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SUBJECT: Demonstration of the Zirflex Process on Irradiated PWR Blanket Fuel

TO: F. L. Culler

FROM: T. A. Gens, G. A. West, L. M. Ferris

ABSTRACT

Fifteen PWR blanket fuel specimens, varying in burnup from 80 to 1100 Mwd/T, were declad with boiling 6 M NH_4F-1.0 M NH_4NO_3 before the UO_2 core was dissolved in 10 M HNO_3. Uranium and plutonium losses to the decladding solution were less than 0.2% in nearly all runs. While these losses are higher than those obtained in laboratory experiments with unirradiated fuel, they are of the same order of magnitude as those obtained in the testing of the hot cell equipment with unirradiated fuel.

In two runs where 9 M HF was used as the decladding reagent, the uranium and plutonium losses averaged 1.0 and 0.4%, respectively.

DISCLAIMER

1.0 INTRODUCTION

Two alternative processes for the dissolution of PWR blanket fuel, normal UO_2 pellets encased in Zircaloy-2, have received intensive laboratory development with unirradiated fuel specimens. In the Zirflex Process,[1,4] the Zircaloy-2 cladding is removed with boiling 6 M NH_4F-1.0 M NH_4NO_3 before the UO_2 is dissolved in 8-10 M HNO_3. Boiling 9 M hydrofluoric acid is used as the decladding reagent in the other process.[2] Seventeen laboratory-scale experiments with irradiated fuel specimens were performed to evaluate the effect of radiation on these processes. Fuel specimen irradiation levels, as determined by chemical analysis, varied from 80 to 1100 Mwd/T. The effect of burnup and decladding time on the uranium and plutonium losses to the decladding solution and to nitric-acid insoluble precipitates, if formed were of particular interest. The experimental procedures followed closely those outlined in the laboratory work.[1,2]

Further Zirflex hot-cell experiments are planned using a modified laboratory flowsheet.[3] A series of successive dissolutions in which the zirconium heel is allowed to accumulate will be required to provide a better indication of losses to be expected under production conditions.

The authors are pleased to acknowledge the experimental work done by J. C. Rose, J. Beams, R. L. Boles, G. E. Woodall, and J. F. Land. Analyses were provided by the groups of C. Lamb, G. R. Wilson, and W. R. Laing of the ORNL Analytical Chemistry Division.

2.0 EXPERIMENTAL PROCEDURE AND RESULTS

The uranium and plutonium losses to the 6 M NH_4F-1 M NH_4NO_3 solutions, while higher than found in laboratory experiments,[1] were acceptable, being well under 0.2% in nearly all runs. Furthermore, the losses were essentially the same as those obtained with unirradiated fuel specimens in preliminary experiments in the hot-cell equipment (Sec. 2.2) showing that irradiation had no measurable effect on the losses.

The uranium and plutonium losses to the 9 M HF solutions averaged 1.0 and 0.4%, respectively, in the two runs performed with irradiated specimens. Laboratory experiments with unirradiated samples also showed uranium losses of about 1%.[2]

Neither burnup nor length of contact time between the core and decladding solution produced any systematic variation in the losses to the decladding solution.

2.1 Procedure

All dissolutions of irradiated fuel specimens were performed in the Carpenter 20 apparatus shown schematically in Fig. 1 and described in detail in Sec. 3.0. Experimental procedures followed closely those outlined in the laboratory work[1,2] (Figs. 2 and 3) except that solutions were removed from

