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TRITIUM DISTRIBUTION IN HIGH POWER ZIRCALOY FUEL ELEMENTS

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TRITIUM DISTRIBUTION IN HIGH POWER FUEL ELEMENTS

L. N. Grossman and J. O. Hegland

ABSTRACT

Tritium distributions have been determined for the fuel and cladding of two ~1/2 inch diameter Zircaloy-2-clad UO_2 fuel elements. Rod CP-206 obtained an average burnup of 12,000 MWd/Te at an average maximum linear power of 17.6 kW/ft; 75% of the tritium was released from the fuel. Rod 12FV obtained average burnup of 21,000 MWd/Te at an average maximum linear power of 31.4 kW/ft; 96% of the tritium was released from the fuel. A correlation between tritium release and power density was obtained. The results of neutron radiography, ceramographic examinations, gamma activity profiles, burnup and fission analyses, puncture gas analyses (including fission gas isotopes) and vacuum outgas analyses of the claddings are also given. Tritium was distributed nonuniformly in the cladding; hydrogen, from general corrosion was uniformly distributed. Thermal diffusion of tritium toward fuel-free regions was indicated in the cladding of Rod 12FV which was irradiated in a pressurized water loop with larger lineal thermal gradients along the cladding.

INTRODUCTION

Essentially all the radioactive products generated in the fuel of a nuclear power plant are transferred to the fuel reprocessing plant. For Zircaloy-clad fuels, the tritium produced from ternary fission¹ and other sources is expected to be divided between the fuel where it is born and the cladding where it is chemically combined.² Current reprocessing plants,³ as exemplified by the General Electric Company Midwest Fuel Recovery Plant (near Morris, Illinois), dispose of the cladding by permanent underwater burial in a stainless-steel-lined concrete container. The vault design permits retrieval of the scrap for off-site disposal at some future time, if desired. Alternate disposal of the cladding under consideration is concrete grouting of the entire vault.

During reprocessing,³ tritium which was received in the fuel ends up in the purified process water. Tritium is the only significant radioisotope in that water after purification. Since the amount of process water to be disposed of is relatively small, it has been considered practical to release it in the ventilation air after dilution through use of a 300-foot tall stack. There is flexibility in the hold-up time (up to several months) and release rate of the purified process water, thus allowing disposal during more favorable meteorological conditions.

The division between that tritium which is buried with the cladding and that which is retained in the purified process water is the division between cladding and fuel tritium. For low and moderate power-rated fuel rods, it has been shown⁴⁻⁶ that roughly 10% of the tritium inventory migrates to and is inerted in the cladding, while about 90% of the tritium remains in the oxide fuel. For fuel rods which receive higher power ratings (and higher fuel temperatures) than those previously reported,⁴⁻⁶ it was anticipated that a larger fraction of the tritium should be inerted in the cladding and destined for burial. Similarly, a smaller fraction of tritium should remain in the fuel and therefore destined for the process water during reprocessing.

The objective of the present study was to measure the partitioning of tritium between the fuel and the cladding (and the gas phase) of a high power-rated rod and a rod typical of current power reactor design.

FUEL ELEMENT HISTORY

Two fuel rods meeting the objectives of this study were selected. Rod 12FV of Assembly EX-12A had been irradiated at a burnup-average maximum linear power density of 31.4 kW/ft (peak at 49.7 kW/ft) in the General Electric Test Reactor. The average burnup obtained was 21,000 MWd/Te; the peak burnup was 23,500 MWd/Te. Rod CP-206 of Bundle D-50 was irradiated to a burnup-average maximum linear power density of 17.6 kW/ft (peak 22.3 kW/ft) in the Big Rock Point Reactor. Rod CP-206 obtained an average burnup of 12,000 MWd/Te with a peak of 15,500 MWd/Te.

ROD 12FV

Rod 12FV was built and irradiated as part of the High Performance UO_2 Program.⁷ The objective of the program, initiated in 1961, was to explore systematically the feasibility and limitations of UO_2 fuel elements operated for extended periods of time with central melting. Assembly EX-12A was the final assembly of this program and achieved sustained maximum heat flux levels in excess of one million Btu/h-ft². It was irradiated during GETR Cycles 54 through 58 in mid-1964. Rod 12FV had been irradiated to a maximum of about 11,000 MWd/Te as part of a previous assembly, EPW-12V during GETR Cycles 47 through 52 in late 1963. Both assemblies were irradiated in the Pressurized Water Loop (PWL) of the General Electric Test Reactor (GETR). The PWL is a general-purpose in-core loop with water as the primary coolant at about 350 °F, and a hydrogen over-pressure of 1200 psi. The peak thermal flux in the loop is about 3×10^{14} nv, which can produce heat fluxes up to 15×10^6 Btu/h-ft² (470 W/cm²) in the fuel elements.

Rod 12FV was made of Zircaloy-2 cladding and UO_2 fuel. The Zircaloy-2 chemical and mechanical analyses are shown in Table 1. The cladding was nominally 1.435 cm (0.565 in.) o.d. and 0.076 cm (0.030 in.) thick. The Zircaloy-2 was steam autoclaved at 750°F, resulting in a thin ($\sim 1/2 \mu\text{m}$) adherent black oxide layer on all surfaces. The fuel was vibratory-compacted UO_2 with the chemical analysis shown in Table 1. The as-received arc-fused UO_2 powder (from Spencer Chemical Co.) was heated for 4 h in wet hydrogen at 1650°C to assure an O-to-U ratio near 2.00. The powder was then seived and blended to give a particle size distribution for a high compaction efficiency. The fuel was loaded to 83% of theoretical density over a column length of 76 cm (29.9 in.). A total weight of 889 g UO_2 was used. The over-all fuel rod length was 99.1 cm (39.0 in.). The free volume of the rod was filled with helium at atmospheric pressure before the final seal weld was made on the end plug. Before sealing of the fuel element, it was vacuum outgassed at 480°C to remove moisture.

