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SECOND ELECTROLYTIC DISSOLUTION CAMPAIGN OF EBR-II FUEL AT ICPP

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SECOND ELECTROLYTIC DISSOLUTION CAMPAIGN OF EBR-II FUEL AT ICPP

by

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IDAHO CHEMICAL PROGRAMS

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ABSTRACT

The second fuel reprocessing campaign using the electrolytic dissolution facility at the Idaho Chemical Processing Plant (ICPP) took place from February 1975 to November 1976. Several forms of EBR-II fuel, and various scrap fuels containing highly-enriched uranium, were dissolved in nitric acid in the dissolver's electric field. Gadolinium was used as a soluble nuclear poison to guarantee nuclear safety in the headend processing equipment.

SUMMARY

Approximately 1690 kg of total uranium, mostly 52.5-70% enriched, were recovered from EBR-II fuel and scrap during the second electrolytic dissolution campaign from February 1975 through November 1976. The campaign included five dissolution first-cycle extraction segments and four second- and third-cycle plus product denitration segments. The dissolution and extraction systems performed adequately; improvements incorporated into the extraction systems during the first campaign continued to enhance their performance, resulting in uranium losses well below throwaway limits. However, the denitration system's poor performance resulted in an excessive amount of downtime.

While dissolution rates of EBR-II Mark IA fuel subassemblies and skull oxide improved from those of the first campaign, the dissolution rates of clad pins in aluminum cans were lower than anticipated.

The use of gadolinium as a nuclear poison to ensure criticality safety was satisfactory, and the digital counting methods used in the nuclear poison detection system performed well.

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SECOND ELECTROLYTIC DISSOLUTION CAMPAIGN OF EBR-II FUEL AT ICPP

I. INTRODUCTION

The second campaign of the ICPP electrolytic dissolution system, which causes anodic dissolution of metals contained in the electric field, was run from February 1975 to November 1976. Most of the fuel processed was EBR-II fuel: Mark IA subassemblies, skull oxide and clad pins in aluminum cans. Other fuels processed were EBR-II Mark II fuels, fuels in oversize cans, sodium-contaminated scrap, and cold Army Package Power Reactor (APPR) fuel.

The EBR-II fuels are essentially an alloy of fissium(a) and uranium clad in stainless steel tubes. The metal cladding is dissolved electrolytically, the uranium chemically. Mercury was added to the dissolvent to catalyze the reaction for the aluminum cans. Criticality safety was maintained by adding gadolinium to the nitric acid dissolvent. The resultant acid dissolver product was adjusted and processed through the first-cycle extraction equipment using a 10 volume-percent tributyl phosphate (TBP) flowsheet. The product was then concentrated and sent to intercycle storage; when intercycle storage was filled to capacity, the first-cycle system was shut down and the product was processed through second- and third-cycle extractions using hexone as a solvent.

The third-cycle product was reprocessed through the fluidized-bed denitration system where the uranyl nitrate solution was converted to a granular uranium trioxide (UO_3). The solid product was then packaged and shipped. Figure 1 shows the history of the campaign.

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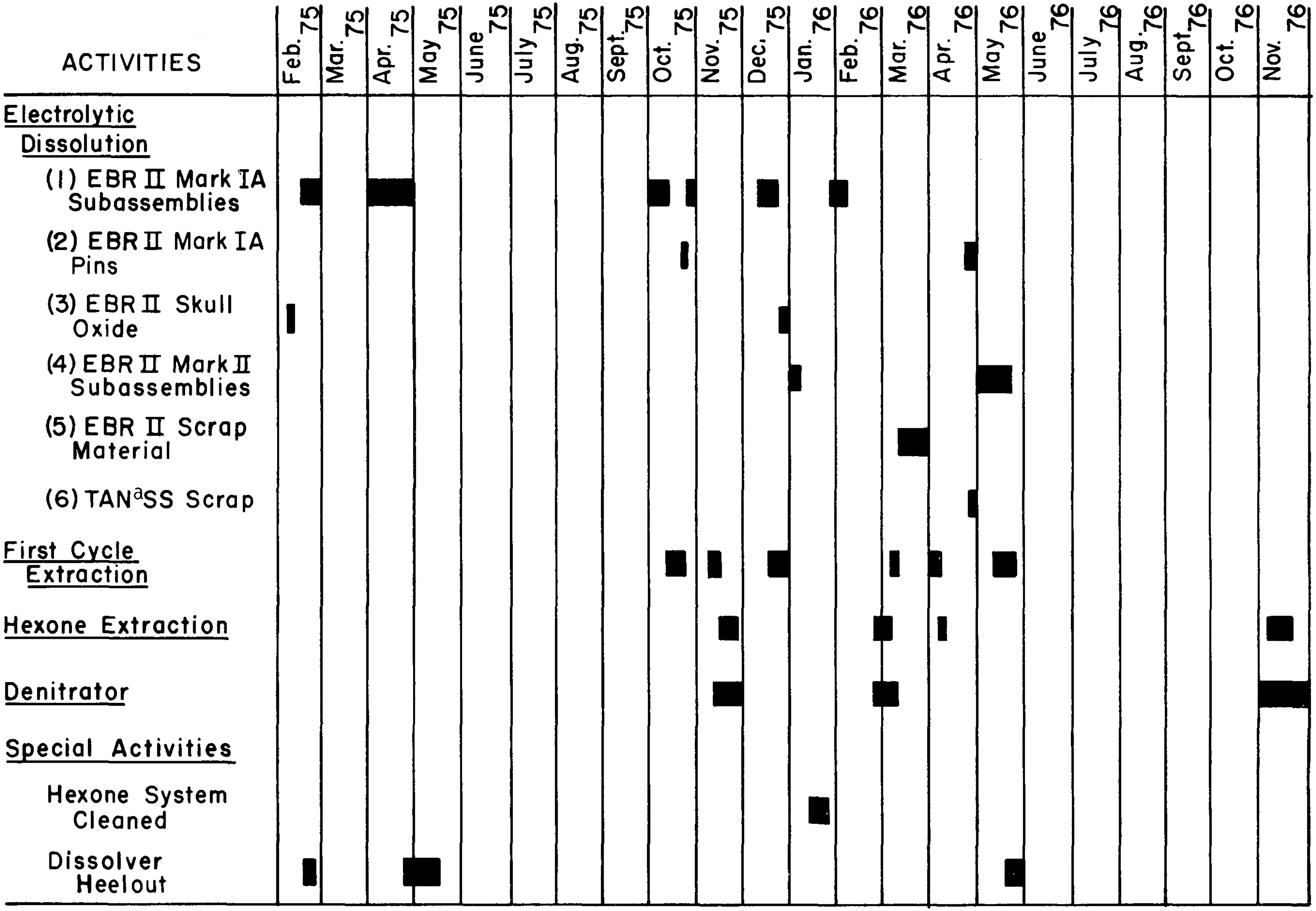
[a] The composition of fissium is mainly Mo, Ru with small amounts of Rh, Pd, Zr, and Nb.



