 days at 10 kW/ft)	5-4
5-3.	As-Ground Cross Section of Sphere-Pac Pin C-74 (Irradiated 1 day at 15 kW/ft)	5-5
5-4.	As-Ground Cross Section of Sphere-Pac Pin C-82 (Irradiated 4 days at 20 kW/ft)	5-5
5-5.	As-Ground Cross Section of Pellet Pin C-1 (Irradiated 4 days at 6 kW/ft)	5-6
5-6.	As-Ground Cross Section of Pellet Pin C-102 (Irradiated 4 days at 10 kW/ft)	5-6
5-7.	As-Ground Photomosaic of Pellet Pin C-15 (Irradiated 1 day at 15 kW/ft)	5-7
5-8.	As-Ground Cross Section of Pellet Pin C-88 (Irradiated 3.5 days at 20 kW/ft)	5-8
6-1.	Photomicrograph of UO ₂ Insulator Pellet	6-4
6-2.	Photomicrograph of EBR-II Fuel Pellet	6-4
6-3.	Photomicrograph of EBR-II Coarse Microspheres	6-4
6-4.	Alpha Autoradiograph of EBR-II Fuel Pellet	6-4
6-5.	Alpha Autoradiograph of EBR-II Coarse Microspheres	6-4

1. INTRODUCTION

This is the 18th progress report on a continuing program of experimental work on sol-gel and related methods for preparing mixed uranium-plutonium oxide fuels for fast reactor application. The program is sponsored by the U.S. Atomic Energy Commission under Contract No. AT(30-1)-3714. This report covers the period from November 1, 1970, to January 31, 1971.

The work program for the contract establishes several technical tasks, including fuel preparation and fabrication, reactor testing in both thermal and fast neutron fields, and analytical support. For FY-1971, the primary objectives are to prepare 16 additional EBR-II pins containing fully enriched UO_2 -20% PuO_2 , and to continue the post-irradiation examination of the BAWTR capsules consistent with the funds available.

The 16 EBR-II pins include five pellet, five vipac, and six sphere-pac pins. This number will provide sufficient pins for the scheduled reloading at 50,000 and 75,000 MWd/t burnup, as well as two spares or archive samples for each fuel form. The fuel for these pins has been prepared, the pellets and shards have been fabricated, and the fuel has been loaded into the pins. Xe-tagging, top end cap welding, and final inspection will be completed next quarter.

Nine of the 16 capsules irradiated in the BAWTR were disassembled in B&W's hot cell facilities during FY-1970. Gas analyses and detailed metallographic examination of the 27 pins contained in these capsules are in progress. The progress in all technical areas of the program is reviewed in the following sections.

2. RESULTS AND CONCLUSIONS

2.1. Fuel Preparation

The FY-1971 work program for this task was completed during the past quarter.

2.2. Fuel Fabrication

The second batch of UO_2 insulator pellets required for the EBR-II reload pins has been prepared. The surfaces of the pellets were contaminated with molybdenum during the firing, but mechanical cleaning removed enough of the contamination to make the pellets acceptable. The fabrication of shards to be used as vipac feed was also completed. Fuel was loaded into the 16 EBR-II reload pins. Fourteen of the 16 were within the smeared density specifications. The smeared density of the two spare pellet pins was slightly higher than specified, but close enough to be considered acceptable. This completes the FY-1971 work program for this task.

2.3. Irradiation Testing

Eighteen acceptable EBR-II pins were shipped to the EBR-II site and are being inspected by EBR-II personnel.

Gas analyses and detailed metallographic examinations of 27 fuel pins previously irradiated in the BAWTR are progressing in B&W's hot cell facilities. All of the pins have been punctured under vacuum to collect the fission and sorbed gases released. Sixteen of the pins have been sectioned in the transverse direction, and specimens are undergoing metallographic examination. Photomicrographs of the "as-ground" cross sections have been examined, and several preliminary conclusions have been reached. Apparently, particulate fuels restructure so rapidly that their low initial thermal conductivity does not result in center melting when the linear heat rate is rapidly increased up to 20 kW/ft. The

photomicrographs of sphere-pac pins show that spheres are still present next to the cladding.