Rod 12FV received 5501 hours at full power and reached a final maximum burnup of 23,500 MWd/Te. The time-average maximum power rating over its irradiation history was therefore 31.6 kW/ft. The average power ratings during the final four GETR cycles were significantly lower than the earlier ratings and are given in Table 2. The burnup-average maximum power rating* over the full power history of Rod 12FV is 31.4 kW/ft.

ROD CP-206

Rod CP-206 was fabricated and irradiated as part of the Power Reactor High Performance UO_2 Program⁸ initiated in 1966. The Power Reactor Program was a continuation of the High Performance UO_2 Program⁷ begun in 1961. The objective of the Power Reactor Program was to operate UO_2 fuel elements with varying degrees of central melting at rated power to high burnup in a commercial power reactor core. The Power Reactor Program fuel elements were irradiated in the Big Rock Point (BRP) Reactor, near Charlevoix, Michigan, through the cooperation of Consumers Power Company, a sponsoring party to the Power Reactor Program.

Rod CP-206 was assembled as a component of Bundle D-50, an 8 × 8 bundle designed^{8a} to operate at "Intermediate" fuel performance with incipient central melting. The cladding was nominally 0.570 in. o.d. × 0.035 inch wall autoclaved Zircaloy-2; the chemical and mechanical analyses are given in Table 3. The fuel was pelletized UO_2 sintered to a nominal 95% of theoretical density in wet hydrogen at about 1650°C. The fuel pellets were dished on one end to provide a truncated cone about 5% of the pellet volume. The pellets were ground to provide a nominal diametral gap of 0.012 inch. The chemical analyses of the pellets are given in Table 3. The smeared fuel density was approximately 90% of theoretical density.

The fuel column length in Rod CP-206 was 67.28 inches. The over-all fuel element length was 74.75 inch. Since the fuel column of Rod CP-206 was shorter than the standard BRP fuel rod (because of the larger plenum necessitated by expected higher fission gas release), tantalum strips were placed on the plenum to serve as neutron absorbers to prevent flux peaking in the upper end of the adjacent standard fuel assemblies. The rod was back-filled with 1 atm He after fuel loading and before welding of the upper end plug.

* Burnup-average power rating is $\int \frac{(\text{kW/ft}) d (\text{b.u.})}{\text{b.u.}}$

TABLE 3. Pre-Irradiation Chemical and Mechanical Analyses* of Rod CP206

Cladding				Fuel	
				Impurity	ppm
Heat No. K-1013, 3943, and 370115 (typical)				Al	54
Corrosion Weight Gain: 17 mg/dm ² in 3 days at 750°F, 1500 psi steam				Bi	< 1
Chemical Analysis:				B	0.3
Alloy Elements		%		Ca	< 2
Sn		1.5		Cd	< 0.5
Fe		0.13		Cr	24
Cr		0.08		Co	< 2
Ni		0.06		Cu	< 1
Fe + Cr + N		0.27		Fe	30
Impurity		ppm		Pb	< 1
Al		30		Mg	2
B		0.2		Mn	< 2
C		130		Mo	< 3
Ca		< 10		Si	16
Cd		< 0.2		Ag	< 0.1
Cl		< 15		Na	< 15
Co		< 5		Sn	< 1
Impurity		ppm		V	< 11
Ca		< 10		Zn	< 10
Hf		70		Ni	6
Mg		< 10		Sb	< 2
Mn		< 10		N	< 5
Mo		< 10		C	4
Pb		< 20		F	4
Si		< 40		Cl	< 3
Impurity		ppm		U-235 % 5.05 ± 0.04	
Ti		< 20		O-to-U ratio 2.016	
V		< 5			
W		< 25			
U		< 1			
H		6			
N		30			
O		1200			
Room Temperature Longitudinal Mechanical Properties					
Ultimate Tensile Strength, ksi (kg/m ²)		92			
Yield Strength (0.2% offset), ksi (kg/m ²)		70			
Elongation in 2 inches, %		25			
Hardness (Rockwell B)		94			
Grain Size.		ASTM-11			
Flare TEst:		>15% of o.d. at 60 degrees			
Burst Test:		13,000 psi burst pressure			

*GEAP-10032, June, 1969

Rod CP-206 was located at an outside face of the 8 X 8 Bundle D-50 in position 6A. The bundle features are described further in Reference 8a. Rod CP-206 received 8544 full-power hours between March 1968 and April 1969. It reached a final maximum burnup of 15,600 MWd/Te. The average burnup of the fuel was 12,000 MWd/Te. The time-average maximum power rating was 16.1 kW/ft. The rod reached its peak maximum power rating for 384 h at 22.8 kW/ft at approximately 3000 MWd/Te. The burnup-average maximum power rating was 17.6 kW/ft with lower ratings received during the final approximate 4000 MWd/Te. The final approximate 1500 MWd/Te was accumulated at the lowest sustained maximum power rating, 11.4 kW/ft.

EXPERIMENTAL TECHNIQUES

Both rods were nondestructively examined by the following established techniques: visual, neutron radiography, gamma scan,⁹ and burnup analysis.¹⁰ For Rod CP-206 two gamma scans were performed, one which recorded all gamma energies (total gamma activity) and one which recorded only those gammas with energies between about 0.55 and 0.7 MeV. Twenty mil slits were used in both cases. Rod 12FV was scanned only in the single channel (0.55 - 0.7 MeV) mode with 62-mil slits. The larger slits were required because of the long decay time undergone by Rod 12FV. The two rods were then punctured and the internal gases collected for fission gas analyses^{9,11} and for

tritium analysis.¹² The rods were then sectioned into 3-inch segments (for CP-206) and 2-inch segments (for 12FV) according to the instructions given in Appendix A.

