

The Dissolution and Characterization of Aluminum Clad Oxide Fuel

by

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OXIDE FUEL (U)

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SUMMARY

Laboratory dissolution studies of aluminum clad mixed oxide fuel rods have been conducted using two different F-Canyon decladding and dissolving flowsheets. During the first phase of the experimental program, materials from three different color coded fuel rods were dissolved in caustic and nitric acid solutions. The final phase of the laboratory program involved the dissolution and characterization of materials from three cracked pellet fuel rods using a selected caustic/ nitric acid flowsheet.

Laboratory results obtained from the initial dissolution studies identified several inconsistencies and potential problem issues with the behavior of materials from the color coded fuel rods. Based on these findings and influenced by the difficulties introduced by using the RCRA listed mercury during processing, the flowsheet selected for dissolving these aluminum clad fuel rods in F-Canyon dissolvers was the two-step caustic decladding/ nitric acid dissolution flowsheet.

The final phase of the experimental program involved testing materials from three cracked pellet fuel rods using the selected flowsheet. Again all aluminum fuel rod components dissolved during the decladding step. However, some uranium and plutonium bearing solids remained with the caustic decladding solution which could be sent to waste. The quantities of uranium and plutonium expected to remain with the caustic solutions are small.

Fluoride ions will need to be present in the nitric acid dissolver solution to dissolve all solids. At 0.05 molar fluoride concentration, no plutonium bearing solids remained in the product solutions.

INTRODUCTION

Aluminum clad fuel rods containing uranium and plutonium oxide pellets are currently stored in F-Area vaults. As part of the SRS program to deinventory and stabilize vault materials, six selected fuel rods were sent to SRTC for flowsheet development and materials characterization studies. Selected for the first phase of the laboratory program were three different color coded fuel rods expected to contain uranium and plutonium mixed oxide fuel. The final three fuel rods examined contained pellets which X-Ray analysis had indicated could contain numerous cracks.

Two standard dissolution flowsheets previously used in F-Canyon dissolvers were selected to conduct the initial series of laboratory dissolution experiments. Materials from the three color coded fuel rods were dissolved using a two-step caustic decladding/nitric acid dissolution flowsheet and a nitric acid/mercury codissolution flowsheet.

Results from the initial dissolution and solids characterization work resulted in selection of the two-step caustic decladding/nitric acid flowsheet for dissolving these aluminum clad fuel rods in F-Canyon dissolvers. The final series of laboratory experiments evaluated three cracked pellet fuel rods to verify their dissolution behavior with the selected flowsheet.

EXPERIMENTAL PROGRAM

The objectives of the experimental program were to evaluate the dissolution behavior of each fuel rod type under different flowsheet conditions and to characterize the pellets and any insoluble materials that remained after each dissolution step. Initially three fuel rods containing three different types of DU/Pu mixed oxide fuel pellets were sent to SRTC from the F-Area storage vault. Each fuel rod had a different color code representing three different plutonium isotopic compositions. The aluminum cladding, end pieces, and oxide pellets from each of three color coded rods were dissolved in caustic and nitric acid solutions. Any insoluble material left after each dissolution step was analyzed for elemental composition. Product solutions were analyzed for plutonium concentrations and for total and free acid concentrations.

Following completion of the initial dissolution and characterization studies for the three color coded fuel rods in caustic and nitric acid solutions, three additional fuel rods were examined using the preferred caustic, nitric acid/fluoride flowsheet. The objectives of this final phase of the experimental program were to evaluate the dissolution behavior of "cracked" fuel pellets, characterize residues, and to optimize the dissolving conditions for the caustic, nitric acid/fluoride flowsheet for F-Canyon dissolvers. Selection of the cracked pellet fuel rods sent to SRTC was based on an earlier x-ray examination. Two were yellow coded 54 inch fuel rods and the third was a 17 inch, brown color coded fuel rod.