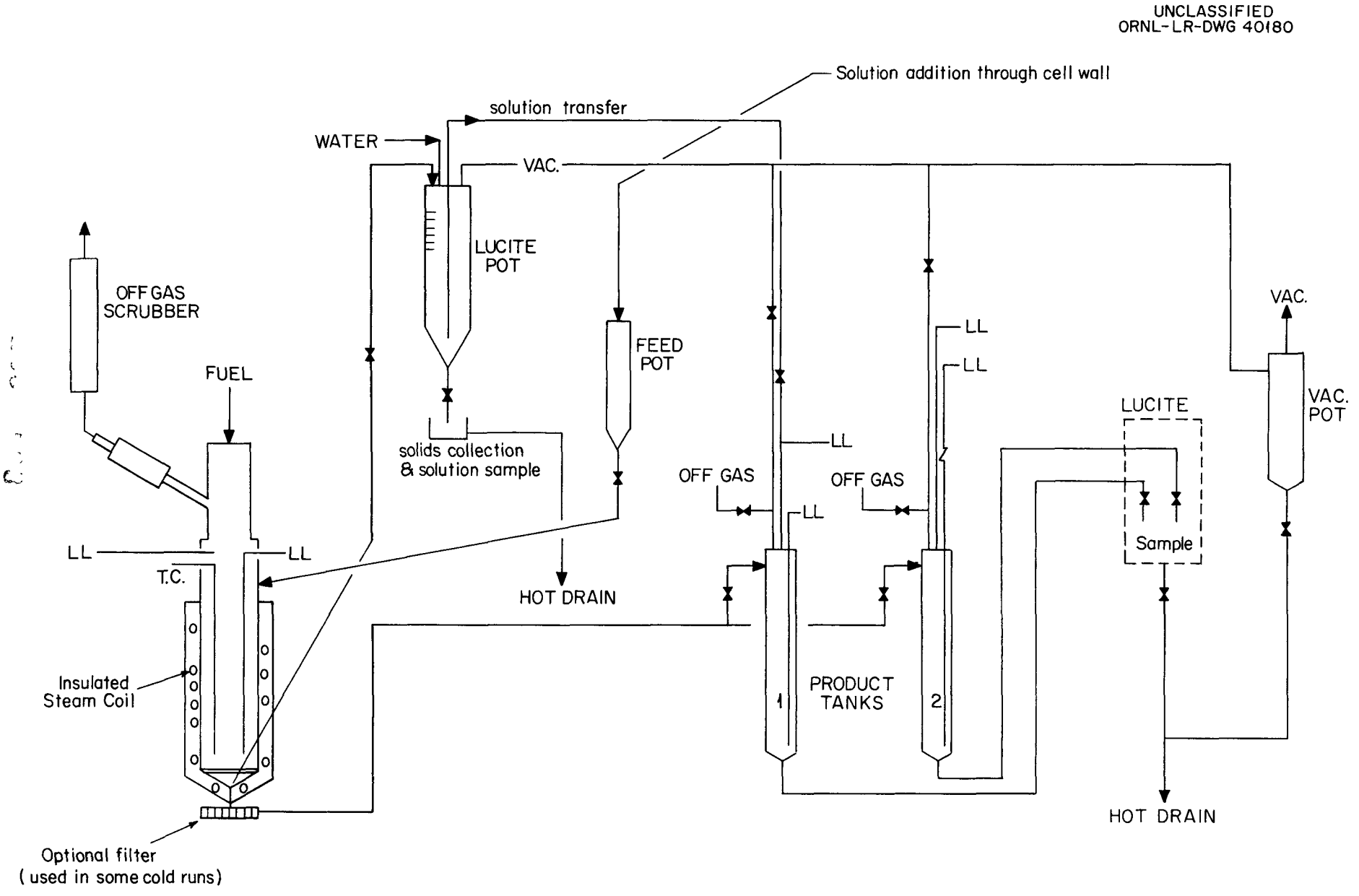

Fig. 1. Schematic Diagram of PWR Dissolving Equipment.