2.4. Analytical Support

All of the fuel for the EBR-II reload pins has been completely characterized. The results show that the impurity levels are within those specified for fast reactor fuel. This completes the FY-1971 work program for this task.

3. FUEL PREPARATION (R. V. Carlson)

The FY-1971 fuel order includes 1200 g of coprecipitated powder and 600 g of sol-gel microspheres containing 20% PuO_2 in UO_2 enriched to 93% ^{235}U for the EBR-II irradiation tests. The fuel order was completed during the past quarter.¹ Approximately 1700 g of coprecipitated powder for fuel fabrication and 840 g of sol-gel microspheres for pin loading were delivered (see section 4). No additional fuel is to be prepared.

4. FUEL FABRICATION (A. H. Bremser)

The FY-1971 order includes the fabrication of sufficient pellets and shards from coprecipitated powder (see section 3) to load five pins with each type of fuel for the EBR-II irradiation tests. In addition, six pins are to be loaded with sol-gel microspheres. Most of these 16 pins will be loaded in the EBR-II bundle during scheduled outages, and the remainder will serve as archive samples or as replacements for rejected pins.

4.1. Fabrication of UO_2 Insulator Pellets (A. H. Bremser, D. L. Fowler, L. J. Ferrell)

About 500 UO_2 insulator pellets are required for the EBR-II reload pins. The first batch was fabricated previously.² However, difficulties were encountered during the sintering of the remaining pellets—the surfaces of two separate batches were contaminated with spots of molybdenum. By a process of elimination, the problem was thought to be a furnace malfunction that could not be readily repaired. Therefore, the pellets in the two contaminated batches were separated by density, and an attempt was made to decontaminate the acceptable pellets—those with a density of $93 \pm 2\%$ theoretical. The ends were cleaned with silicon carbide paper, and the diametral surface was assumed to be cleaned during the centerless grinding. After the pellets were washed and dried to remove residual grinding media, one of those that had been more heavily contaminated was submitted for semi-quantitative analysis by emission spectrography. The results showed that the pellet contained less than 100 ppm of molybdenum. Therefore, the pellets were accepted and combined with the first batch to form one lot. The lot was then sampled in accordance with the quality verification plan,³ and the remaining pellets were assembled into seven-inch stacks.

4.2. Fabrication of EBR-II Fuel (L. J. Ferrell, A. H. Bremser)

Acceptable pellets for the pellet pins and green pellets for the shard fuel were fabricated previously.² The green pellets were fired during this quarter, and 80 were selected for density measurement. The average density was 93.5% of theoretical, well within the specification of $93 \pm 2\%$ of theoretical. The pellets were then crushed and screened into the required size fractions for vipac feed material.

4.3. Loading EBR-II Pellet Pins (A. H. Bremser)

Five pellet pins were loaded—three with fuel prepared in FY-1971 and two with fuel prepared in FY-1970. The latter pellets were obtained by unloading two pins whose cladding had been damaged during the final inspection of the first lot of EBR-II pins. Several of these pellets, which were chipped during unloading, were replaced with FY-1971 fuel pellets. The new pellets were placed at the ends of the fuel stacks. The density of the FY-1971 pellets was near the upper limit of the specification ($93 \pm 2\%$ theoretical), and the diameter (0.206 inch) was at the midpoint of the specification (0.206 ± 0.001 inch). However, based on a pin ID of 0.218 inch, the use of this fuel would result in an average pin smeared density of 84.0% theoretical. Since (1) the specification's smeared density is $82 \pm 1\%$ theoretical, (2) the pins containing FY-1970 pellets have a smeared density near 83% theoretical, and (3) only one of the pellet pins containing FY-1971 pellets is scheduled for loading into the EBR-II, it was decided to sort the FY-1971 pellets by density to obtain one pin with the lowest possible smeared density. This resulted in the three pins loaded with FY-1971 pellets having smeared densities of 82.5, 84.7, and 84.8% theoretical. The latter two pins are spares. Loading data for the second lot of EBR-II pellet pins are shown in Table 4-1.