a Test Area North

Figure 1. SECOND ELECTROLYTIC CAMPAIGN PROCESS

II. PROCESS DESCRIPTION

1. Headend Dissolution

Several categories of EBR-II fuels were processed during the campaign: Mark IA fuel subassembly sections, clad fuel pins in aluminum cans, skull oxide in aluminum cans, cans of scrap fuel with exposed sodium, cans too large to fit into the dissolver, one canned ingot, canned Mark II subassembly sections, and uncanned Mark II subassembly sections.

The fuel sections and cans were transferred from the storage basin to the top of the process cell and then dropped to a table in the cell. Remotely operated manipulators placed the fuel in the dissolver where dissolver product solution (essentially a metal nitrate-nitric acid solution with a nuclear poison) was circulated continuously to the bottom of the dissolver. Fresh dissolvent was added continuously to the recirculating product. With the exception of initial periods required to reach flowsheet concentrations, heel-out periods, and unscheduled downtime, product was removed continuously to accountability tanks prior to first-cycle extraction. (See references 1 and 2 for detailed descriptions of the process theory and equipment.)

Typical material balances for the dissolution of the EBR-II Mark IA fuel subassembly sections and the first-cycle extraction of the resulting dissolver solution are shown in Figures 2 and 3. Typical material balances for clad EBR-II fuel elements in aluminum cans are shown in Figures 4 and 5. Figures 6 and 7 show the flowsheets used for processing skull oxide in aluminum cans.

2. First-Cycle Extraction

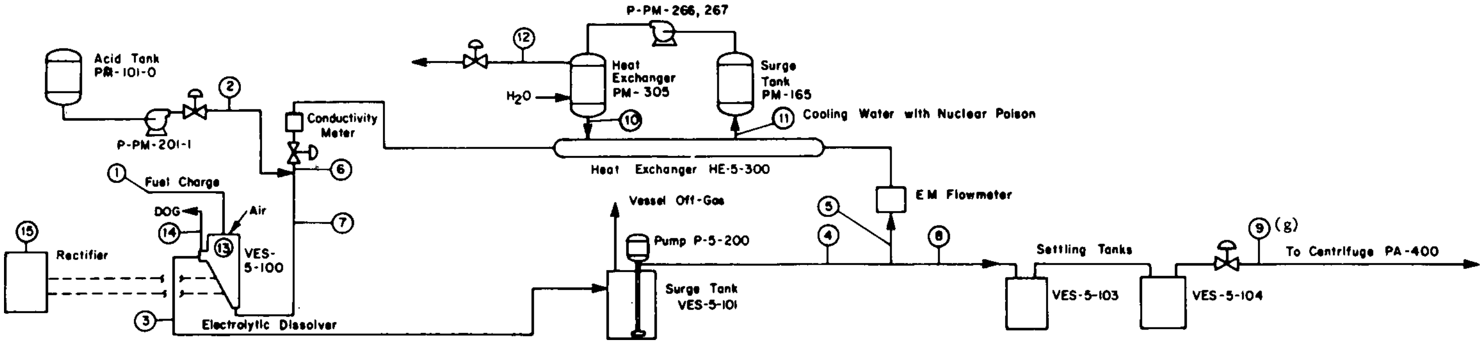
The extraction phase of the electrolytic dissolution process is similar to that used in other EBR-II fuel processing campaigns at the ICPP. In the extraction column the uranium is extracted into 10 volume percent TBP in NPH solvent. Plutonium and neptunium, present in very small quantities, cannot be economically recovered. Figures 3, 5, and 7 show the extraction flowsheets used during the campaign.

3. Second- and Third-Cycle Extraction

The uranium is further purified in second- and third-cycle extraction equipment. Methyl isobutyl ketone (hexone) is used as solvent in the system; Figure 8 shows a typical flowsheet.

4. Product Denitration

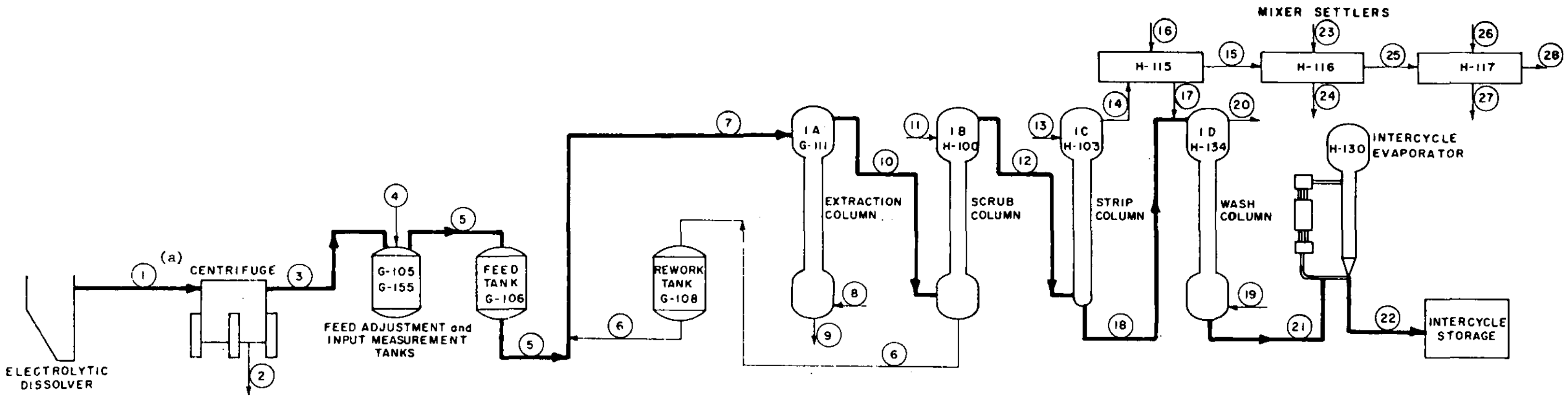
Decontaminated product from the third extraction cycle is stored in critically safe tanks at a uranium concentration of 350 g/L. It is then converted into solid UO_3 granules in the fluidized-bed denitrator and packaged into containers in a glove box. Figure 9 provides an equipment flowsheet and a material balance. (See reference 1 for a more detailed description of the process.)