Cladding specimens for tritium analysis were cut into approximately 3/8-inch squares and cleaned in nitric acid (to remove UO_2) and hydrochloric acid (to remove crud from the BRP Rod CP-206) in the Remote Handling Laboratory. The specimens were rinsed in distilled water and dried for transfer to NL-Chemistry for vacuum outgassing. The cladding specimens were vacuum outgassed¹³ for 30 min. at 1300°C and the gas collected and its quantity measured. The outgas was then aliquoted and transferred to NL-Ceramic Development for conversion¹² of the HT and T_2 to water. The recovered tritiated water aliquot (typically approximately 0.1 ml) was then rinsed out into about 10 cc of scintillation fluid and analyzed by liquid scintillation counting.¹⁴ Typically one or two further dilutions with scintillation fluid were required to bring the specimen aliquot into optimum counting range. For a few specimens the balance of the vacuum outgas specimen was analyzed by gas chromatography for the species present.

Fuel specimens weighing typically 10 g were treated in a manner similar to that of Goode and Cox⁴ to recover the tritium. The recovery apparatus (two were used) are shown in Figure 1, and the instructions for recovery are given in Appendix B. Two recoveries per 8 h day could be made following those instructions.

Standard specimens were prepared for the cladding and fuel tritium analyses. An 8.83 g specimen of unirradiated UO_2 was dissolved according to the procedure given in Appendix B except 10 cc of certified activity water containing 2.6×10^7 dpm (1.3×10^{-3} μg) tritium was added to the dissolved fuel before distillation. Zirconium alloy foil containing tritium was prepared by diluting pure T_2 in H_2 and then hydriding the alloy until all gas was gettered as indicated by pressure drop. The alloy foil was then chopped and mixed to assure homogeneity of the standard. Unfortunately, a procedural error in standard preparation resulted in about 3 times as much T in the standard as planned and precluded use of the standard as a precision calibration. The standard foil was used to evaluate the reproducibility of the cladding tritium analysis chain.

Ceramographic examinations¹² of one longitudinal and one transverse section of each rod were performed. The longitudinal sections were ground to the axial midpoint and then polished. The transverse sections were etched and photomicrographed at 50X to determine the grain structure of the fuel. The cladding in all four specimens was etched and photomicrographed at 250X to show hydride precipitation.

EXPERIMENTAL RESULTS

NEUTRON RADIOGRAPHY

Neutron radiographs were obtained using two exposures, to bring out fuel and cladding details, respectively. Figure 2 shows the results for Rod CP-206 and Figure 3 shows the results for Rod 12FV. The figures are folded for convenience in presentation, neutron radiography was performed on the intact rod. The codes used in sectioning are shown to scale along each rod.

GAMMA ACTIVITY AND MAJOR RESULT TABULATION

The gamma activity profiles of Rod CP-206 are shown in Figure 4. The profile for Rod 12FV is shown in Figure 5. Figures 6 and 7 also show the gamma activity profiles along with the major experimental results. Figures 6 and 7 show the location of the various specimens for tritium, burnup, and ceramographic analyses. A summary of the results of the tritium, burnup, and total outgas are presented in tabular form on the figures.

GAS CHROMATOGRAPHY OF CLADDING OUTGAS

Table 4 shows the gas chromatographic analyses obtained on the six offgas specimens from the cladding of Rod CP-206. The gas chromatographic data are generally reliable to within a few percent.

TABLE 4. Gas Chromatographic Analysis of CP206 Cladding Offgas

Sample Location	Sample Weight (g)	Total Outgas ($\mu\text{l/g}$)	Volume Percent Offgas Composition						ppm H_2
			H_2	O_2	N_2	CH_4	CO	CO_2	
D	0.462	474	90.3	nd	nd	nd	11.4	nd	38
F	0.387	434	79.1	nd	nd	nd	20.4	nd	30
H	0.518	359	80.6	nd	nd	nd	17.8	nd	25
N	0.383	401	97.9	nd	nd	nd	3.2	nd	35
P	0.490	456	99.4	nd	nd	nd	2.0	nd	40
V	0.361	294	44.2	nd	nd	nd	55.1	nd	11

nd = not detected

CERAMOGRAPHIC ANALYSES

Figures 8 through 13 show the results of ceramographic analyses of the fuel and cladding. Rod 12FV showed evidence of fuel melting over about 70% of the rod diameter (Figures 10 and 11). Small metallic inclusions were seen throughout the outer regions (noncolumnar) of the fuel. The outermost region of the cladding, representing about 10% of the wall thickness, contained the highest concentration of hydride precipitates (Figure 13). The hydride content in this region of high concentration is estimated to be about 500 ppm hydrogen. Throughout most of the cladding, exclusive of the outside diameter region, the hydride content is estimated to be about 50 ppm (Figure 12).

Rod CP-206 shows no indication of central melting in Figures 9 or 10. The region of grain growth extends over about 55% the diameter (Figure 11). Small metallic inclusions could be seen in the central regions of the fuel and extended out over about 120 mils from the thermal center. The hydride precipitates were distributed uniformly throughout the cladding (Figures 12 and 13); the hydrogen content of the cladding is estimated at 50 ppm. A layer of crud 1-1/2 and 2 mils thick can be seen in Figure 13 with a thin oxide layer beneath it and adherent to the cladding.

BURNUP ANALYSES

Three burnup specimens were analyzed from each rod. Their positions are indicated in Figures 6 and 7. The results are given in Table 5 and 6 for Rods 12FV and CP-206, respectively. The Nd method¹⁰ for burnup analysis is estimated to be accurate to $\pm 2\%$; the heavy element method is less accurate.