Dissolution Studies

The first series of laboratory dissolution experiments was conducted by adding complete sections of aluminum cladding, end pieces, and pellets to the NaOH/NaNO₃ caustic decladding solutions. All aluminum components were expected to dissolve in the caustic solutions, leaving the pellets behind. Any brown powder or black pellet fragments which collected in the caustic solution were weighed and characterized. A summary of the caustic dissolving conditions for each of the color coded fuel rods is presented in Table I, FUEL ROD EXPERIMENTAL CAUSTIC DISSOLVING (DECLADDING) CONDITIONS.

Following decladding, the pellets which remained were removed from the caustic solution and dissolved in nitric acid and nitric acid-KF solutions. The initial attempt was to completely dissolve the pellets without the use of fluoride to minimize corrosion. Weight measurements and solids characterizations were performed on insoluble materials left after the dissolution of pellets. A summary of the dissolving conditions for materials from each of the three color coded rods in nitric acid solutions is presented in Table II, FUEL ROD EXPERIMENTAL NITRIC ACID DISSOLVING CONDITIONS.

The next series of laboratory dissolution experiments was conducted by adding complete sections of cladding, end pieces, and pellets to nitric acid/mercury solutions. Again the need for fluoride ions to accelerate the dissolution rates for aluminum components and pellets was evaluated. All aluminum materials and complete pellet dissolution was expected to occur in the combined, one-step, nitric acid/mercury solution. Weight measurements and materials characterizations were made on any solids left after each dissolution experiment. A summary of the dissolving conditions for each of the three color coded fuel rods in nitric acid/mercury solutions is presented in Table III, FUEL ROD EXPERIMENTAL NITRIC ACID/MERCURY DISSOLVING CONDITIONS.

The final series of laboratory dissolution experiments began following a visual examination of each of the pellets as they were removed from the cracked pellet fuel rods. Complete sections of aluminum cladding, end pieces, and pellets were added to the NaOH/NaNO₃ caustic decladding solutions at the recommended one to ten NaOH to NaNO₃ mass ratio and at or above the recommended one to six aluminum to NaOH mass ratio. Following the decladding operation, pellets were removed, and brown solids and small pellet fragments filtered from the caustic solution. A summary of the caustic dissolving condition for each of the three cracked pellet fuel rods is presented in Table VII, X-RAY FUEL ROD FINAL CAUSTIC DECLADDING CONDITIONS.

Following decladding, removal of the pellets, and filtration of brown solids and pellet fragments, two separate dissolution studies were conducted in 10.1 molar nitric acid/0.05 molar fluoride solutions. After the dissolution of filtered solids and the intact pellets was completed, plutonium concentrations in each product solution was measured to determine the amount of plutonium in pellet chips which could be sent to waste with the caustic decladding solution and the weight percent plutonium in the pellets. A summary of the dissolving conditions for each of the three cracked pellet fuel rods in nitric acid/fluoride solutions is presented in Table VIII, X-RAY FUEL ROD FINAL NITRIC ACID/FLUORIDE DISSOLVING CONDITIONS.

Experimental Results

Although no analyses were performed directly on the caustic solutions after each decladding step, the weight measurements and materials characterization of brown solids and possible pellet fragments remaining in the caustic solutions provided useful data concerning potential plutonium and other material losses in the caustic waste stream. A summary of the dissolving results for each of the three color coded fuel rods in caustic decladding solutions is presented in Table IV, FUEL ROD EXPERIMENTAL CAUSTIC DISSOLVING (DECLADDING) RESULTS.

Following dissolution of decladded pellets in nitric acid solutions, both the solutions and any remaining solids were analyzed. The plutonium concentrations in product solutions were measured to establish the weight percent plutonium in the mixed oxide fuel pellets from each color coded rod. The total and free acid concentrations helped to establish dissolution rates as a function of acid concentration and to determine the moles of acid consumed per gram of material dissolved. The analysis of remaining solids determined if all plutonium had dissolved, identified the composition of insoluble residues which could be sent to waste, and helped identify reasons for the differences in the dissolution behavior between the three types of mixed oxide fuel rods. A summary of the dissolving results of pellets from the three color coded fuel rods in nitric acid solutions is presented in Table V, FUEL ROD EXPERIMENTAL NITRIC ACID DISSOLVING RESULTS.