Stream	1	2	3	4 & 5	6 & 7	8	9	10	11	12	13	14	15	16	17	18
Description	Average Fuel Charge (b)	Acid Dissolvent	Overflow to Surge Tank	Recirculation Solution to Heat Exchanger	Recirculation Solution from Heat Exchanger	Product Stream to Settling Tanks	Product Stream to Centrifuge (g)	Cooling Water to Heat Exchanger	Cooling Water from Heat Exchanger	Secondary Cooling Water	Dissolver Off-Gas	Diluted Dissolver Off-Gas	Rectifier			
Flow, ℓ/hr		21	4500	4500(d)	4500(d)	21	21	3.6×10^4	3.6×10^4	(e)						
Uranium, g/hr	2083															
Stainless steel, g/hr	1083															
Sodium, g/hr	24															
Fissium, g/hr	111															
Uranium, g/ℓ			100	100	100	100	100									
Stainless steel, g/ℓ(a)			49	49	49	49	48.5									
Fissium, g/ℓ(a)			5.3	5.3	5.3	5.3	5.3									
Undissolved solids, g/ℓ			3.7(c)	3.7	3.7	3.7	3.2									
H^+, M		9.0	4.2	4.2	4.2	4.2	4.2									
NO_3, M		9.0	8.0	8.0	8.0	8.0	8.0									
Flow, scfm											0.8	38				
Temperature, °F		70	130	125	115	120	120	104	107	107						
Pressure, psig		55	0	55	1	55	5	85	80	100						
SP.GR. @ 25°C		1.28	1.44	1.44	1.44	1.44	1.44									
NO_2, %											41	2				
H_2, %											3	0.1				
Amps													5000			
Volts													5-20(f)			
Gd, g/ℓ		2.6	2.6	2.6	2.6	2.6	2.6	2.6	2.6							
Na, g/ℓ			1.1	1.1	1.1	1.1	1.1									
U-235, g/ℓ			52.6	52.6	52.6	52.6	52.6									

(a) The concentration of these streams include some undissolved solids.
(b) One fuel section charged each 162 minutes.
(c) 1 g/ℓ stainless steel, 2.7 g/ℓ fissium.
(d) Small flowrate of product stream is neglected.
(e) Flowrate varies.
(f) Voltage depends on resistance in dissolver, which changes continuously.
(g) This becomes stream (1) on the accompanying extraction flowsheet.

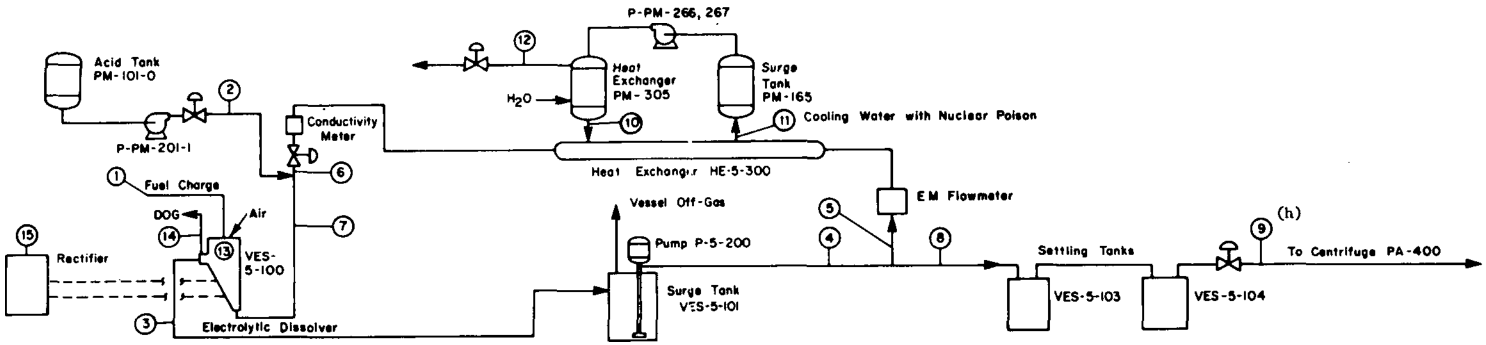
Figure 2. Typical Material Balance for Dissolution of EBR-II Mark IA Fuel Assemblies