The total gamma activity profile for Rod CP-206 (Figure 4) was used to scale the burnup values of Table 6 over the full length of the fuel column. Integration of the scaled values along the length resulted in an average burnup figure for the fuel. The burnup values from Table 7 were scaled to the previously published^{7c} total gamma activity profile for Rod 12FV, the scaled values were also averaged by integrations. The scaling and averaging procedures resulted in average burnup values of 12,000 MWd/te for Rod CP-206 and 21,000 MWd/Te for Rod 12FV.

TRITIUM CONTENT OF CLADDING

Table 7 gives the experimental data obtained from vacuum outgassing of the cladding specimens. One zirconium alloy blank (no tritium) and three alloy standards containing the same level of tritium were also outgassed. Aliquots of the total outgas were taken for conversion to water and counting for tritium. The conversion and counting data are also given in Table 7. Carryover of tritium in the conversion system gave a background of about 10^5 dpm tritium which shows up on the "Blank" and "Zr-no T" specimens in Table 7; no correction was made for this background.

TABLE 5. Burnup and Fissions for Rod 12FV

	<u>Segment H</u>	<u>Segment K</u>	<u>Segment O</u>
<u>Burnup (MWd/Te)</u>			
Nd Method	23,237	23,484	19,680
Heavy Element Method	23,255	22,797	19,457
<u>Fissions (at. %)</u>			
U-235	1.74	1.72	1.55
Pu-239	0.50	0.48	0.33
Pu-241	0.03	0.03	0.02
U-238	0.15	0.15	0.13
Total	2.42	2.37	2.03

TABLE 6. Burnup and Fissions for Rod CP206

	<u>Segment N</u>	<u>Segment P</u>	<u>Segment R</u>
<u>Burnup (MWd/Te)</u>			
Nd Method	15,561	15,530	13,166
Heavy Element Method	16,357	16,417	14,504
<u>Fissions (at. %)</u>			
U-235	1.45	1.45	1.30
Pu-239	0.13	0.14	0.105
Pu-241	0.004	0.004	0.003
U-238	0.12	0.12	0.11
Total	1.71	1.72	1.52

TRITIUM CONTENT OF FUEL

Table 8 gives the experimental data from dissolution, distillation, and counting of tritium in fuel samples. The "Blank" specimens were pellets of unirradiated UO_2 which were dissolved in the Radioactive Handling Laboratory apparatus and carried through the full analytical train. The standard sample showed recovery of $1.11 \times 10^{-3} \mu\text{g}$ tritium ($1.3 \times 10^{-3} \mu\text{g}$ tritium added). No corrections for recovery efficiency were added to the data in Table 8.

PUNCTURE GAS ANALYSES

The two rods were punctured before sectioning and the total gas content measured. Rod CP-206 had a total gas content of 168.7 ml STP and Rod 12FV contained 568.9 ml (STP). Of these totals, 110.5 ml and 64.55 ml were transferred out of the Remote Handling Operation for analyses. Five analyses were performed: (a) total gas composition by mass spectrometry, (b) isotopic composition of the fission gases Xe and Kr, (c) precision Xe-to-Xe + Kr ratio by mass spectrometry, (d) tritium content of the gas, and (e) total Xe + Kr by Kr-85 assay and the data from (b) and (c). The results of the first two analyses are presented in Tables 9 and 10.

The gas compositions in Table 9 are accurate to within $\pm 20\%$ for major species and order-of-magnitude values for minor species. No gases other than those shown in Table 9 were detected. The detection limit is about 0.01 volume percent.

TABLE 7. Experimental Data for Tritium in Cladding

Specimen	Weight (g)	Date Outgassed	Outgas (μ l-STP)	Outgas in Aliquot (%)	H-3 in Aliquot (dpm)	Tritium in Cladding (μ g/g)	Outgas from Cladding (μ l/g)
Zr-(no T)	0.237	5-11-71	27.2	41.6	2.2×10^5	$<1 \times 10^{-4}$	115
Std. A	0.328	12	45.5	38.5	4.9×10^6	1.8×10^{-3}	138
CP-206-N	0.383	14	157.6	30.1	3.2×10^7	1.3×10^{-2}	411
CP-206-D	0.462	14	222.5	34.7	3.3×10^7	9.4×10^{-3}	482
CP-206-P	0.490	14	227.6	34.1	8.2×10^7	2.3×10^{-2}	464
CP-206-V	0.361	15	120.6	33.0	2.6×10^7	1.0×10^{-2}	334
CP-206-F	0.387	15	182.2	35.8	3.5×10^7	1.2×10^{-2}	471
CP-206-H	0.518	15	200.2	35.8	2.8×10^7	7.1×10^{-3}	387
Std. B	0.509	18	115.3	33.1	1.0×10^7	2.9×10^{-3}	227
CP-206-J	0.534	19	195.6	40.8	7.0×10^7	1.5×10^{-2}	366
CP-206-C	0.585	19	291.6	41.2	5.0×10^7	9.6×10^{-3}	499
CP-206-W	0.479	20	178.3	39.6	3.0×10^7	7.5×10^{-3}	373
CP-206-R	0.586	20	240.8	40.5	7.1×10^7	1.4×10^{-2}	411
CP-206-A	0.582	20	201.4	39.4	3.1×10^7	6.1×10^{-3}	346
CP-206-T	0.321	20	123.8	39.1	3.3×10^7	1.1×10^{-2}	386
CP-206-B	0.327	20	553.2	33.3	4.4×10^6	1.9×10^{-3}	1693
12FV-H	0.330	21	1172.4	25.7	2.3×10^7	1.2×10^{-2}	3556
12FV-R	0.456	27	1675.8	18.8	5.5×10^7	2.9×10^{-2}	3607
CP-206-X	0.442	21	187.3	40.0	2.3×10^7	6.9×10^{-3}	424
12FV-Q	0.345	21	1117.7	27.4	2.5×10^7	1.2×10^{-2}	3243
12FV-K	0.366	25	827.9	35.4	1.2×10^7	4.3×10^{-3}	2263
12FV-O	0.345	25	1183.9	29.9	2.0×10^6	9.0×10^{-4}	3428
12FV-J	0.326	25	959.6	31.1	1.4×10^7	6.2×10^{-3}	2948
12FV-S	0.408	25	1208.0	31.0	3.1×10^7	1.1×10^{-2}	2959
Blank	—	26	2.5	56.3	4.1×10^4	—	—
12FV-L	0.373	26	1025.3	35.7	3.6×10^7	1.3×10^{-2}	2746
12FV-E	0.303	26	1132.7	27.1	2.2×10^7	1.2×10^{-2}	3742
12FV-P	0.356	27	1295.4	27.5	1.3×10^8	5.9×10^{-2}	3639
12FV-T	0.761	27	325.9	40.4	4.8×10^6	7.2×10^{-4}	428
12FV-N	0.334	27	1386.0	26.2	3.5×10^6	1.9×10^{-3}	4147
CP-206-V	0.461	27	206.6	39.3	5.7×10^7	1.5×10^{-2}	448
12FV-M	0.507	28	1724.3	22.6	7.2×10^6	2.9×10^{-3}	3399
CP-206-Y	0.740	28	200.2	38.3	2.6×10^7	4.3×10^{-3}	270
Std. C	0.861	6-2-71	70.4	36.5	1.1×10^7	1.6×10^{-3}	82