Table 4-1. FY-1971 EBR-II Pellet Pin Data

		Pin				
		D-41	D-38	D-51	D-50	D-42
UO ₂ blanket pellets	Length, in.	5.92	5.92	5.90	5.91	5.90
	Weight, g	36.54	35.15	35.26	34.78	34.79
Fiberfrax	Length, in.	0.06	0.06	0.06	0.06	0.06
Bottom insulators	Length, in.	0.500	0.500	0.503	0.502	0.502
	Weight, g	3.06	3.04	3.06	3.03	3.11
Fuel	Length, in.	13.53	13.44	13.45	13.51	13.50
	Weight, g	74.90	76.43	76.59	75.37	75.04
	Smeared dens.	82.45	84.71	84.82	83.10	82.80
Top insulators	Length, in.	0.501	0.502	0.500	0.502	0.500
	Weight, g	3.04	3.02	2.94	3.08	2.95
Fiberfrax	Length, in.	0.06	0.06	0.06	0.06	0.06
Nickel reflector	Ident No.	D-17	D-24	D-1	40	41
Spring	Ident No.	D-17	D-24	D-1	40	41
Plenum sleeve	Ident No.	D-16	D-24	D-6	42	40
(U,Pu)O ₂ fuel	Year made	FY-71	FY-71	FY-71	FY-71-70 ^(a)	FY-71-70 ^(b)
UO ₂	Year made	FY-71	FY-71	FY-71	FY-71	FY-71

(a) Bottom three pellets and top two pellets FY-71 fuel; remainder FY-70 fuel.

(b) Bottom two pellets and one top pellet FY-71 fuel; remainder FY-70 fuel.

4.4. Loading EBR-II Vipac Pins (D. L. Fowler)

A vipac technique was used to load five pins with FY-1971 particulate feed (see section 4.2). The fuel was loaded sequentially in three 4.5-inch segments to a smeared density of $78 \pm 2\%$ theoretical in order to obtain vipac pins similar to the first batch of EBR-II pins.⁴ Data for these pins are shown in Table 4-2, and the smeared density of each segment is given in Table 4-3.

Table 4-2. FY-1971 EBR-II Vipac Pin Data

		Pin				
		D-37	D-54	D-53	D-40	D-34
UO ₂ blanket pellets	Length, in.	5.91	5.95	6.10	5.95	5.93
	Weight, g	34.83	35.23	36.20	35.31	35.20
Fiberfrax	Length, in.	0.06	0.06	0.06	0.06	0.06
Bottom insulators	Length, in.	0.501	0.501	0.500	0.502	0.501
	Weight, g	3.10	3.05	3.10	2.99	3.00
Fuel	Length, in.	13.52	13.42	13.59	13.53	13.59
	Weight, g	70.76	70.89	70.89	70.89	70.89
	Smeared dens.	77.96	78.69	77.71	78.05	77.71
Top insulators	Length, in.	0.502	0.500	0.500	0.502	0.500
	Weight, g	3.05	3.10	3.01	3.07	3.11
Fiberfrax	Length, in.	0.06	0.06	0.06	0.06	0.06
Nickel reflector	Ident No.	46	45	44	43	42
Spring	Ident No.	46	45	44	43	42
Plenum sleeve	Ident No.	D-7	D-11	D-25	41	43
(U, Pu)O ₂ fuel	Year made	FY-71	FY-71	FY-71	FY-71	FY-71
UO ₂	Year made	FY-71	FY-71	FY-71	FY-71	FY-71

Table 4-3. Smeared Density of Each Segment of FY-1971 Vipac Pins

Pin	Smeared Density, % theor		
	Bottom segment	Middle segment	Top segment
D-37	77.61	75.45	81.09
D-54	78.75	77.88	79.46
D-53	78.05	77.36	77.71
D-40	78.22	77.88	78.05
D-34	76.36	76.86	80.00

4.5. Loading EBR-II Sphere-Pac Pins (A. H. Bremser)

After initial firing of the coarse spheres, their density was too low to permit the loading of sphere-pac pins to the specification's smeared density of $78 \pm 2\%$ theoretical. Therefore, the spheres were refired to 1500C under a flowing N_2 -6% H_2 atmosphere with a two-hour soak at temperature. The density of the refired spheres was suitable for loading the sphere-pac pins. Data for these pins are shown in Table 4-4.