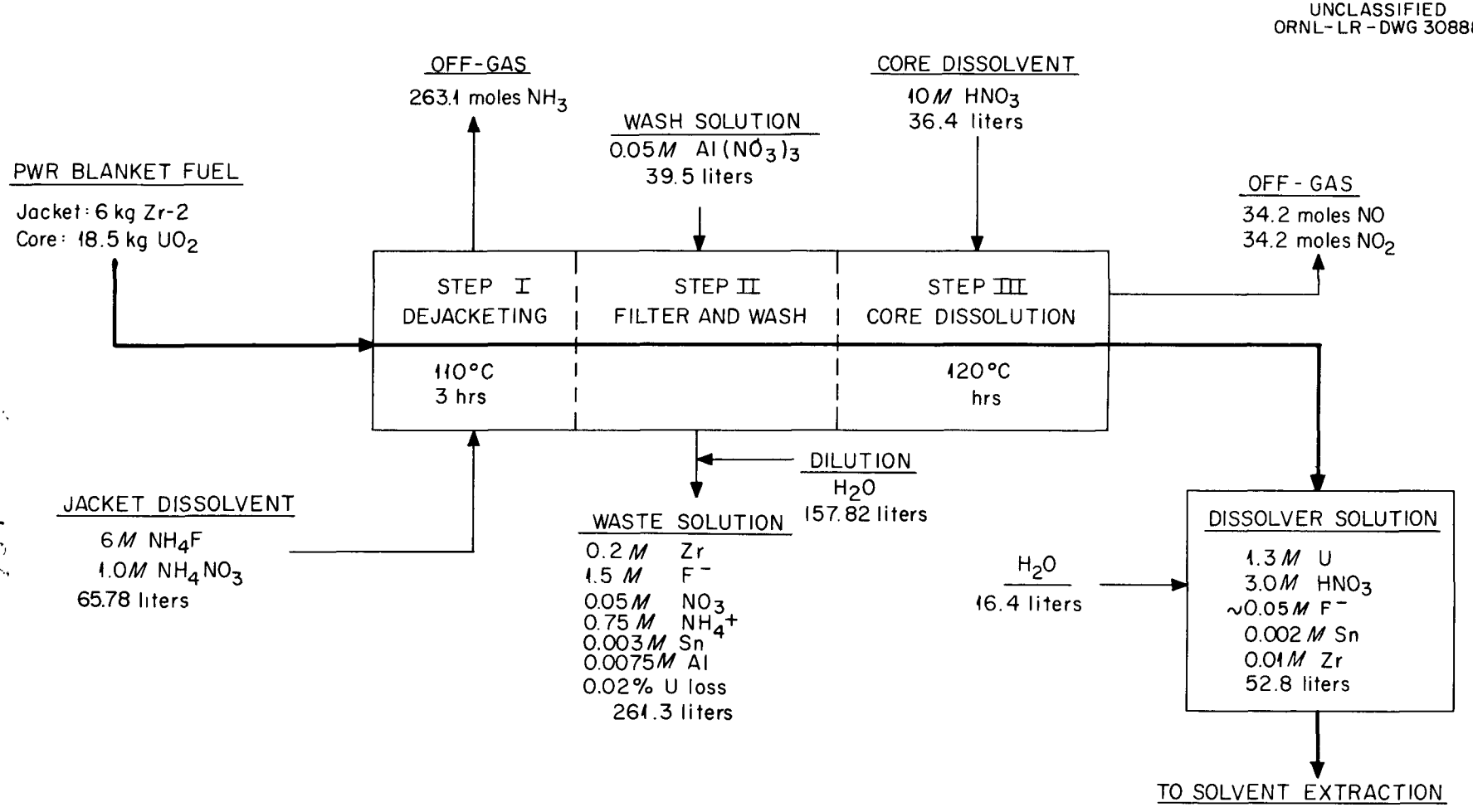

Fig. 2. Tentative Flowsheet for Dejacketing Zircaloy-2 Jacketed Fuels with Aqueous NH_4F.

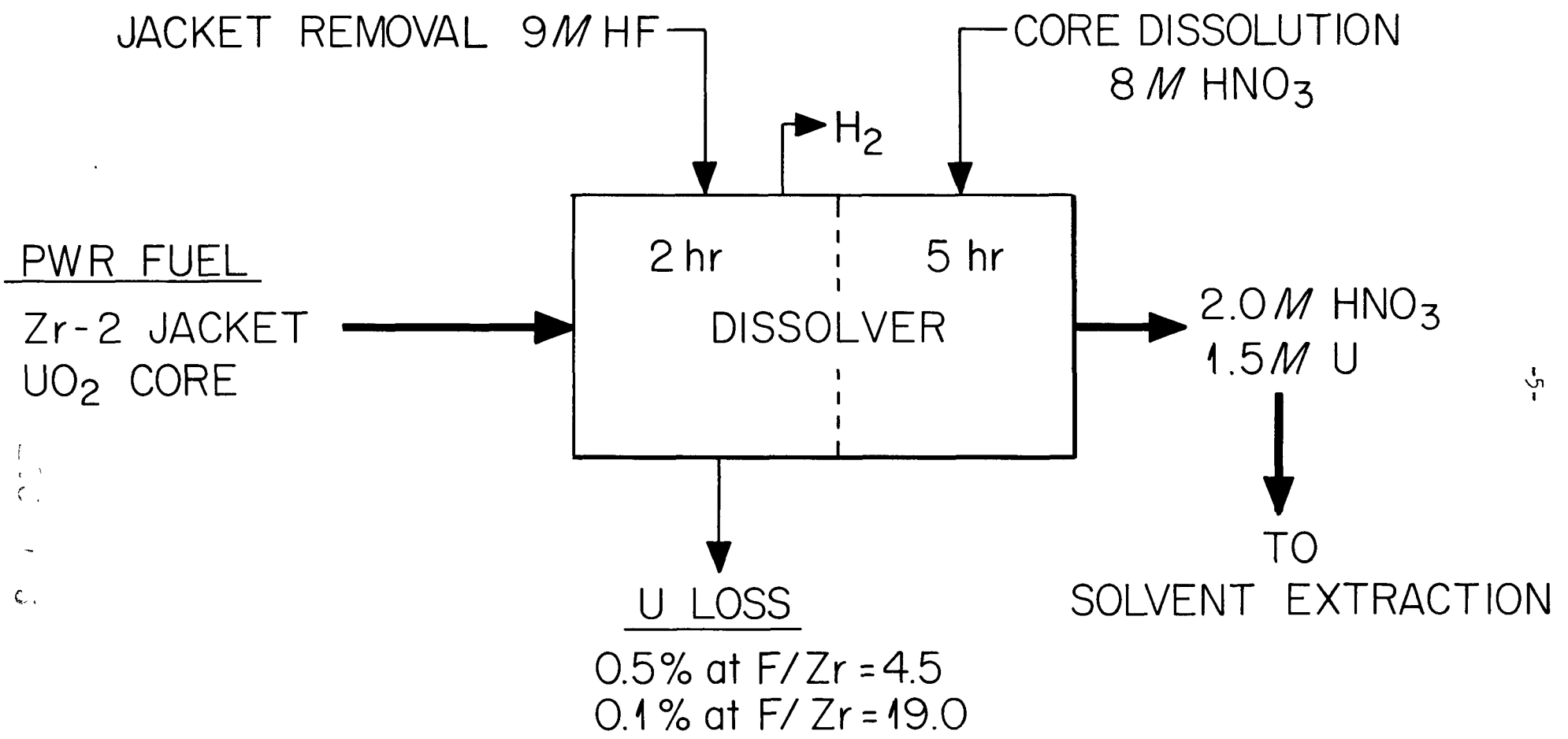

Fig. 3. Tentative Flowsheet for Zircaloy-2 Cladding Removal with HF.

the dissolver, by applying the vacuum to a dip tube, to the Lucite vessel where solid-liquid separations were achieved by settling. Slurries and liquid samples were removed from the bottom of the Lucite pot. In the laboratory work, all solutions were clarified by filtration. The equipment needed for filtration was also installed in the hot cell (right half of Fig. 1) but was used only in test runs with unirradiated fuel. Liquid level indicators (labelled L.L. in Fig. 1) were installed in all vessels. Decladding times were varied to determine if extended exposure of the core to the decladding solution would result in significantly higher uranium and plutonium losses.