The reasons for analyzing dissolver solutions and insoluble residues were similar for the nitric acid/mercury product solutions as they were for the previous nitric acid solutions without mercury. One apparent difference observed was the moles of acid consumed per gram of aluminum components and pellets dissolved. A summary of the dissolving results of aluminum materials and pellets from the three color coded rods in nitric acid/mercury solutions is presented in Table VI, FUEL ROD EXPERIMENTAL NITRIC ACID/MERCURY DISSOLVING RESULTS.

As expected, all aluminum components dissolved in the final caustic decladding solution. Although few pellet fragments containing plutonium were detected previously during the initial caustic decladding studies of the three color coded fuel rods (TABLE IV), pellet fragments containing plutonium were found in all caustic decladding solutions from each of the cracked pellets fuel rods. Only the presence of brown powder was consistent in all caustic decladding solutions. A summary of the caustic decladding results for each of the three cracked pellet fuel rods is presented in Table IX, X-RAY FUEL ROD FINAL CAUSTIC DELCADDING RESULTS.

The filtered brown solids and pellet fragments from the cracked fuel pellets and the intact pellets were dissolved in different nitric acid/fluoride solutions. Plutonium concentrations were analyzed in both product solutions to determine the amount of plutonium in the pellet fragments which could be sent to waste and to determine the weight present plutonium in these cracked fuel pellets. A summary of the dissolving of solids and pellets from the three cracked pellet fuel rods in nitric acid/fluoride solutions is presented in Table X, X-RAY FUEL ROD FINAL NITRIC ACID/FLUORIDE DISSOLVING RESULTS.

DISCUSSION

All experimental test results and the dissolving conditions evaluated during the initial phase of the laboratory test program are presented in TABLES I through VI. During these laboratory experiments, the characterization of solution and solids and the dissolution behavior of materials from the three color coded fuel rods were examined.

Tables I and IV present the decladding conditions used and the experimental results obtained from the decladding step. The information obtained from these experiments established that all aluminum components completely dissolved, that the uranium brown solids remaining in caustic solutions did not contain plutonium, that plutonium bearing pellet chips are generated during caustic decladding, and that other impurities identified in the brown solids are from cross contamination.

Tables II and V present the nitric acid dissolving conditions used and the experimental results obtained from the acid dissolution of materials left undissolved after decladding. The information obtained from these experiments established that fluoride ions are required to completely dissolve all pellet material from the red fuel rod, that UNH crystals formed after dissolving 100 grams of pellets in 250 ml of dissolver solution, and that pellet dissolution times are relatively constant between 3-10 molar nitric acid concentrations.

Tables III and VI present the nitric acid/mercury dissolving conditions used and the experimental results obtained from the codissolution of aluminum and pellet materials. The information obtained from these experiments established that the male end pieces will not dissolve in nitric acid solutions and reconfirmed that fluoride ions need to be present to completely dissolve all red rod pellet materials in nitric acid solutions.

The final series of laboratory experiments evaluated the dissolution behavior of three cracked pellet fuel rods using only the two step caustic decladding/ nitric dissolution flowsheet. Selection of this dissolving flowsheet for use in the F-Canyon dissolver was based primarily on the difficulty in dissolving the aluminum male end pieces in any of the nitric acid solutions. Two other changes made to the caustic decladding/nitric acid dissolution flowsheet for the final verification experiments were decladding with the correct aluminum to NaOH to NaNO₃ mass ratio and dissolving with 0.05 molar fluoride ions in the nitric acid solutions.

Tables VII and IX present the final decladding conditions used and the experimental results obtained during decladding of the cracked pellet fuel rods. The conditions tested in the laboratory attempted to duplicate actual F-Canyon dissolver conditions and the results obtained are those expected when this batch of aluminum clad fuel rods is processed. The information obtained from these experiments established that over two hours at boiling will be required to completely dissolve all aluminum end pieces in the caustic solution, that from two to five weight percent of the uranium will remain with the decladding solution as brown powder, and that about two weight percent of the pellets could remain with the decladding solutions as small pellet fragments.

Tables VIII and X present the final nitric acid/fluoride dissolving conditions and the experimental results obtained during dissolving of pellets, pellet chips, and brown powder. The information obtained from these experiments established that all pellet materials could require up to four hours to completely dissolve, and that about two percent of the plutonium could be sent to waste in the small pellet fragments.