Table 4-4. FY-1971 EBR-II Sphere-Pac Pin Data

		Pin					
		D-43	D-52	D-36	D-44	D-35	D-39
UO ₂ blanket pellets	Length, in.	6.08	6.03	6.03	6.02	5.93	5.93
	Weight, g	36.54	37.22	36.85	36.85	35.14	35.14
Fiberfrax	Length, in.	0.09	0.06	0.06	0.06	0.06	0.06
Bottom insulators	Length, in.	0.500	0.500	0.500	0.500	0.501	0.501
	Weight, g	2.98	3.10	3.11	3.11	3.10	3.06
Fuel	Length, in.	13.49	13.56	13.55	13.46	13.52	13.59
	Weight, g	71.19	71.19	71.19	71.19	71.11	71.19
	Smeared dens.	78.61	78.20	78.26	78.78	78.35	78.03
Top insulators	Length, in.	0.502	0.500	0.500	0.500	0.501	0.501
	Weight, g	3.08	3.10	3.02	3.11	3.04	3.04
Fiberfrax	Length, in.	0.06	0.06	0.06	0.06	0.06	0.06
Nickel reflector	Ident No.	50	51	48	47	49	D-2
Spring	Ident No.	50	51	48	47	49	D-2
Plenum sleeve	Ident No.	D-28	D-32	D-8	D-13	D-1	D-15
(U,Pu)O ₂ fuel	Year made	FY-71	FY-71	FY-71	FY-71	FY-71	FY-71
UO ₂	Year made	FY-71	FY-70	FY-70	FY-70	FY-71	FY-71

5. IRRADIATION TESTING (E. N. Harbinson)

5.1. Time Required for Inpile Restructuring (E. N. Harbinson, R. E. Womack)

The effects of heat rate and time on the restructuring characteristics of sol-gel $\text{UO}_2\text{-PuO}_2$ spheres and of angular particles and pellets prepared from coprecipitated $\text{UO}_2\text{-PuO}_2$ powder are being evaluated. Sixteen capsules containing three pins each—one vipac, one sphere-pac, and one pellet—have been irradiated in the thermal flux of the BAWTR for from 8 hours to 55 days at linear heat ratings of 6 to 20 kW/ft. The irradiation history of these 16 capsules is described in BAW-3714-19.⁵

The post-irradiation examination of 27 fuel pins from nine previously disassembled capsules⁵ is progressing in B&W's hot cell facilities. Table 5-1 describes these 27 pins and presents their irradiation history.

All of the pins have been punctured under vacuum to collect the fission and sorbed gases released. The total quantity of gases present and the internal void volume of the fuel pins were measured. The gas composition is being determined by mass spectrometry, but the data are not complete. A complete analysis of these data will be presented in the next quarterly report.

Sixteen of the pins have been sectioned in the transverse direction, and specimens have been obtained for metallographic examination. These specimens have been mounted and ground, but they have not been polished or etched. Photomicrographs of the as-ground cross sections of sphere-pac and pellet pins operated at 6, 10, 15, and 20 kW/ft are shown in Figures 5-1 through 5-8. Central voids are apparent in all pins operated at 10, 15, and 20 kW/ft but not in those operated at 6 kW/ft.

Table 5-1. Description of Irradiated BAWTR
Fuel Pins Being Examined

Capsule No.	Fuel pin No.	Fuel form	Design linear heat rate, kW/ft	Actual exposure time, days
GA-2	C-24	Sphere-pac	15	15
	C-23	Vipac	15	15
	C-42	Pellet	15	15
GA-4	C-51	Sphere-pac	20	1
	C-49	Vipac	20	1
	C-100	Pellet	20	1
GA-6	C-134	Sphere-pac	20	11
	C-45	Vipac	20	11
	C-97	Pellet	20	11
GA-8 ^(a)	C-82	Sphere-pac	20	4
	C-43	Vipac	20	4
	C-99	Pellet	20	4
GA-10	C-46	Sphere-pac	20	3.5
	C-122	Sphere-pac ^(b)	20	3.5
	C-88	Pellet	20	3.5
GA-11	C-74	Sphere-pac	15	1
	C-139	Vipac	15	1
	C-15	Pellet	15	1
GA-13 ^(a)	C-80	Sphere-pac	15	4
	C-83	Vipac	15	4
	C-103	Pellet	15	4
GA-14 ^(a)	C-78	Sphere-pac	10	4
	C-34	Vipac	10	4
	C-102	Pellet	10	4
GA-15 ^(a)	C-69	Sphere-pac	6	4
	C-75	Vipac	6	4
	C-1	Pellet	6	4

(a) Capsules GA-8, GA-13, GA-14, and GA-15 were irradiated simultaneously.