After decladding, the dissolver was rinsed with three-100 ml portions of cold water to remove the last traces of the decladding solution and to dissolve solids such as $(NH_4)_3ZrF_7$, if formed. Ten molar nitric acid was then added and the core was digested for 3 hr. The resulting solution was removed and the core residue was digested for an additional hour in 10 M HNO_3. This treatment was followed by a boiling water wash and a final overnight digestion with 6 M NH_4F to remove the zirconium end caps.

2.2 Runs with Unirradiated Fuel

Seven preliminary experiments with unirradiated fuel samples were performed to test the apparatus. In the first six runs all solutions were clarified by passage through a Teflon filter. The filter proved inoperable because of solids formation and was removed after the sixth run. Several hours were required to remove the decladding and subsequent wash solutions. The Lucite settling vessel was used in all subsequent runs. Uranium losses in the first six runs were generally less than 0.1% and adequate removal of the decladding solution was demonstrated (Table 1). In these runs, less than 4% of the zirconium was found in the core solution. With no filter in the system, an average of 23% of the zirconium was found in the core solution (run 7, Table 1; Table 3). This result is attributed to the slow dissolution rate in cold water of $(NH_4)_3ZrF_7$ which precipitated from the decladding solutions. This precipitate was also responsible for the inoperability of the filter. The presence of $(NH_4)_3ZrF_7$ during core dissolution also leads to nitric acid-insoluble precipitates such as $ZrF_4 \cdot H_2O$. The problem of zirconium carry-over could have been avoided if analytical results would have been available before the experiments with irradiated fuel were begun.

Some of the runs were performed with PWR-type fuel elements which had been autoclaved 1 month at 1200 psig steam pressure and 800 psig oxygen pressure at 300°C. These runs indicated that the oxide coat produced on the clad by the autoclave treatment would not seriously impede dissolution in 6 M NH_4F.

In the only experiment with unirradiated fuel where decladding was achieved with 9 M HF, the uranium loss to the decladding solution was 3.4%.

Table 1. Summary of Cold Demonstration Runs Using 6 M NH_4F-1 M NH_4NO_3 or 9 M HF to Declad PWR Blanket Fuel Procedure of Figs. 2 and 3 was followed

Run No.	Decladding Solution		Core Solution		
	% U	% Zr	% U	% Zr	M F^-
1	0.08	98.2	99.9	1.8	0.09
2*	3.4	99.8	96.6	0.2	0.14
3	1.3**	99.7**	98.7	0.3	0.20
4	0.03	96.0	99.97	4.0	0.75
5	0.4	96.9	99.6	3.1	0.17
6	0.9	96.3	99.1	3.7	0.25
7	0.03	78.7	99.97	21.3	2.19

*Only run using HF to declad

**Includes a precipitate containing 0.4% of the U and 1.9% of the Zr which was removed from the filter

2.3 Runs with Irradiated Fuel

With irradiated fuel, soluble uranium losses to the 6 M NH_4F-1.0 M NH_4NO_3 decladding solutions were generally less than 0.2% and did not vary systematically with decladding time or burnup (Table 2). In general, the losses were no higher than those with unirradiated fuel. Detailed analyses of the waste decladding solutions are listed under "Zr" in Table 3. The fuel specimens in run 4-11 and 15-17 had been in contact with pressurized water (1800-2200 psig) at 500-600°C during irradiation. Decladding periods as short as 3 hr apparently provided satisfactory decladding of these specimens (Table 3).

Three hours was allowed for the first digestion of the UO_2 core in 10 M HNO_3. In this period, greater than 95% of the UO_2 dissolved (see row "U" in Table 3). In the second, 1-hr, digestion in 10 M HNO_3, significant quantities (0.1 to 10%) of the uranium and plutonium were recovered (row "UW", in Table 3). The boiling water wash also removed more uranium and plutonium after the second core dissolution (WW in Table 3). Following the water wash, the dissolver was cleaned overnight with refluxing 6 M NH_4F. Varying quantities of uranium and plutonium (up to 0.65%) were always found in the 6 M NH_4F solution (Zr B in Table 3).

Before sampling, solutions were allowed to stand 1 hr in the transparent Lucite settling vessel. Any precipitates which settled from the solution were transferred to a small sampling bottle. Precipitates from the decladding solution were washed with water and hot nitric acid and the combined wash solutions analyzed for uranium, plutonium, and zirconium (S in Table 3). The nitric acid-insoluble materials collected from the core solution and the decladding solution were combined and analyzed for uranium, plutonium, and zirconium (Ppt in Table 3).