CONCLUSIONS

The basic two-step caustic decladding/nitric acid dissolving flowsheet, previously used in F-Canyon dissolvers, can be used to process these aluminum clad oxide fuel pellets. The significant differences from past dissolvings will be the uranium brown powder and the mixed oxide pellet fragments that are expected to remain with the decladding solution, and the fluoride ions that need to be present in the nitric acid solutions to completely dissolve all pellet materials.

Other conclusions were reached as a result of investigations and experiments performed on the fuel rods before and while the laboratory program was being conducted.

- (1) There are no uranium only fuel rods.
- (2) X-Ray examination of fuel rods indicated cracked pellets could be present in some of the fuel rods. However, physical examination of the "cracked" pellets found them to retain their structural integrity during "drop tests" and during decladding. There appeared to be no significant differences in dissolution behavior between the x-ray cracked pellets and the pellets from the first three fuel rods, which had not been x-rayed.
- (3) An oily substance was found on the surface of pellets removed from one of the cracked pellet fuel rods. Gas chromatographic and infrared analysis found only an unknown grease or oily substance containing just carbon-hydrogen bonds.
- (4) Varying amounts of a fine brown powder were on the surface of all pellets from all fuel rods. This material contained only uranium, no plutonium.
- (5) Some of the smaller black pellet chips could remain with the caustic decladding solution. About two percent of the plutonium could be removed from the dissolver by this mechanism.
- (6) The normal flowsheet operating procedures F-Canyon has used to declad and dissolve aluminum clad fuel should be followed during the processing of these aluminum clad fuel rods. The decladding period should be at least three hours at boiling due to the difficulty in dissolving the male end pieces. Also, the aluminum to NaOH to NaNO_3 mass ratios should be those which have been successfully used in the past.

The main difference identified during the laboratory testing of the nitric acid dissolving conditions was the presence of 0.05 molar fluoride to assure complete dissolution of all oxide pellets. The nitric acid dissolving period should be at least four hours in the 85-90C temperature range. However, before processing begins, a calculation of the total mass of uranium to be dissolved will need to be made to assure no post precipitation of uranyl nitrate (UNH) occurs. The bases for this calculation are the amount of plutonium in the fuel rods and the experimental result which found UNH precipitated after 100 grams of pellets had dissolved in 250 ml of nitric acid solution.

TABLE I, FUEL ROD EXPERIMENTAL CAUSTIC DISSOLVING(DECLADDING) CONDITIONS

Expt #	Comp, Dissolver Solution.	Vol. Dissolver Solution. (ml)	Type Material Added	Wt Material Added (g)	Dissolving Time (min)	Dissolving Temp (degC)
Green-1	50 wt.% NaOH	100	pellets	31.16	18	85-90
	25 wt.% NaNO3	200	Al clad, Al end			
Green-7	NaOH	100	male	1.93	45	80-93
	NaNO3	200	end			
Red-2	NaOH	100	Al clad	3.95	134	64-82
	NaNO3	200	end(fem)			
Red-4	NaOH	100	Al tube	0.70	45	82-85
	NaNO3	200				
Red-12A	NaOH	295	male	1.79	33	85-98
	NaNO3	total	end			
Red-13	NaOH	255	Al	0.90	4	boiling (105)
	NaNO3	total	tube			
Brown-1	NaOH	100	pellets,	46.14	27	boiling (105)
	NaNO3	200	Al clad, end(fem)			
Brown-7	NaOH	205	male	1.71	105	84-95
	NaNO3	total	end			