STREAM	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19 & 20	21	22	23	24	25	26 & 27	28
DESCRIPTION	DISSOLVER PRODUCT (a)	SOLIDS TO PEW	CENTRIFUGED DISSOLVER PRODUCT	FEED ADJUSTMENT SOLUTION	ADJUSTED DISSOLVER PRODUCT	IBR	IAF	IAX	IAR	IAP	IBS	IBP	ICX	ICR	ISR	ISX	ISW	ICP	IDX IDR	IDP	FIRST CYCLE PRODUCT	II SX	II SW	II SR	III SX III SW	III SR (IAX)
Flow, ℓ/hr	21		21	79	100	50	150	300	150	300	50	300	200	300	300	50	50	200	10(f)	250	6.9	30	30	300	30	300
H^+, M	4.2		4.2	3.2	3.4	0.7	2.5		2.2	0.2	-0.2(e)	0	0.01			0.01	0.01	0.01		0.01	0.4				0.01	
NO_3^-, M	8.0		8.0	3.2	4.2	2.4	3.6		3.2	0.2	1.5	0.1	0.01			0.01	0.01	0.10		0.08	2.9				0.01	
SS, g/ℓ	48.5(b)		48.		10.2		6.8		6.8																	
Na, g/ℓ	1.1		1.1		0.2		0.1		0.1																	
Gd, g/ℓ	2.6		2.6		0.5		0.4		0.4				0.05					0.05		0.04	1.45					
Fissium, g/ℓ	5.3(c)		2.7		0.6		0.4		0.4																	
U, g/ℓ	100		100		20.8	0.4	14.0		1.8×10^{-3}	7.0		6.9		2.2×10^{-3}	1×10^{-4}		0.01	10.4		8.3	300		6×10^{-4}			
U-235, g/ℓ	52.6		52.6		10.9	0.2	7.4		0.9×10^{-3}	3.7		3.6		1.2×10^{-3}	5×10^{-5}		0.005	5.4		4.4	158					
Solids, g/hr ave	65	65																								
Al, M						0.5	0.2		0.2		0.5															
NH_4, M						0.2	0.1		0.1		0.2															
TBP, %								10(d)		10		10		10	10									10		10
Na_2CO_3, M																						0.5	0.5			
SP.GR. @ 25°C							1.15																			

(a) Stream (9) on the dissolution flowsheet.
(b) 0.5 g/ℓ SS solids present.
(c) Fifty percent of the fissium is insoluble in the dissolvent.
(d) 10% TBP in NPH.
(e) Acid deficient with NH_4OH.
(f) AMSCO

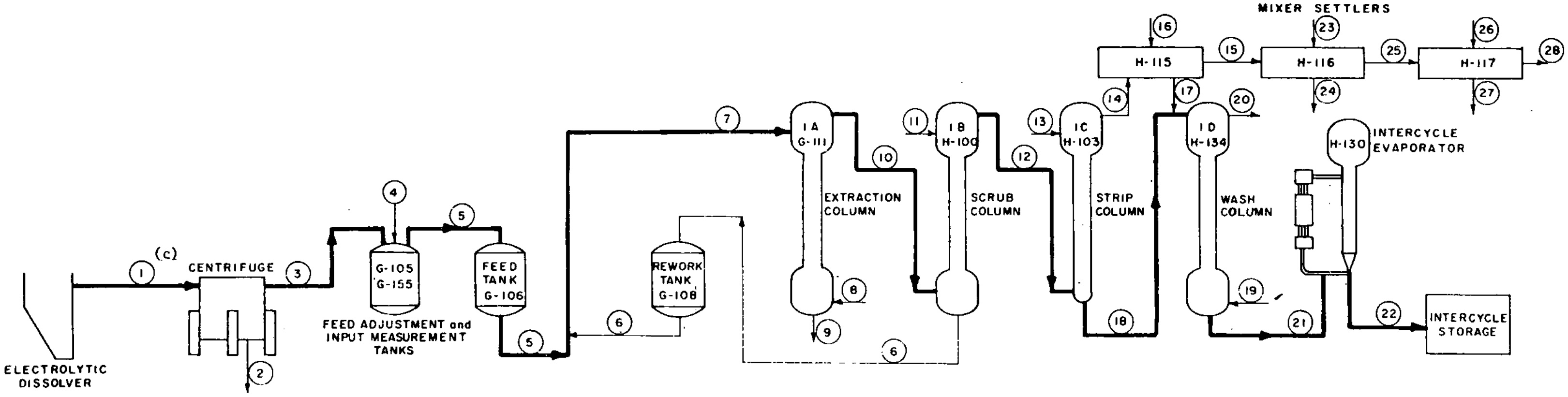
Figure 3. Typical Material Balance for First-Cycle Extraction of EBR-II Fuel Assemblies



Stream	1	2	3	4 & 5	6 & 7	8	9	10	11	12	13	14	15	16	17	18
Description	Average Fuel Charge (c)	Acid Dissolvent	Overflow to Surge Tank	Recirculation Solution to Heat Exchanger	Recirculation Solution from Heat Exchanger	Product Stream to Settling Tanks	Product Stream to Centrifuge (h)	Cooling Water to Heat Exchanger	Cooling Water from Heat Exchanger	Secondary Cooling Water	Dissolver Off-Gas	Diluted Dissolver Off-Gas	Rectifier			
Flow, ℓ/hr		41	4500	4500(e)	4500(e)	41	41	3.6×10^4	3.6×10^4	(f)						
Uranium, g/hr	2085															
Fissium, g/hr	110															
Aluminum, g/hr	1240															
Uranium, g/ℓ			50.8	50.8	50.8	50.8	50.8									
Fissium, g/ℓ(a)			2.7	2.7	2.7	2.7	2.7									
H^+, M		9.0	4.1	4.1	4.1	4.1	4.1									
NO_3, M		9.0	7.5	7.5	7.5	7.5	7.5									
Aluminum, M			1.1	1.1	1.1	1.1	1.1									
Flow, scfm											0.8	38				
Temperature, °F		70	130	125	115	125	120	104	107	107						
Pressure, psig		55		55	1	55	5	85	80	100						
SP.GR. @ 25°C		1.28	1.34	1.34	1.34	1.34	1.34									
NO_2, %											35	1.3				
H_2, %											3	0.1				
Amps													5000			
Volts													5-20(g)			
Gd, g/ℓ		4.3	4.3	4.3	4.3	4.3	4.3									
Undissolved Solids g/ℓ			1.3(d)	1.3	1.3	1.3	1.3	2.6	2.6							
U-235, g/ℓ			26.7	26.7	26.7	26.7	26.7									
$Hg(NO_3)_2$ M(b)		0.01	0.01	0.01	0.01	0.01	0.01									

(a) The concentration of these streams include some undissolved solids.
(b) Optional.
(c) One can charged each 164 minutes.
(d) Fissium only.
(e) Small flowrate of product stream is neglected.
(f) Flowrate varies.
(g) Voltage depends on resistance in dissolver which changes continuously.
(h) This becomes stream (1) on the accompanying extraction flowsheet.