The presence of an average of 23% of the zirconium in the core solutions after decladding in 6 M NH_4F-1 M NH_4NO_3 (Table 3) showed that ammonium fluorozirconate, $(NH_4)_3\ ZrF_7$, precipitated from the decladding solution and remained in the dissolver, despite the water washes, until the core dissolution. Fluoride analyses from runs 1, 2, and 3 showed 1.4, 1.5, and 1.4 M F, respectively, in the core solutions corresponding to F/Zr mole ratios between 5.5 and 7.

A nitric acid-insoluble precipitate was obtained in the first nine runs and in run 17. None was found in the two runs using 9 M HF (runs 10 and 11) suggesting that the nitric acid-insoluble precipitate is related to the formation of ammonium fluorozirconate. Formation of the nitric acid-insoluble material can be prevented by thorough water washing after decladding, i.e., complete removal of $(NH_4)_3\ ZrF_7$ prior to core dissolution. Therefore, no loss of uranium and plutonium to such as precipitate is expected in the process.

3.0 APPENDIX

3.1 Description of Equipment

The essential parts of the equipment (Figs. 1 and 4) resemble closely those described in the laboratory development work, except that Carpenter 20

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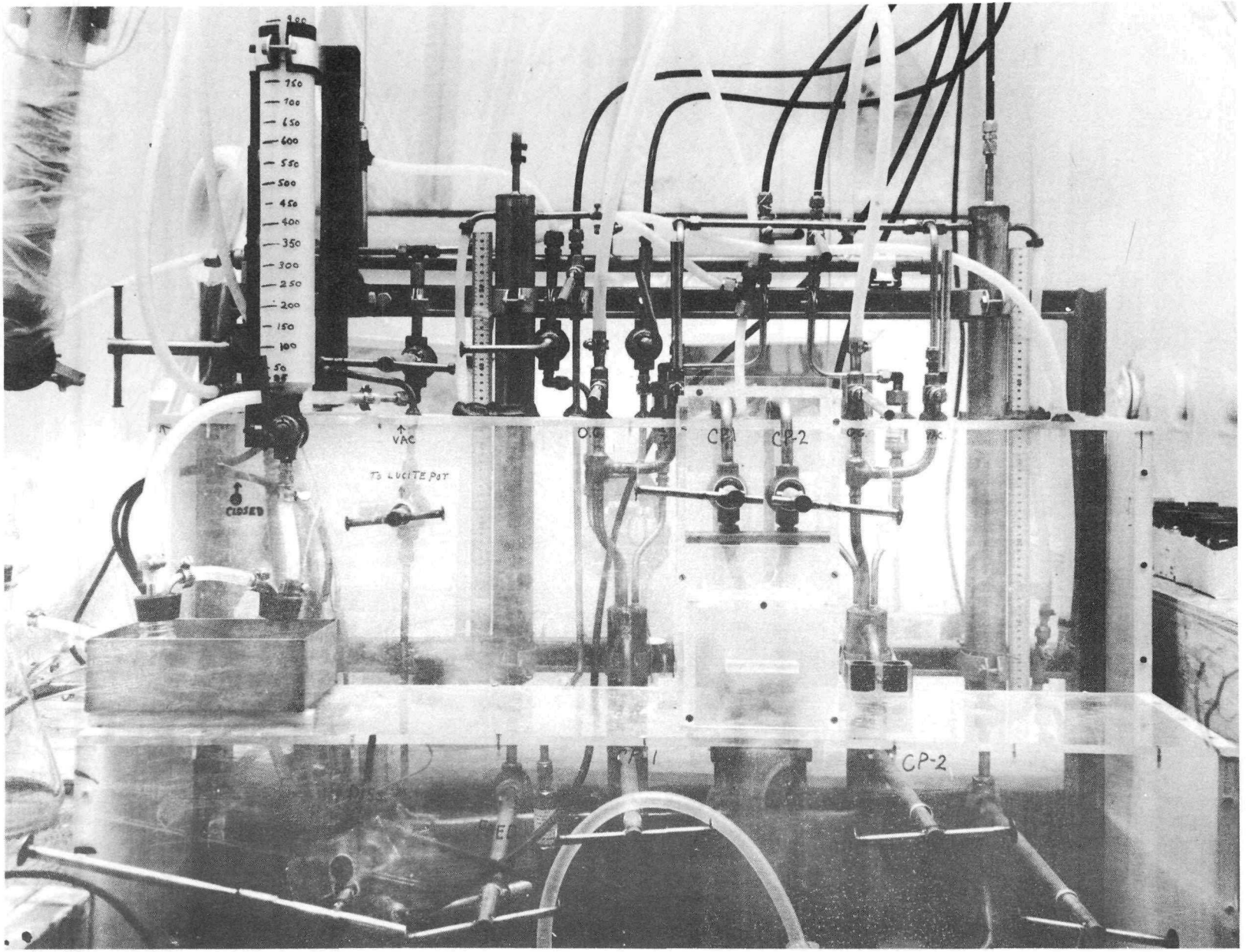

Fig. 4. Hot Cell Equipment.