The reproducibilities of isotopic abundances reported in Table 10 were calculated at 95% confidence limits. The accuracy of any abundance figure is within $\pm 2\%$ of the reported value. For example, the isotopic abundance of Kr-83 in CP-206 lies between 13.27 and 13.81% with 13.54 being the most probable value; the reproducibility (tolerance) of the value 13.54 is ± 0.09 at the 95% confidence limit level.

The ratios of Xe-to-Xe + Kr were measured to be 0.884 and 0.905 for CP-206 and PWL-12V, respectively. Those values are accurate to within $\pm 2\%$ of the true sample values. The accuracy of either ratio relative to the other ratio is within $\pm 0.5\%$.

The total tritium contents of the gas samples analyzed were $\leq 1 \times 10^{-5} \mu\text{g}$ for both rods. The tritium contents on the total gas inside the rods were therefore $\leq 1 \times 10^{-3} \mu\text{g}$ and $\leq 5 \times 10^{-3} \mu\text{g}$ for Rods CP-206 and 12 FV, respectively.

The Kr-85 contents of the two gases were measured on aliquots of 0.175 torr-l and 0.0975 torr-l for CP-206 and 12FV respectively. The atoms of Kr-85 in the aliquots were measured to an accuracy of $\pm 10\%$ as 3.7×10^{16} and 1.6×10^{16} . Those data, together with the isotopic data in Table 10 and the precision measured Xe-to-Xe + Kr ratios correspond to total Xe + Kr releases of 155 ml (STP) and 570 ml (STP) in Rods CP-206 and 12FV, respectively. The accuracy of those total release figures is estimated to be within $\pm 15\%$ of the true values.

TABLE 8. Experimental Data for Tritium in Fuel

Specimen	Weight (g)	Tritium in Aliquot (dpm/ml)	Tritium in Fuel ($\mu\text{g/g}$)
CP-206-A	7.85	3.15×10^6	1.11×10^{-3}
CP-206-C	13.08	9.86×10^6	2.28×10^{-3}
CP-206-D	8.10	7.34×10^6	2.73×10^{-3}
CP-206-F	8.79	7.27×10^6	2.57×10^{-3}
CP-206-H	11.50	1.32×10^6	3.48×10^{-3}
CP-206-J	9.33	1.18×10^7	3.62×10^{-3}
CP-206-N	10.01	1.18×10^7	3.54×10^{-3}
CP-206-P	9.96	1.35×10^7	3.96×10^{-3}
CP-206-R	7.47	7.96×10^7	3.14×10^{-3}
CP-206-T	8.06	6.36×10^6	2.31×10^{-3}
CP-206-V-2	7.93	7.27×10^6	2.46×10^{-3}
CP-206-V-3	10.86	2.88×10^6	6.72×10^{-4}
Blank-1	6.23	1.77×10^4	7.70×10^{-6}
Blank-2	9.02	3.97×10^4	1.19×10^{-5}
Blank-3	8.03	1.04×10^4	3.51×10^{-6}
Standard	8.83	3.52×10^5	1.26×10^{-4}
12FV-B	10.75	1.39×10^6	3.54×10^{-4}
12FV-E	9.79	2.20×10^6	6.25×10^{-4}
12FV-H	9.95	2.12×10^6	6.16×10^{-4}
12FV-J	9.50	2.20×10^6	6.34×10^{-4}
12FV-K	10.17	1.97×10^6	5.74×10^{-4}
12FV-L	10.22	2.38×10^6	6.25×10^{-4}
12FV-O	6.30	8.21×10^6	3.54×10^{-4}

TABLE 9 Gas Compositions by Mass Spectrometry

	Volume Percent	
	CP-206	12FV
Xe	82	86
Kr	11	9
He	7	3
N ₂	n d	3
CO ₂	n d	0.03
H ₂ O	<0.02	<0.02

TABLE 10 Isotopic Abundances of Xe and Kr

	CP 206	12FV
Kr-83	13.54 ± 0.09	13.58 ± 0.12
Kr-84	28.06 ± 0.16	28.75 ± 0.15
Kr-85	6.12 ± 0.09	4.86 ± 0.07
Kr-86	52.29 ± 0.20	52.81 ± 0.16
Xe-131	10.94 ± 0.11	10.52 ± 0.06
Xe-132	17.77 ± 0.11	17.59 ± 0.05
Xe-134	29.94 ± 0.10	29.15 ± 0.07
Xe-136	41.35 ± 0.16	42.75 ± 0.10

DISCUSSION OF RESULTS

TRITIUM PARTIONING

The experimental data for tritium in the fuel and cladding (Tables 7 and 8) have been plotted in Figures 14 and 15. The figures show the linear concentration of tritium (in $\mu\text{g}\cdot\text{cm}^{-1}$) along the two rods. The figures show a well-defined trend along the length of the fuel in both cases. The distribution of tritium in the cladding of Rod CP-206 is scattered, but reasonably well-behaved, but the tritium distribution in the cladding of 12FV is erratic, especially in the region at the top of the fuel column and extending into the plenum.