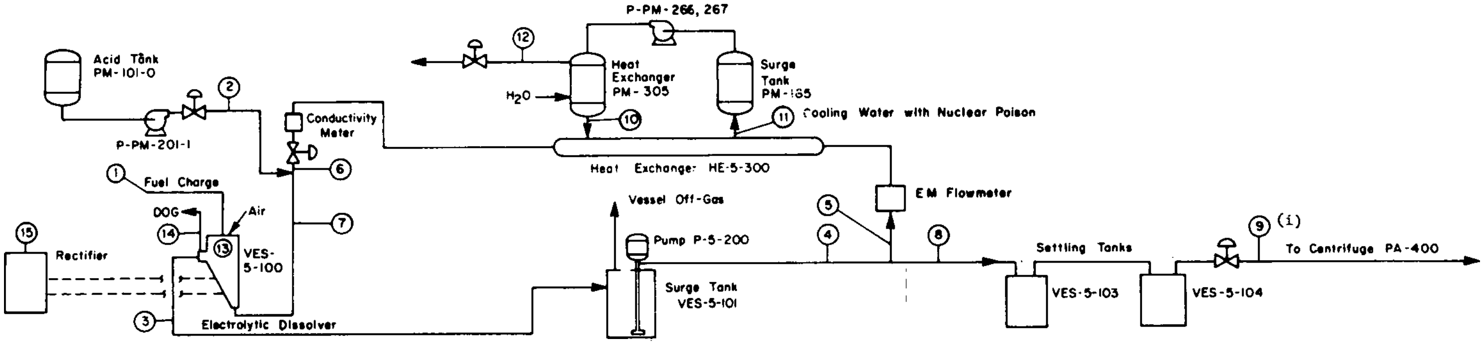
Figure 4. Typical Material Balance for Dissolution of Clad Fuel Elements in Aluminum Cans



STREAM	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19 & 20	21	22	23	24	25	26 & 27	28
DESCRIPTION	DISSOLVER PRODUCT	SOLIDS TO PEW (c)	CENTRIFUGED DISSOLVER PRODUCT	FEED ADJUSTMENT SOLUTION	ADJUSTED DISSOLVER PRODUCT	IBR	IAF	IAX	IAR	IAP	IBS	IBP	ICX	ICR	ISR	ISX	ISW	ICP	IDX IDR	IDP	FIRST CYCLE PRODUCT	IISX	IISW	IISR	IIISX IIISW	IIISR (IAX)
Flow, ℓ/hr	41		41	59	100	50	150	300	150	300	50	300	200	300	300	50	50	200	10(f)	250	6.9	30	30	300	30	300
H^+, M	4.1		4.1	2.9	3.4	0.7	2.5		2.2	0.2	-0.2(e)	0	0.01			0.01	0.01	0.01		0.01	0.4				0.01	
NO_3, M	7.5		7.5	2.9	4.8	2.4	4.0		3.5	0.2	1.5	0.6	0.01			0.01	0.01	0.1		0.1	2.9				0.01	
Al, M	1.1		1.1		0.4	0.5	0.5		0.5		0.5															
Gd, g/ℓ	4.3		4.3		1.8		1.2		1.2				0.05					0.05		0.04	1.45					
Fissium, g/ℓ(a)	2.7		1.4		0.6		0.4		0.4																	
U, g/ℓ	50.8		50.8		20.8	0.4	14		1.8×10^{-3}	7.0		6.9		2.2×10^{-3}	1×10^{-4}		0.01	10.4		8.3	300		6×10^{-4}			
U-235, g/ℓ	26.7		26.7		10.9	0.2	7.4		$.9 \times 10^{-3}$	3.7		3.6		1.2×10^{-3}	5×10^{-5}		0.005	5.4		4.4	158					
Solids, g/hr	55	55																								
NH_4, M						0.2	0.1		0.1		0.2															
TBP, %								10(d)		10		10		10	10									10		10
Na_2CO_3																						0.5	0.5			
SP.GR. @ 25°C							1.1792																			
$Hg(NO_3)_2$ (b)																										

(a) The concentration of these streams include some undissolved solids.
(b) Optional.
(c) Stream (9) on the dissolution flowsheet.
(d) Ten percent TBP in NPH.
(e) Acid deficient with NH_4OH.
(f) AMSCO.

Figure 5. Typical Material Balance for First-Cycle Extraction of Clad Fuel Elements in Aluminum Cans



Stream	1	2	3	4 & 5	6 & 7	8	9	10	11	12	13	14	15	16	17	18
Description	Average Fuel Charge (d)	Acid Dissolvent	Overflow to Surge Tank	Recirculation Solution to Heat Exchanger	Recirculation Solution from Heat Exchanger	Product Stream to Settling Tanks	Product Stream (g,i) to Centrifuge	Cooling Water to Heat Exchanger	Cooling Water from Heat Exchanger	Secondary Cooling Water	Dissolver Off-Gas	Diluted Dissolver Off-Gas	Rectifier			
Flow, ℓ/hr		57	4500	4500[(f)]	4500[(f)]	57	57	3.6×10^4	3.6×10^4	(g)						
Uranium, g/hr	1370															
Fissium, g/hr	70															
Aluminum, g/hr	1710															
Solids, g/hr[(a)]	84															
Uranium, g/ℓ			24	24	24	24	24									
Fissium, g/l[(b)]			1.2	1.2	1.2	1.2	1.2									
H^+, M		9.0	5.4	5.4	5.4	5.4	5.4									
NO_3, M		9.0	7.9	7.9	7.9	7.9	7.9									
Aluminum, M			1.1	1.1	1.1	1.1	1.1									
Flow, scfm											0.8	38				
Temperature, °F		70	130	125	115	125	120	104	107	107						
Pressure, psig		55		55	1	55	5	85	80	100						
SP.GR. @ 25°C		1.28	1.34	1.34	1.34	1.34	1.34									
NO_2, %											35	1.3				
H_2, %											3	0.1				
Amps													5000			
Volts													5-20[(h)]			
Gd, g/ℓ		3.9	3.9	3.9	3.9	3.9	3.9	2.6	2.6							
Undissolved solids, g/ℓ			0.6[(e)]	0.6	0.6	0.6	0.6									
U-235, g/ℓ			12.6	12.6	12.6	12.6	12.6									
$Hg(NO_3)_2$, M[(c)]		0.01	0.01	0.01	0.01	0.01	0.01									

(a) Solid material associated with the fuel.