(b) Fuel pin C-122 contained spherical sol-gel particles prepared by ORNL.

No significant difference in the size of the central void has been noted in any of the pins operated at 15 or 20 kW/ft for time periods ranging from 8 hours to 15 days. In addition, our present data indicate

that particulate fuels restructure so rapidly that their low initial thermal conductivity does not result in center melting when the linear heat rate is rapidly increased to as much as 20 kW/ft.

Coarse spheres can be seen next to the cladding in all the sphere-pac pins examined. However, in the pins exposed at 15 and 20 kW/ft, there is evidence that a portion of the diameters of the coarse spheres in the outer row has restructured.

Polishing and etching of the specimens is in progress. When this work is completed, a more detailed metallographic examination will be performed.

5.2. EBR-II Irradiation (E. N. Harbinson)

The primary objective of the EBR-II irradiations is to compare the operational characteristics of particulate fuels and pellets irradiated under identical conditions in a fast reactor. The effect of heat rate and burnup on the overall performance of sol-gel spherical particles and of angular particles and pellets prepared from coprecipitated UO_2 -20% PuO_2 powder will be compared and evaluated. Twenty-eight unencapsulated pins will be irradiated at 14 to 16 kW/ft in the EBR-II as part of a 37-pin subassembly to be shared with ORNL.

The 18 fuel pins required for the initial loading of the subassembly were shipped to the EBR-II site during this report period. The pins are being inspected by EBR-II personnel. A description of these pins and their fabrication data are given in BAW-3714-19.⁶

Fuel was loaded into 16 additional EBR-II pins (see section 4). Ten of these will be placed in the EBR-II bundle during scheduled outages. The remaining six pins will serve as spare or archive samples. All 16 pins will be xenon-tagged, welded, and inspected during the next quarter.

Figure 5-1. As-Ground Cross Section of Sphere-Pac Pin
C-69 (Irradiated 4 days at 6 kW/ft)

Figure 5-2. As-Ground Cross Section of Sphere-Pac Pin
C-78 (Irradiated 4 days at 10 kW/ft)

Figure 5-3. As-Ground Cross Section of Sphere-Pac Pin
C-74 (Irradiated 1 day at 15 kW/ft)

Figure 5-4. As-Ground Cross Section of Sphere-Pac Pin
C-82 (Irradiated 4 days at 20 kW/ft)

Figure 5-5. As-Ground Cross Section of Pellet Pin C-1
(Irradiated 4 days at 6 kW/ft)

Figure 5-6. As-Ground Cross Section of Pellet Pin C-102
(Irradiated 4 days at 10 kW/ft)

Figures 5-5 and 5-6

Figure 5-7. As-Ground Photomosaic of
Pellet Pin C-15 (Irradiated
1 day at 15 kW/ft)

Figure 5-8. As-Ground Cross Section of Pellet Pin C-88
(Irradiated 3.5 days at 20 kW/ft)

6. ANALYTICAL SUPPORT (R. H. Dodd)

6.1. Characterization of EBR-II Fuel (R. H. Dodd, R. J. Boden, A. H. Bremser)

Characterization of the fuel and insulator pellets prepared in FY-1971 for the EBR-II irradiation program was completed. The data are presented in Tables 6-1 and 6-2. Although the fuel is not as pure as that prepared in FY-1970,⁷ the results show that the impurity levels are within those specified for fast reactor fuel.

6.2. Ceramographic Examination of EBR-II Fuel (A. H. Bremser)

Polished transverse and longitudinal sections of both UO_2 pellet batches were examined ceramographically. There was no evidence of a second phase in the longitudinal section, but in the transverse section a metallic-appearing phase was present in the interior of the pellet. A typical photomicrograph is shown in Figure 6-1.

Ceramographic and alpha-autoradiographic examination of the fuel pellets and microspheres indicated that no second phase is present in these materials and that the plutonium is homogeneously distributed throughout. Typical photomicrographs are shown in Figures 6-2 and 6-3, and alpha autoradiographs are shown in Figures 6-4 and 6-5.