TABLE II, FUEL ROD EXPERIMENTAL NITRIC ACID DISSOLVING CONDITIONS

Expt. #	Comp. Dissol Soln. (M)	Vol. Dissol Soln. (ml)	Type Matl Added	Wt. Matl Added (g)	Dissol Time (min)	Dissol Temp. (degC)	Vol. Final Soln. (ml)	Final Tot/Free H+ Conc. (M)
Green-2	10.1 HNO ₃	150	1.5 pellets	26.51	98	82-85	150	10.5/ 8.5
Green-3	8.5 HNO ₃	150	2.5 pellets	45.31	163	70-72	140	9.8/ 4.5
Red-3	10.1 HNO ₃	300	pellets, tube, end(fem)	43.97	175	85-92	300	10.6/ 9.75
Red-8	9.75 HNO ₃	300	pellet	20.40	178	78-88	ND	ND
Red-8A	9.75 HNO ₃ 0.08 F-	300	black solids	ND	12	66	300	10.3/ 8.9
Red-9	8.9 HNO ₃ 0.08 F-	300	pellet	20.80	173	86-90	300	10.1/ 8.4
Red-12	10.1 HNO ₃ 0.08 F-	300	pellet	17.0	180	75-90	300	10.4/ 9.4
Red-15	10.1 HNO ₃ 0.08 F-	300	pellet	20.89	100	81-92	300	10.6/ 9.8
Brown-2	10.1 HNO ₃	300	pellet	19.57	173	85-90	285	10.6/ 7.1
Brown-2A	7.1 HNO ₃	285	pellet, chips	20.85 1.36	102	82-90	275	10.6/ 5.9
Brown-5	5.9 HNO ₃	275	pellet	17.83	104	88-90	255	10.7/ 5.2
Brown-6	5.2 HNO ₃	255	pellet	20.68	108	76-89	250	ND
Brown-8	ND	250	pellet	20.64	111	89-90	250	ND

TABLE III. FUEL ROD EXPERIMENTAL NITRIC ACID/MERCURY DISSOLVING CONDITIONS

Expt. #	Comp. Dissol Soln. (M)	Vol. Dissol Soln. (ml)	Type Mater Added	Wt. Mater Added (gr)	Dissol Time (min)	Dissol Temp. (degC)	Vol. Final Soln. (ml)	Final Tot/Free H+ Conc. (M)
Green-4 (CB-1)	7.4 H+ 56ppmHg yes F-	150	Al tube	3.66	37	78-103	150	7.7/ 3.1
Green-5 (CB-1)	3.1 H+ 56ppmHg yes F-	150	two pellets	35.81	130	80-90	125	7.6/ 1.5
Green-6 (CB-1)	4.05 H+ ?ppmHg yes F-	215	Al tube, male end	5.48	119	80-88	215	7.6/ 1.75
Green-8 (CB-1)	4.4 H+ ?ppmHg yes F-	315	Al tube	2.12	29	90-97	315	8.4/ 3.66
Green-9 (CB-1)	3.66 H+ ?ppmHg yes F-	315	two pellets	35.34	168	82-85	315	8.7/ 2.36
Red-1	10.1 H+ 56ppmHg no F-	300	pellets, Al tube, end(fem)	43.96	195	84-86	275	10.5/ 8.7
Red-5 (CB-1)	4.2 H+ ?ppmHg yes F-	255	one pellet	ND	240	84-87	260	ND
Red-6 (CB-1)	4.2 H+ ?ppmHg yes F-	260	Al tube	0.65	130	80-88	ND	ND
Red-7	8.7 H+ 112ppmHg no F-	275	Al tube	0.80	204	82-91	ND	ND
Red-7A	8.7 H+ 112ppmHg 0.08 F-	275	Al tube	0.27	46	85-90	250	10.5/ 8.25

TABLE III, continued

Expt. #	Comp. Dissol Soln. (M)	Vol. Dissol Soln. (ml)	Type Mater Added	Wt. Mater Added (gr)	Dissol Time (min)	Dissol Temp. (degC)	Vol. Final Soln. (ml)	Final Tot/Free H+ Conc. (M)
Red-11	10.1 H+ 56ppmF- 0.08 F-	300	pellets, male end	46.48	170	77-88	295	9.7/
Brown-3 (no F-)	10.1 H+ 56ppmHg	300	pellets, Al tube	42.44	128	83-90	290	10.0/ 4.1
Brown-4 (no F-)	4.1 H+ 56ppmHg	290	pellets, clad, male end	46.75	252	89-91	280	9.2/ 2.3