(b) The concentrations of these streams include some undissolved solids.

(c) Optional.

(d) One can charged each 67 minutes.

(e) Fissium.

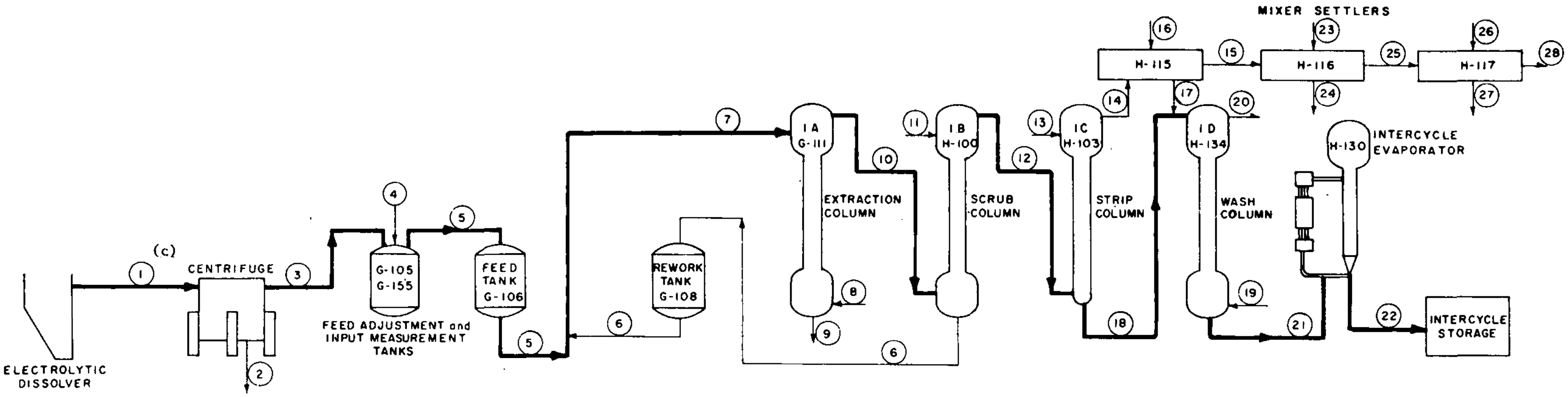
(f) Small flowrate of product stream is neglected.

(g) Flowrate varies.

(h) Voltage depends on resistance in dissolver, which is continuously changing.

(i) This becomes stream (1) on the accompanying extraction flowsheet.

Figure 6. Typical Material Balance for Dissolution of Skull Oxide in Aluminum Cans



STREAM	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19 & 20	21	22	23	24	25	26 & 27	28
DESCRIPTION	DISSOLVER PRODUCT	SOLIDS To PEW (c)	CENTRIFUGED DISSOLVER PRODUCT	FEED ADJUSTMENT SOLUTION	ADJUSTED DISSOLVER PRODUCT	IBR	IAF	IAX	IAR	IAP	IBS	IBP	ICX	ICR	ISR	ISX	ISW	ICP	IDX IDR	IDP	FIRST CYCLE PRODUCT	IISX	IISW	IISR	IIISX IIISW	IIISR (IAX)
Flow, ℓ/hr	57		57	43	100	50	150	300	150	300	50	300	200	300	300	50	50	200	10[(f)]	250	4.6	30	30	300	30	300
H^+, M	5.4		5.4	0.7	3.4	0.7	2.5		2.2	0.1	-0.2[(e)]	0	0.01			0.01	0.01	0.01		0.01	0.5				0.01	
NO_3^-, M	7.9		7.9	0.7	4.8	2.4	4		3.6	0.2	1.5	0.1	0.01			0.01	0.01	0.07		0.06	3.0				0.01	
Al, M	1.1		1.1		0.6	0.5	0.6		0.6		0.5															
Gd, g/ℓ	3.9		3.9		2.2		1.5		1.5				0.05					0.05		0.04	1.45					
Fissium, g/l[(a)]	1.2		0.6		0.4		0.2		0.2																	
U, g/ℓ	24		24		13.7	0.3	9.2		1.5×10^{-3}	4.6		4.6		8×10^{-4}	1×10^{-4}		5×10^{-3}	6.8		5.5	300		6×10^{-4}			
U-235, g/ℓ	12.6		12.6		7.2	0.2	4.8		7×10^{-4}	2.4		2.4		4×10^{-4}	5×10^{-5}		2×10^{-3}	3.6		2.9	158					
Solids, g/ℓ	34	34																								
NH_4, M						0.2	0.1		0.1		0.2															
TBP, %								10[(d)]		10		10		10	10									10		10
Na_2CO_3, M																						0.5	0.5			
SP.GR. @ 25°C							1.1889																			
$Hg(NO_3)_2$, M[(b)]	0.01		0.01		0.006		0.004		0.004																	

(a) The concentration of these streams include some undissolved solids.

(b) Optional.