Integration of the area under the curves in Figure 14 results in measured total tritium values of $6.0\ \mu\text{g}$ in the fuel and $5.3\ \mu\text{g}$ in the cladding, for a total of $11.3\ \mu\text{g}$. Based on a yield of 1×10^{-4} atoms tritium per fission, and a tritium half-life of 12.3 h, the calculated yield from fission is about $24.2\ \mu\text{g}$. The average scatter in the measured tritium of the cladding sections (Figure 14) is about $\pm 30\%$ (compared to about $\pm 10\%$ for the fuel) which suggests that the unanalyzed cladding may contain some unaccounted-for tritium.

The cladding tritium values measured for 12FV (Figure 15) are too scattered to perform an accurate integration along the cladding for total tritium. A rough figure of $2\ \mu\text{g}$ total tritium in the cladding may be estimated from those data. The fuel tritium data (Figure 15) are very consistent and may be integrated to yield a good figure of $0.48\ \mu\text{g}$ tritium in the fuel. Again using a fission yield of 1×10^{-4} and a half-life of 12.3 h for tritium, the calculated yield from 12FV is found to be $13.1\ \mu\text{g}$. The erratic distribution of tritium in the cladding again suggests that the unaccounted-for tritium may be in the cladding.

The average tritium retained in fuel for rods CP-206 and 12FV are 26% and 3.7% respectively. Percent retained tritium from the fuel was calculated based on the measured fuel tritium contents and the calculated tritium production values. This method was chosen because of the unaccounted-for tritium. The tritium values measured for the fuel are believed accurate to within $\pm 10\%$ and are consistent to within the same tolerance (Figures 14 and 15). The accuracy is based on the 91% recovered standard sample (Table 8) and on a standard which was 98% recovered in an identical apparatus.¹⁵

The tritium released from the fuel may be plotted against average linear power rating as shown in Figure 16. The data of Goode and Cox⁴ and Melehan⁵ are also shown. All rods were Zircaloy-2 clad UO_2 , about 1/2 inch diameter. The low power elements received about 32,000 MWd/Te and 11,400 MWd/Te average burnup. A clear trend can be seen in Figure 16, that tritium release from the fuel is strongly dependent on linear power rating. The power rating dependence is clearly much stronger than any burnup dependence for these specimens.

LONGITUDINAL TRITIUM MIGRATION

The longitudinal distribution of tritium in the fuel (Figures 14 and 15) follow the burnup profiles (Figures 4 and 5). For CP-206, the tritium in the cladding also follows the burnup profile, with very little tritium in the fuel-free areas (*e.g.*, the plenum). For 12FV, the longitudinal tritium distribution in the cladding (Figure 15) shows definite signs of thermal migration.^{16,17} The difference in longitudinal tritium migration behavior between the two rods can be interpreted based on the experimental conditions. Rod CP-206 was irradiated in a boiling water reactor, where the longitudinal temperature extremes do not exceed about 50°C . Rod 12FV was irradiated on the Pressurized Water Loop of GETR; in this case, the plenum was about 150°C cooler than the active part of the element. In addition, a sharp temperature gradient existed adjacent to the plenum, where the fuel was operating very hot as can be seen in Figure 3. Since the thermal flux is dependent¹⁷ on both temperature and temperature gradient, the observed tritium peak in the plenum and near the hottest fuel of 12FV is qualitatively in agreement with theory. The very low tritium levels observed in the plenum and end-cap regions of the CP-206 cladding is the result of small temperature gradients in the fuel cladding of a BWR.

HYDROGEN PICKUP

The hydrogen levels in the cladding of 12FV are about an order of magnitude higher than those in CP-206 as shown by the total outgas data (Figure 6). Typically 20-40 ppm H₂ were found in CP-206 and 100-200 ppm H₂ were found in 12FV, according to the total outgas and gas chromatographic analyses. The excess hydride was present principally at the cladding o.d. in 12FV (Figure 13). These observations can also be explained on the difference between the two irradiation facilities. The PWL was pressurized with hydrogen, while the Big Point Reactor pressure is maintained by the steam from boiling. Increased hydrogen pickup during aqueous corrosion has been shown¹⁸ to be dependent on hydrogen overpressure. The accumulation of hydrogen at the o.d. is a well-documented¹⁶ manifestation of hydrogen thermal diffusion. The correlation of total outgas with tritium content on Rod-12FV (Figure 6) indicates that the hydrogen was taken into the cladding during corrosion (dependence on power level) and distributed longitudinally by thermal diffusion (dependence on cladding temperature gradient).

FISSION GAS RELEASE

The puncture gas analyses (Table 9) show that fission product Xe and Kr were the main gases present. Based on an assumed value of 0.33 atoms stable Xe + Kr per fission, the total fission gas productions are 0.70 and 0.48 moles for CP-206 and 12FV, respectively. The fission gas recovered from the rods during puncturing was 0.16 and 0.57 moles respectively, or 23% and 119% of the assumed productions.