TABLE IV. FUEL ROD EXPERIMENTAL CAUSTIC (DECLADDING) RESULTS

Expt. #	Type Material Dissolved	Type Material Left	Comp. Material Left	Grams Material Left	Wt.% Material Left
Green-1	all Al clad, end	pellet, brown powder	PuO ₂ / UO ₂ U	17.91	ND
Green-7	all end piece	none	NA	NA	NA
Red-2	all Al, end	brown powder	U,Fe	1.31	2.98
Red-4	all Al Tube	brown powder	U,Na, Mg,Al	0.97	2.20
Red-12A	all Al end	none	NA	NA	NA
Red-13	all Al tube	none	NA	NA	NA
Brown-1	all Al clad, end	pellets, frag., brown powder	NA	40.92	NA
			Fe,Cr,Ni	1.3	2.8
			U,Mo,Al Ca,Ti,Cu	1.08	2.3
Brown-7	all Al end	brown powder	Mg,Al,U Ti,Fe,Na	0.76	NA

TABLE V. FUEL ROD EXPERIMENTAL NITRIC ACID DISSOLVING RESULTS

Expt. #	Type Material Dissolved	Type Material Left	Comp. Material Left	Wt.% Pu/ Pellet	Moles H+ Used/gr Dissolved
Green-2	all pellets	none	NA	0.40	9E-3
Green-3	all pellets	none	NA	0.37	14E-3
Red-3	pellets	black solids, all Al	U,Pu* Al,Si	low* (0.25)	ND
Red-8	pellet	black solids	ND	ND	ND
Red-8A	all solids	none	NA	0.27	12E-3
Red-9	all pellet	none	NA	0.34	7E-3
Red-12	all pellet	trace solids	U	0.63	14E-3
Red-15	all pellet	trace solids	U,Si,Al Fe,Cr,Ni	0.37	ND
Brown-2	all pellet	none	NA	0.29	5E-3
Brown-2A	all chips, pellet	none	NA	0.30	17E-3
Brown-5	all pellet	none	NA	0.455	4E-3
Brown-6	all pellet	none	NA	0.383	ND
Brown-8	all pellet	none	UNH	0.365	ND

TABLE VI. FUEL ROD EXPERIMENTAL NITRIC ACID/MERCURY DISSOLVING RESULTS

Expt. #	Type Mater Dissol	Type Mater Left	Comp. Mater Left	Grams Mater Left	Wt.% Pu/ Pellet	Moles H+ Used/gr Dissol
Green-4 (CB-1)	all Al	none	NA	NA	NA	176E-3
Green-5 (CB-1)	all pellets	none	NA	NA	NA	8E-3
Green-6 (CB-1)	all Al tube	male end	Al	1.93	NA	139E-3
Green-8 (CB-1)	all Al tube	none	NA	NA	NA	111E-3
Green-9 (CB-1)	all pellets	none	NA	NA	NA	13E-3
Red-1 (no F-)	very little	chips, tube end(fem)	Pu,Al,Si Fe,Cr	ND	0.317	17E-3
Red-5 (CB-1)	some pellet	black solids	U,Si Al	2.18	ND	ND
Red-6 (CB-1)	all tube	none	NA	NA	NA	ND
Red-7 (no F-)	very slowly	some Al tube	Al	0.27	NA	NA
Red-7A (yes F-)	all Al	none	NA	NA	NA	44E-3
Red-11 (yes F-)	all tube pellets	male end	Al	1.79	0.37	18E-3
Brown-3 (no F-)	all tube pellets	2 specks	Si,Al,U Fe,Mg,Cl	0.10	0.31	43E-3
Brown-4 (no F-)	pellets tube	male end, O-ring	Al	1.70	0.33	14E-3