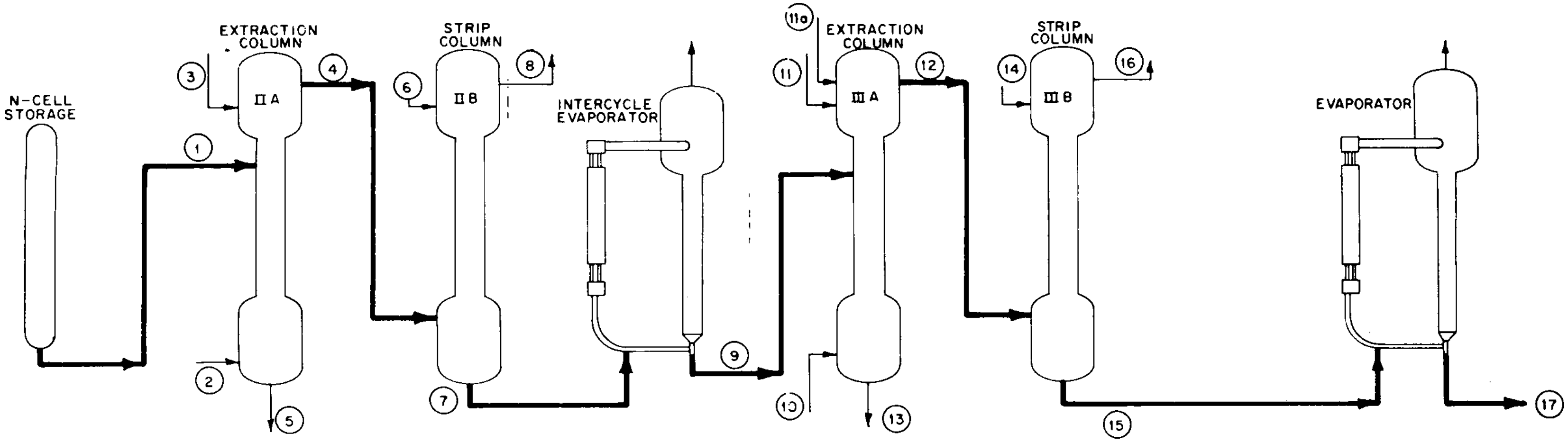
(c) Stream (9) on the dissolution flowsheet.

(d) Ten percent TBP in NPH.

(e) Acid deficient with NH_4OH.

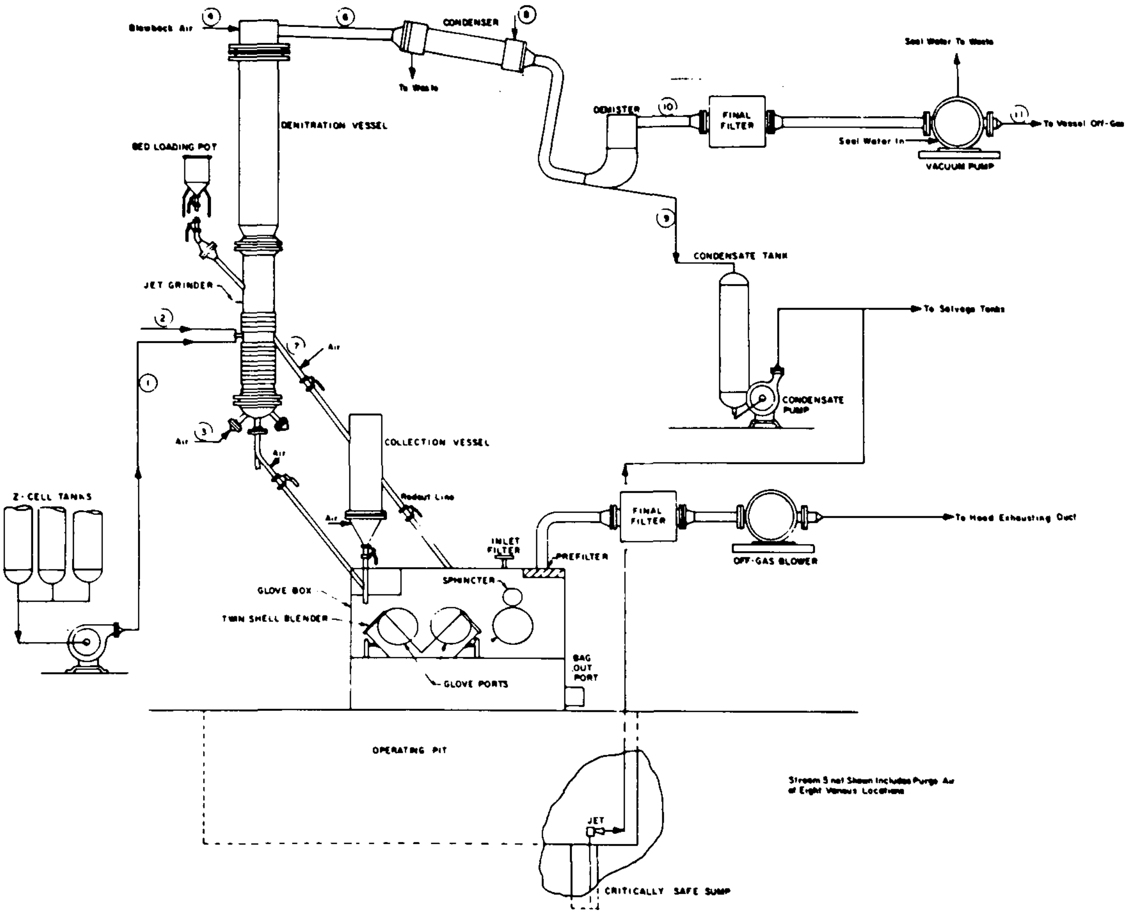
(f) AMSCO.