CONCLUSIONS

The amount of tritium destined for the process water during reprocessing is strongly dependent on the linear power rating of the fuel element. Figure 17 shows the tritium contents of fuel as functions of burnup and maximum linear power rating. The solid lines in Figure 17 are based on the results of this study and earlier studies. The dashed lines are interpolations based on the curve in Figure 16. The solid lines are estimated to be accurate to about $\pm 10\%$ for fuel cycle times of about 3 years at 20,000 MWd/Te.

Table 11 summarizes the experimental results of this study for total tritium in the fuel rods. Calculated total tritium yields from ternary fission are 24.2 and 13.1 μg for Rods CP-206 and 12FV, respectively. The fuel data in Table 11 are accurate to within $\pm 10\%$. The cladding data are less accurate (especially for 12FV) for two reasons: (1) the absolute recovery efficiency for cladding tritium is unknown due to an error (magnitude unknown) in standard preparation; and (2) the sampling procedure for cladding specimens may have resulted in missing some localized areas of high tritium concentration; that such areas exist is indicated by the variability in tritium contents between cladding specimens (Figures 15 and 16). Similar local variability of tritium in cladding was observed by Melehan.⁵ A more accurate assessment of total cladding tritium by the technique used will require more cladding specimens and a repeated standard recovery calibration of the analytical chain.

TABLE 11. Experimental Tritium Partitioning Summary

	Tritium (μg)			Total Found
	Fuel	Cladding	Gas	
CP-206	6.0	5.3	$<10^{-3}$	11.3
12FV	0.48	~ 2	$<5 \times 10^{-3}$	2.5

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FIGURE 1. TWIN DISTILLATION APPARATUS FOR RECOVERY OF TRITIUM FROM FUEL

FIGURE 2a. NEUTRON RADIOGRAPHY OF ROD CP206

FIGURE 2b. NEUTRON RADIOGRAPHY OF ROD CP206

FIGURE 3. NEUTRON RADIOGRAPHY OF ROD 12FV

Top

Bottom

4a. Total Gamma Energy — Full Scale: 3240 cps (20 mV)

4b. $0.55 < E < 0.7 \text{ MeV}$ — Full Scale: 1032 cps (20 mV)

Scanned: 4/17/71
 Slit Size: 0.020 inch
 Rod Speed: 1 in./min

FIGURE 4. GAMMA ACTIVITY PROFILES FOR CP206

Date Scanned: 4/26/71
Slit Size: 0.062 inch
Rod Speed: 1 in./min
Gamma Energies: 0.55 to 0.7 MeV
Full Scale: 5800 cpm (20 nV)

FIGURE 5. GAMMA ACTIVITY PROFILE FOR 12FV

CLADDING ³ H μg/g (Cladding)	FUEL ³ H μg/g (Fuel)	TOTAL OUTGAS μl/g (Cladding)	BURNUP MWd/Te
1.9 X 10 ⁻³	3.54 X 10 ⁻⁴	1693.30	
1.2 X 10 ⁻²	6.25 X 10 ⁻⁴	3742.12	
1.2 X 10 ⁻²	6.16 X 10 ⁻⁴	3555.83	2.32 X 10 ⁴
6.2 X 10 ⁻³	6.34 X 10 ⁻⁴	2948.17	
4.3 X 10 ⁻³	5.74 X 10 ⁻⁴	2262.69	2.35 X 10 ⁴
1.3 X 10 ⁻²	6.25 X 10 ⁻⁴	2745.96	
2.9 X 10 ⁻³		3399.05	
1.9 X 10 ⁻³		4147.09	1.97 X 10 ⁴
9 X 10 ⁻⁴	3.54 X 10 ⁻⁴	3427.68	
5.9 X 10 ⁻²		3638.67	
1.2 X 10 ⁻²		3243.44	
2.9 X 10 ⁻²		3606.88	
1.1 X 10 ⁻²		2958.63	
7.2 X 10 ⁻⁴		428.46

FUEL AND CLADDING SAMPLE

CLADDING SAMPLE ONLY

BURNUP SAMPLE

CERAMOGRAPHIC SAMPLE (LONGITUDINAL)

CERAMOGRAPHIC SAMPLE (TRANSVERSE)

FIGURE 7. BURNUP, TRITIUM, AND HYDROGEN EXPERIMENTAL VALUES
ALONG ROD 12FV

FIGURE 8. LONGITUDINAL MACROSECTION FROM ROD 12FV, TOP OF FREEZING VOID (SEGMENT O)

FIGURE 9. LONGITUDINAL MACROSECTION FROM ROD CP206, SEGMENT R

As Ground

Rod 12FV, Segment H

~5X

As Ground

Rod CP206, Segment P

~5X

FIGURE 10. TRANSVERSE SECTION MACROGRAPHS

FIGURE 11. FUEL MICROSTRUCTURES

Etched

250X

Rod CP206 Longitudinal Section

Etched

250X

Rod CP206 Transverse Section

Etched

250X

Rod 12FV Longitudinal Section

Etched

250X

Rod 12FV Transverse Section

FIGURE 12. TYPICAL CLADDING MICROSTRUCTURES SHOWING HYDRIDES

Etched

Rod CP206 Longitudinal Section

250X

Etched

Rod CP206 Transverse Section

250X

Etched

Rod 12FV Longitudinal Section

250X

Etched

Rod 12FV Transverse Section

250X

FIGURE 13. MICROSTRUCTURES SHOWING HYDRIDE AT CLADDING OUTSIDE DIAMETER AND CRUD LAYERS

FIGURE 14. TRITIUM DISTRIBUTION IN ROD CP206

FIGURE 15. TRITIUM DISTRIBUTION IN ROD 12FV

FIGURE 16 TRITIUM RELEASE VS LINEAR POWER

FIGURE 17. TRITIUM IN FUEL VS. BURNUP

APPENDIX A

INSTRUCTIONS FOR SECTIONING OF FUEL ELEMENTS

GENERAL

1. Dimensions referred to are taken from the neutron radiographs.
2. Tritium samples are to be 3/8 inch long sections.
3. Burnup samples are to be 3/8 inch long sections of fuel
4. Ceramographic samples are to be 3/4 inch long sections of fuel and cladding. (One longitudinal and one transverse from each rod as noted.)
5. Tritium samples are *not* to include a pellet interface.
6. Tritium samples are to be taken from the top end of segments if possible. (Note any necessary change in sample locations)
7. Tritium samples include fuel and cladding in fuel column area Cladding only in plenum area. (Unless otherwise noted.)
8. Burnup samples are to be taken next to tritium samples