Table 6-1. FY-1971 EBR-II Fuel Characterization^(a)

Analysis	(U, Pu)O ₂ pellets	(U, Pu)O ₂ shards	(U, Pu)O ₂ coarse spheres	(U, Pu)O ₂ fine spheres	UO ₂ insulator pellets
Pu/(Pu+U), wt %	19.67 ± 0.07	19.69 ± 0.07	19.61 ± 0.13	19.88 ± 0.02	--
²³⁵ U content, wt %	92.20 ^(b)	92.20 ^(b)	92.20 ^(b)	92.20 ^(b)	0.7 ^(c)
O/M, atom ratio	1.990 ± 0.006	1.994 ± 0.003	1.992 ± 0.008	1.992 ± 0.005	2.035 ± 0.014
Moisture, ppm oxide	8.0 ± 5.1	44.8 ± 3.2	17.4 ± 4.8	55.0 ± 21.8	6.2 ± 3.0
Sorbed gas, cc/g	0.017	0.105	0.037 ^(d)	0.037 ^(d)	0.007
Density, % theor	--	93.5 ± 2.8 ^(e)	NM ^(f)	NM ^(f)	92.0 ± 3.4
Nitrogen, ppm metal	30.5 ± 3.5	40.8 ± 5.1	12.5 ± 3.8	9.5 ± 1.9	7.8 ± 0.5
Carbon, ppm metal	27.0 ± 3.7	39.5 ± 7.6	7.7 ± 2.5	19.9 ± 5.1	15.8 ± 4.1
Chloride, ppm metal	22.9 ± 2.9	20.6 ± 6.4	18.3 ± 5.7	25.2 ± 5.7	19.3 ± 8.9
Fluoride, ppm metal	2.7 ± 0.2	3.6 ± 0.6	3.3 ± 1.3	20.4 ± 7.0	5.5 ± 3.2

(a) Errors are for 95% confidence limits on the mean.

(b) Input values.

(c) Not determined, but should be less than normal.

(d) Sorbed gas analysis was performed on a mixture of 73 wt % coarse and 27 wt % fine microspheres in order to simulate the fuel composition in a pin.

(e) Value for pellets used to prepare shards.

(f) Not measured.

Table 6-2. Trace Metallic Impurities in FY-1971 EBR-II Fuel^(a)

Element	(U,Pu)O ₂ pellets	(U,Pu)O ₂ shards	(U,Pu)O ₂ coarse spheres	(U,Pu)O ₂ fine spheres	UO ₂ insulator pellets
Aluminum	230	42	150	180	<15
Beryllium	<1	<1	<1	<1	NM ^(b)
Bismuth	<1	<1	<1	<1	<10
Boron	1.9	1.5	<1	3	1.5
Cadmium	<1	<1	<1	<1	<1
Calcium	125	12	15	15	10
Chromium	42	25	10	100	50
Copper	35	6	80	10	<1
Iron	65	60	100	200	70
Lead	<10	<10	<10	<10	2
Lithium	<2	<2	<2	<2	NM ^(b)
Magnesium	11	6	6	6	3
Manganese	7	7	10	50	4
Molybdenum	<50	<50	<50	70	500
Nickel	<10	<10	50	100	<10
Silicon	125	50	100	300	14
Silver	<1	<1	<1	2	<0.5
Sodium	<10	<10	80	80	NM ^(b)
Tin	5	1	10	10	30
Titanium	<10	<10	<10	<10	NM ^(b)
Vanadium	<10	<10	<10	<10	<3
Zinc	<50	<50	<50	<50	<50

^(a) Values given are in ppm of metal and are the mean of four determinations.

^(b) Not measured.

Table 6-2

Figure 6-1. Photomicrograph of UO_2 Insulator Pellet

25X

Figure 6-2. Photomicrograph of EBR-II Fuel Pellet

25X

Figure 6-3. Photomicrograph of EBR-II Coarse Microspheres

100X

Figure 6-4. Alpha Autoradiograph of EBR-II Fuel Pellet

9X

Figure 6-5. Alpha Autoradiograph of EBR-II Coarse Microspheres

9X

7. REFERENCES

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