TABLE VII, X-RAY FUEL ROD FINAL CAUSTIC DECLADDING CONDITIONS

Expt. #	Comp. Dissolver Solution	Mass Dissolver Solution (grams)	Type Mater. Added	Weight Mater. Added (grams)	Dissolv Time (min.)	Dissolv. Temp. (degC)
X-Ray-1 (brown)	25wt.% NaNO3 50wt.% NaOH	235 22.3	pellets, Al tube	55.36 2.72	13	76-102
X-Ray-2 (brown)	25wt.% NaNO3 50wt.% NaOH	235 22.1	pellets, Al tube	62.11 2.72	12	83-107
X-Ray-5 (brown)	25wt.% NaNO3 50wt.% NaOH	235 22.7	pellets, Al tube, male end	59.44 6.11	100	104-106
X-Ray-6 (yellow)	25wt.% NaNO3 50wt.% NaOH	241 22.1	pellets, Al tube, female end	58.27 5.52	82	104
X-Ray-8 (yellow)	25wt.% NaNO3 50wt.% NaOH	235 22.7	pellets, Al tube, male end	40.95 ND	127	104
X-Ray-9 (yellow2)	25wt.% NaNO3 50wt.% NaOH	235 23.4	pellets, Al tube, male end	55.15 6.44	141	105

TABLE VIII. X-RAY FUEL ROD FINAL NITRIC ACID/FLUORIDE DISSOLVING CONDITIONS

Expt. #	Comp. Dissolver Solution	Volume Dissolver Solution (ml)	Type Material Added	Weight Material Added (grams)	Dissolving Time (min.)	Dissolving Temp. (degC)
X-Ray-3 (brown)	10.1M HNO ₃ 0.05M HF	100	chips, brown powder	total 3.32	80	86
X-Ray-4 (brown)	10.1M HNO ₃ 0.05M HF	300	three pellets	55.34	230	82-85
X-Ray-7 (yellow)	10.1M HNO ₃ 0.05M HF	200	three pellets	55.71	120	82-90
X-Ray-7a (yellow)	10.1M HNO ₃ 0.05M HF	100	chips, brown powder	total 3.45	125	83-85
X-Ray-10 (yellow2)	10.1M HNO ₃ 0.05M HF	100	chips, brown powder	total 4.13	103	84-87
X-Ray-11 (yellow2)	10.1M HNO ₃ 0.05M HF	300	three pellets	55.15	208	84-86

TABLE IX. X-RAY FUEL ROD FINAL CAUSTIC DECLADDING RESULTS

Expt. #	Type Material Dissolved	Type Material Left	Composition Material Left	Weight % Material Left	Grams Pu in Material
X-Ray-1 (brown)	all Al mater.	pellets, chips, brown powder	Pu/U oxides, Pu/U oxides U,Na	NA NA 0.87	ND ND none
X-Ray-2 (brown)	all Al mater	pellets, chips, brown powder	Pu/U oxides Pu/U oxides U,Na	NA NA 1.37	0.185 2.7E-3 none
X-Ray-5 (brown)	all Al mater.	pellets, chips, brown powder	Pu/U oxides Pu/U oxides U,Na,Al	NA NA ND	ND ND none
X-Ray-6 (yellow)	all Al mater.	pellets, chips, brown powder	Pu/U oxides Pu/U oxides U,Na,Al	NA NA 1.95	0.179 2.9E-3 none
X-Ray-8 (yellow)	all Al mater.	pellets, chips, brown powder	Pu/U oxides Pu/U oxides U,Na,Al	NA NA ND	ND ND none
X-Ray-9 (yellow2)	all Al mater.	pellets, chips, brown powder	Pu/U oxides Pu/U oxides U,Na,Al	NA NA 5.5	0.20 1.4E-3 none

TABLE X. X-RAY FUEL ROD FINAL NITRIC ACID/FLUORIDE DISSOLVING RESULTS

Expt. #	Type Material Dissolved	Type Material Left	Weight % Material per pellet	Percent of Pu in Material	Weight % Pu in Material
X-Ray-3 (brown)	all chips, all brown powder	none	1.46	1.46	0.33
			1.37	none	none
X-Ray-4 (brown)	all pellets	none	100	98.5	0.33
X-Ray-7 (yellow)	all pellets	none	100	98.5	0.31
X-Ray-7A (yellow)	all chips all brown powder	none	1.5	1.5	0.31
			1.95	none	none
X-Ray-10 (yellow2)	all chips all brown powder	none	0.71	0.71	0.36
			5.5	none	none
X-Ray-11 (yellow2)	all pellets	none	100	99.3	0.36

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