ROD CP206 (BRP)

1. Define zero inches as flat end of bottom end plug First pellet is approximately 1.19 inches up from zero. Fuel column length is approximately 66 3/8 inches
2. Starting from zero reference, cut rod into 3 inch segments Identify each segment with a letter, starting with A at the bottom and ending with Y at the top
3. Identify orientation of each segment
4. Using ground rules described above, remove samples from segments according to Table A-1

TABLE A-1. Sample Locations for Rod CP206

<u>Segment</u>	<u>Tritium Samples</u>	<u>Burnup Samples</u>	<u>Ceramographic Samples</u>
A	X		
B			
C	X		
D	X		
E			
F	X		
G			
H	X		
I			
J	X		
K			
L			
M			
N	X	X	
O			
P	X	X	X transverse (next to burnup)
Q			
R	X	X	X longitudinal (include pellet interface)
S			
T	X		
U			
V	X (1/4 inch from top)		
W	X		
X	X		
Y	X (end plug disk)		

MAINTAIN IDENTIFICATION OF ALL SAMPLES

ROD 12FV (PWL)

1. Define zero inches as flat end of bottom end plug. First pellet is approximately 1 inch up from zero. Fuel column length is approximately 30-5/8 inches. (Length includes 5/8 inch of powder in bottom end plug.)
2. Starting from zero reference, cut rod into 1-inch segments. Identify each segment with a letter, starting with A at the bottom and ending with T at the top.
3. Identify orientation of each segment.
4. Using ground rules described above, remove samples from the segments according to Table A-2.

NOTE

Segments M and N should be originally cut as one 4-inch segment. Carefully split the 4-inch segment longitudinally trying to maintain fuel in position. Call for inspection of 4-inch longitudinal sections at this point.

MAINTAIN IDENTIFICATION OF ALL SAMPLES

TABLE A-2. Specimen Locations for Rod 12FV

<u>Segment</u>	<u>Tritium Samples</u>	<u>Burnup Samples</u>	<u>Ceramographic Samples</u>
A			
B	X		
C			
D			
E	X		
F			
G			
H	X	X	X transverse (next to burnup)
I			
J	X		
K	X	X	
L	X		
M	X (clad only)		
N	X (clad only)		
O	X	X (in molten zone)	X longitudinal (top of molten zone, Center of O)
P	X (clad only)		
Q	X		
R	X		
S	X		
T	X (end plug disk)		

MAINTAIN IDENTITY OF ALL SAMPLES

APPENDIX B

INSTRUCTIONS FOR RECOVERY OF TRITIUM FROM FUEL

A summary of events is as follows: A fuel sample of known weight is placed in a receiver and dissolved in acid with low heat under reflux. When the reaction is complete and heat input is increased to force a portion of the dissolver solution up the reflux column as vapor and out the condenser as liquid. This liquid is essentially water. The water is then sampled and transferred to the radiochemistry laboratory for tritium analysis; the remaining solutions are stored. A detailed work sheet guide will be followed with entries of weights, volumes, and other pertinent data carefully recorded. Constant attention will be required to maintain sample identity.

1. Locate sample (note fuel and cladding are in same vial), remove cladding, weigh vial. Place fuel in dissolver pot and reweigh vial. Return cladding to vial. *Maintain identity.*
2. Load receiver with 7 to 9 cc of distilled water. Note actual volume and place on equipment; check for completeness of setup (top stopcock closed).
3. Measure out approximately 50 cc of dissolver solution 8M HNO_3 (note actual volume accurately) and add to pot.
4. Dissolve fuel with low heat Variac at 40 — reduce if rapid reaction is indicated. Check security by observing bubbles in receiver. (No heat was required except on the unirradiated specimen.)
5. As fuel dissolution nears completion, start helium flow to pre-cool the condenser and line, set to 90 CFH.
6. After dissolution, apply insulation blanket to the reflux column and raise the heat. Set Variac to 100 and distill over a volume of 25 ml. *Watch condensation line carefully* add or subtract heat to prevent vapor passage.
7. Turn off heat and open top stopcock to prevent back up of distillate.
8. Stop helium flow when temperature is down to near ambient cell temperature.
9. Remove receiver and sample about 5 ml in a polysealed glass bottle. Measure aliquot accurately. Transfer to NL - Analytical Chemistry.
10. Store distillate in polyethylene bottle and store dissolver solution in a second bottle.
11. Rinse both the pot and receiver with distilled water.
12. Fill pot with approximately 50 ml of distilled water and run heat at 115 V to drive over about 10 to 15 ml.

TRITIUM DISTILLATION WORK SHEET

Sample Identity _____ Vial _____

Equipment No. _____ Run No. _____ Date _____

Sample Weight: Gross _____ grams

Tare _____ grams

Net _____ grams

Volume of H₂O in receiver _____ mlVolume of 8M HNO₃ + dissolver solution solution _____ ml

Volume of distillate: Receiver total _____ ml

Less initial H₂O _____ ml

Distillate volume _____ ml

Volume of distillate and initial water transferred to J. P. Peterson: _____ ml

Transfer Date: _____

Volume of distillate and water stored in-cell _____ ml

Remarks:VoltageTime

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