metal was used rather than Pyrex glass[1] or polyethylene[2]. Most of the equipment was enclosed in transparent plastic to reduce the amount of contamination inside the hot cell. A small exhaust fan (not visible in Fig. 4) connected to the off-gas system, kept the air pressure inside the enclosure slightly below that in the cell proper. Valve handles were extended so that the valves could be operated outside of the plastic enclosure. All manipulations, including valve operation, were performed with Argonne Model 8 manipulators. The dissolver, marked "DISS", in the lower left corner of Fig. 4, was equipped with a valve with a 1" opening to permit loading of PWR blanket fuel rods (approximately 10" long and 3/8" in diameter). The long lever was required on this valve handle to permit operation with manipulators. The condenser, partly hidden above the dissolver, was connected to the off-gas system through a sodium hydroxide scrubber-trap. Solutions were removed from the dissolver to the transparent, calibrated Lucite settling vessel by applying a vacuum in the Lucite vessel. Analytical samples were removed from the bottom of the Lucite vessel. Nitrogen gas, introduced through a dip tube inside the Lucite vessel, was used to mix solutions and washes so that homogeneous samples could be obtained. The remaining solutions were removed through the dip tube to the hot drain. Most of the equipment shown on the right side of Fig. 4 is an alternative sampling system which was used only in the runs with unirradiated fuel when solutions from the dissolver were filtered. The precipitation of ammonium fluorozirconate prevented the use of filtration in the runs with irradiated fuel specimens.

3.2 Fuel Description and Irradiation Data

The following data, compiled by J. W. Ullman of the Chemical Technology Division Long Range Planning Group, shows the fuel specifications and expected irradiation levels for the PWR blanket.

PWR FUEL SPECIFICATIONS

NUCLEAR	Seed	Blanket
Average Thermal Flux	6×10^{13}	4×10^{13}
Average Fast Flux	2×10^{14}	1×10^{14}
Specific Power (at start)	1390 kw/kg U-235	9.3 kw/kg U
Loading	75 kg U-235	12,990 kg U
Enrichment, % U-235	93	Natural
Irradiation Time (full power	250 days	750 days
Burnup (design conditions)	40% U-235	8200 Mwd/T U

ELEMENT		
Type	Plate	Rod
Unclad Dimensions	2.05" x 0.039" x 70.75"	0.357" dia x 9.25 long
Dimensions with Clad	2.50" x 0.069" x 72.4"	0.411" dia x 10.25"
Clad Thickness	0.015"	0.027"
Fuel	6.33% U-93.67% Zr	UO_2(26 pellets, 0.137 kg U)
Clad Material	Zircaloy-2	Zircaloy-2
Bond	Metallurgical	He

SUBASSEMBLY		
Type	Box	Bundle
Number of Elements	15	120
Dimensions	2.5" x 2.5" x 6'	5.2" x 5.2" x 10.25"
Side Plate Thickness	0.125"	--
Subassemblies per Core	128	791
kg/Subassembly Before Irradiation: U	0.62	16.3
Zr	28	6.0
Oxygen	--	2.12

PWR FUEL SPECIFICATIONS

ASSEMBLY	Seed	Blanket
Number of subassemblies	4	7
Casing Thickness	--	0.2"
Over-all Dimensions	5.5" x 5.5" x 110"	5.6" x 5.6" x 110"
Active Section Length	6'	6'
Active Section Before Irradiation, kg[a]		
U	2.5	115
Zr	127	42
Oxygen	---	15
Sp Spacing Between Subassemblies	0.5"	None (0.235" thick tube sheets)
S and S Material After Irradiation, kg[b]		
U	1.69	114
U-235	1.39	0.57
Pu	--	0.268
Assemblies Per Core	32	113
SHIPMENT		
Cooling Time Before Shipment	120 days	120 days
Preparation	Reduce to 76.25" long	None [c]

[a] without end adaptors or blanket casing

[b] design conditions

[c] bundles can be removed from casing

Table 2. Uranium and Plutonium Losses to the 6 M NH_4F-1 M NH_4NO_3 or 9 M HF Used to Declad Irradiated PWR-Blanket Fuel

Run No.	Decladding Reagent	Losses to the Decladding Solution		Decladding Period, hr	% Recovered from End-cap Cleanup		% Found in the Insoluble Precipitate,		Irradiation Conditions		
		U,%	Pu,%		U	Pu	U	Pu	Approximate Burnup Mwd/T	T, °F	Water Pressure psig
1	6 M NH_4F-1 M NH_4NO_3	0.032	0.008	5	-	-	0.01	0.00	80	110	12
2	"	0.172	0.231	6	0.23	0.02	0.00	0.00	80	110	12
3	"	0.526	0.090	7	0.14	-	0.00	0.18	80	110	12
4	"	0.130	0.021	6	0.08	0.65	0.66	2.23	940	500-600	1800-2200
5	"	0.248	0.039	6	0.08	0.06	0.01	0.02	740	500-600	1800-2200
6	"	0.074	0.059	6	0.06	0.06	0.41	0.53	870	500-600	1800-2200
7	"	0.099	0.084	10	0.14	0.27	0.75	1.53	870	500-600	1800-2200
8	"	0.129	0.145	3	0.10	0.19	0.37	0.51	670	500-600	1800-2200
9	"	0.092	0.059	9	0.19	0.29	none		600	500-600	1800-2200
10	9 M HF	1.270	0.510	3	0.17	0.02	"		540	500-600	1800-2200
11	"	0.828	0.260	3	0.20	0.11	"		1100	500-600	1800-2200
12	6 M NH_4F-1 M NH_4NO_3	0.042	0.013	5	0.15	0.15	"		870	115	9-48
13	"	0.110	0.060	10	0.11	0.12	"		940	115	9-48
14	"	0.088	0.058	10	0.09	0.05	"		670	115	9-48
15[a]	"	0.227	0.478	5	0.17	0.10	"		540	500-600	1800-2200
16[a]	"	0.810	5.96	10	0.17	0.30	"		270	500-600	1800-2200
17[a]	"	0.197	0.145	7	0.19	0.25	0.01	0.04	1100	500-600	1800-2200

[a]Runs 15, 16, and 17 were performed with odd-size elements with incomplete histories. The cladding probably contained serious flaws.

Table 3. Detailed Summary of Demonstration Runs Using 6 M NH_4F-1 M NH_4NO_3 or 9 M HF to Declad Irradiated PWR Blanket Fuel

Code[a]	Reagent	Contact Time, hr	Percent of Total[b] Run 1 U	Run 1 Zr	Run 1 Pu	2 U	2 Zr	2 Pu	3 U	3 Zr	3 Pu	4 U	4 Zr	4 Pu
U	10 M HNO_3	3	98.1	22.6	89.2	92.9	25.7	95.0	92.6	20.2	91.4	97.5	22.9	73.3
UW	10 M HNO_3	1	0.92	0.84	0.13	2.40	1.17	2.17	2.74	5.40	3.65	1.03	1.06	10.8
WW	H_2O	0.2	0.09	-	0.03	0.30	-	0.27	0.27	5.11	0.20	0.15	-	12.9
Zr	6 M NH_4F-[c]													
	1 M NH_4NO_3	3-10	0.03	74.5	0.01	0.17	71.0	0.23	0.53	66.3	0.09	0.13	71.4	0.02
Zr B	6 M NH_4F"													
	1 M NH_4NO_3	~10	-	1.64	-	0.23	0.01	0.02	0.14	1.88	-	0.08	2.82	0.65
S	H_2O	0.2												
	5 M HNO_3	0.5	0.91	0.36	10.6	4.01	2.09	2.36	3.66	1.15	4.47	0.53	1.35	0.13
Ppt			0.01	0.06	0.00		none		0.00	0.05	0.18	0.66	0.53	2.23
Material Balance[d]			101.8	67.7	-	103.5	71.3	-	104.7	99.0	-	79.3	116.0	-

a) Code: U - 1st core dissolution
UW - 2nd core dissolution
WW - boiling water wash after core dissolution
Zr - 1st clad dissolution
ZrB - 2nd clad (and endcap) dissolution
S - precipitate which dissolved upon treatment with water followed by 5 M HNO_3
Ppt - precipitate which remained insoluble in water or 5 M HNO_3

b) All numbers given are percent of the respective element, based on the sum of all analyses.

c) 9 M HF in runs 10 and 11

d) based on 133 g U, 50 g Zr per PWR blanket element

e) Runs 15, 16, and 17 were performed with odd-size elements obtained from Westinghouse Atomic Power Division. All other runs were performed with the standard PWR production element.

Table 3. Detailed Summary of Demonstration Runs Using 6 M NH_4F-1 M NH_4NO_3 or 9 M HF to Declad Irradiated PWR Blanket Fuel

Code[a]	Reagent	Contact Time, hr	Percent of Total[b] Run 1 U[b]	Run 1 Zr	Run 1 Pu	2 U	2 Zr	2 Pu	3 U	3 Zr	3 Pu	4 U	4 Zr	4 Pu
U	10 M HNO_3	3	98.1	22.6	89.2	92.9	25.7	95.0	92.6	20.2	91.4	97.5	22.9	73.3
UW	10 M HNO_3	1	0.92	0.84	0.13	2.40	1.17	2.17	2.74	5.40	3.65	1.03	1.06	10.8
WW	H_2O	0.2	0.09	-	0.03	0.30	-	0.27	0.27	5.11	0.20	0.15	-	12.9
Zr	6 M NH_4F-[c]													
	1 M NH_4NO_3	3-10	0.03	74.5	0.01	0.17	71.0	0.23	0.53	66.3	0.09	0.13	71.4	0.02
Zr B	6 M NH_4F-													
	1 M NH_4NO_3	~10	-	1.64	-	0.23	0.01	0.02	0.14	1.88	-	0.08	2.82	0.65
S	H_2O	0.2												
	5 M HNO_3	0.5	0.91	0.36	10.6	4.01	2.09	2.36	3.66	1.15	4.47	0.53	1.35	0.13
Ppt			0.01	0.06	0.00		none		0.00	0.05	0.18	0.66	0.53	2.23
Material Balance[d]			101.8	67.7	-	103.5	71.3	-	104.7	99.0	-	79.3	116.0	-

a) Code: U - 1st core dissolution
UW - 2nd core dissolution
WW - boiling water wash after core dissolution
Zr - 1st clad dissolution
ZrB - 2nd clad (and endcap) dissolution
S - precipitate which dissolved upon treatment with water followed by 5 M HNO_3
Ppt - precipitate which remained insoluble in water or 5 M HNO_3

b) All numbers given are percent of the respective element, based on the sum of all analyses.

c) 9 M HF in runs 10 and 11

d) based on 133 g U, 50 g Zr per PWR blanket element

e) Runs 15, 16, and 17 were performed with odd-size elements obtained from Westinghouse Atomic Power Division. All other runs were performed with the standard PWR production element.

Table 3 (Continued)

Code	Reagent	Contact Time, hr	Run 5			6			7			8		
			U[b]	Zr	Pu	U	Zr	Pu	U	Zr	Pu	U	Zr	Pu
U	10 M HNO_3	3	96.5	26.0	97.6	97.0	26.8	97.5	94.0	12.8	77.8	96.6	29.5	96.4
UW	10 M HNO_3	1	2.16	2.26	1.78	1.63	3.72	1.72	4.37	4.38	19.0	2.05	3.12	2.21
WW	H_2O	0.2	0.24	-	0.40	0.12	-	0.16	0.30	-	1.26	0.15	-	0.18
Zr	6 M NH_4F-													
	1 M NH_4NO_3	3-10	0.25	70.7	0.04	0.07	68.3	0.06	0.10	79.5	0.08	0.13	62.2	0.15
Zr B	6 M NH_4F-													
	1 M NH_4NO_3	~10	0.08	0.28	0.06	0.06	0.14	0.06	0.14	1.70	0.27	0.10	3.83	0.19
S	H_2O	0.2												
	5 M HNO_3	0.5	0.81	0.76	0.08	0.78	0.83	0.03	0.78	1.27	0.08	0.65	1.08	0.41
Ppt		-	0.01	0.01	0.02	0.41	0.28	0.53	0.32	0.35	1.53	0.37	0.31	0.51
Material Balances			110.8	100.0	-	114.2	118.5	-	101.4	82.1	-	105.7	110	-

Table 3 (Continued)

Code	Reagent	Contact Time, hr	9			10			11			12		
			U	Zr	Pu	U	Zr	Pu	U	Zr	Pu	U	Zr	Pu
U	10 M HNO_3	3	96.2	21.3	95.9	97.1	17.2	97.7	96.1	2.74	97.0	97.5	47.9	96.9
UW	10 M HNO_3	1	3.37	8.68	3.42	0.80	0.70	0.97	1.74	0.65	1.83	2.08	0.99	1.85
WW	H_2O	0.2	0.17	-	0.27	-	-	-	-	-	-	0.19	-	0.44
Zr	6 M NH_4F- 1 M NH_4NO_3	3-10	0.09	65.7	0.06	1.27	81.3	0.51	0.83	95.1	0.26	0.04	49.8	0.01
Zr B	6 M NH_4F- 1 M NH_4NO_3	~10	0.19	3.55	0.29	0.17	0.33	0.02	0.20	0.55	0.11	0.15	0.63	0.15
S	H_2O	0.2												
	5 M HNO_3	0.5	0.03	0.76	0.00	0.72	0.49	0.77	1.18	0.98	0.85	0.09	0.67	0.66
Ppt				none			none			none			none	
Material Balances		-	106.1	100.5	-	88.4	120.0	-	98.8	123.3	-	101.1	103.6	-

Table 3. (Continued)

Code	Reagent	Contact Time, hr	13			14			15			16			17		
			U	Zr	Pu	U	Zr	Pu	U	Zr	Pu	U	Zr	Pu	U	Zr	Pu
U	10 M HNO_3	3	97.3	18.5	98.6	98.7	12.8	84.5	98.7	6.19	98.7	98.3	43.2	92.3	99.1	12.2	99.1
UW	10 M HNO_3	1	1.91	1.44	0.84	0.56	0.55	14.9	0.72	1.12	0.69	0.70	9.79	1.46	0.42	2.95	0.49
WW	H_2O	0.2	0.14	-	0.22	0.08	-	0.10	-	-	-	-	-	-	-	-	-
Zr	6 M NH_4F-1 M NH_4NO_3	3-10	0.11	78.3	0.06	0.09	81.5	0.06	0.23	89.0	0.48	0.81	38.7	5.96	0.20	78.0	0.15
Zr B	6 M NH_4F-1 M NH_4NO_3	~10	0.11	1.04	0.12	0.09	1.88	0.05	0.17	0.60	0.10	0.17	5.36	0.30	0.19	1.11	0.25
S			0.47	0.73	0.15	0.53	3.35	0.45	0.16	3.12	0.01	0.08	-	0.03	0.12	5.06	0.00
Ppt	None			none			none			none			none		0.10	0.75	0.04
Material Balances			96.1	105.2	-	98.2	84.8	-	unknown(e)			unknown(e)			unknown(e)		

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