Figure 7. Typical Material Balance for First-Cycle Extraction of Skull Oxide in Aluminum Cans



STREAM	1	2	3	4	5	6	7	8	9	10	11	11a	12	13	14	15	16	17
DESCRIPTION	II AF	II AX	II AS	II AP	II AR	II BX	II BP	II BR	SECOND CYCLE PRODUCT	III AX	III AS1	III AS2	III AP	III AR	III BX	III BP	III BR	PRODUCT
FLOW, l/batch																		
FLOW, l/hr	11	50	40	51	50	30	31	50	9.5	51	37	4.5	51	51	25	25	51	9.5
SP GR	1.43	0.80	1.31	0.89	1.25	1.00	1.15	0.80	1.48	0.80	1.31	1.00	0.89	1.23	1.00	1.18	0.80	1.48
EXTRACTANT		Hexone		Hexone				Hexone		Hexone			Hexone				Hexone	
$Al(NO_3)_3$, M			2.00		1.60						2.00			1.45				
HNO_3, M	1.0					0.04	0.04		0.13						0.04	0.04		0.09
NH_4OH, M			0.55		0.22						0.32			0.20				
NH_4NO_3, M					0.22									0.04				
U, g/ℓ	300			65	5×10^{-3}		110	5×10^{-3}	350				65	5×10^{-3}		132	5×10^{-3}	350
$Fe(NH_2SO_3)_2$, M			0.08		0.06							0.08		0.06				
Gd, g/ℓ	1.45				0.32													

Figure 8. Typical Material Balance for Second- and Third-Cycle Extraction



STREAM	1	2	3	4	5	6	7	8	9	10	11		
DESCRIPTION	Uranyl Nitrate Solution	Air Flow To Nozzle	Fluidizing Air	Blowback Air	Total Purge Air to Denitrator (d)	Off-Gas From Denitrator	Product Overflow	Condenser Cooling Water	Condensate	Off-Gas To Final Filter	Off-Gas To Vessel Off Gas System		
Flow, l/hr	10(a)							1100	6.5				
Flow, SCFH		130	300(c)	35	80	973				672	672		
Flow, ACFH		329	844	97	202	3482				1800	810		
Temperature, °C	21	21	300	21	300	300	250	13	65	130	21		
Pressure, psia		11.3(b)	10.3	100	11.3	8.0				7.5	12.2		
U, g/l	350												
HNO_3, M	0.13								1.0				
Sp Gr @ 25°C	1.48								1.1				
O_2, g-moles/hr		35	80	10	21	161 (e)				161	161		
N_2, g-moles/hr		129	302	34	90	555				555	555		
NO-NO_2 as NO_3, g-moles/hr						31(f)			6.5	24.5	24.5		
H_2O, g-moles/hr	495					495			374	121	121		
UO_3 kg/hr						Trace	4.21		Trace	Trace			

(a) Maximum flowrate for denitration-varies from 6 to 10 l/hr as the operating bed temperature decreased from 400 to 300°C

(b) Pressure above the nozzle

(c) Fluidizing air required for particle size near 0.3 mm superficial velocity 1.2 ft/sec at vessel vapor space pressure (10.3 psia)

(d) Includes total from 8 purge lines at approximately 10 SCFH each at various locations in the system.

(e) Includes O_2 from reaction.

(f) Includes NO_2 from HNO_3 in feed.

Figure 9. Typical Flowsheet and Material Balance for Product Denitration

III. DISCUSSION OF CAMPAIGN

The fuel reprocessing campaign ran from February 1975 to April 1976. Approximately 1690 kg of uranium were charged to the electrolytic dissolver: 794 kg EBR-II Mark IA subassemblies, 650 kg clad fuel pins in aluminum cans, 60 kg skull oxide, 142 kg EBR-II Mark II fuel, 8 kg ingots, and approximately 36 kg sodium-contaminated scrap.

1. Fuel Information

1.1 EBR-II Mark IA Fuel Subassemblies

EBR-II Mark IA fuel subassembly sections are approximately 20-in.-long Type 304 stainless steel hexagonal shrouds containing 91 fuel elements. Fuel elements are 0.144-in.-diameter pins, 13.5 in. long, contained in 0.18-in.-diameter Type 304 stainless steel tubes. The tubes are 18.2 in. long with end fittings.

The composition of the fueled portion of a Mark IA subassembly section is:

5.62 kg U (52.5% enriched before burnup)
0.069 kg Na
0.30 kg fissium[a]
2.55 kg stainless steel Type 304 (cladding and shroud)

Some 27% of the EBR-II Mark IA subassemblies were control rods containing 61 fuel elements and 30 stainless steel dummy elements. These subassemblies contained 3.76 kg U, 0.46 kg Na, and 0.20 kg fissium.

1.2 Clad Fuel Element in Aluminum Cans

Aluminum[b] cans (3-1/2 in. OD x 23 in. long) weighing 2.4 kg empty and containing clad Mark IA fuel elements were processed during the campaign. The cans contained no more than 3.02 kg of ^{235}U in 52.6 percent enriched uranium. They had screwed lids and were filled or partially filled with water from the fuel storage basin.

1.3 Skull Oxide in Cans

Oxidized residues from EBR-II melt refine process (called skull oxide) were processed in aluminum cans (3-1/2 in. OD x 17 in. long) weighing 1.9 kg empty. The cans contained no more than 900 grams of ^{235}U at an enrichment of 52.6 to 70 percent. Some of the cans also contained water.

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[a] Elements other than U which are deliberately added to the fuel during fabrication—Mo, Ru, Rh, Pd, Zr, Nb.

[b] Type 6061-T6 aluminum.

1.4 Miscellaneous EBR-II Fuels

These fuels include aluminum cans of uranium scrap, Na-contaminated fuel, one canned ingot, and some oversize cans too large to fit into the dissolver. There were also some EBR-II Mark II subassembly sections, canned and uncanned. Some APPR and other scrap fuels were also processed during the campaign.

1.4.1 Ingots in Cans. Uranium melted into ingots contained in aluminum cans (3-1/2 in. OD x 17 in. long) weighing 1.9 kg empty were processed. The cans contained no more than 3.02 kg of ^{235}U at 52.6 to 70 percent enriched uranium. They had screwed lids and were filled or partially filled with water from the fuel storage basin.

1.4.2 Na-Contaminated Fuel. Seven cans of EBR-II scrap elements with exposed sodium and/or fuel meat were processed. This scrap was shipped to the ICPP in special shipping cans consisting of two containers: an inner perforated container which holds the fuel elements, and an external container which is sealed airtight. Only the inner container (2-3/4-in.-OD 20 gage stainless steel tube x 18-3/4 in. long) was charged with the fuel. The external container (3-in. schedule 40 pipe x 18-3/4 in. long) was discarded in the cell and subsequently removed or (optional) discharged to the dissolver at a later date. The average enrichment of the fuels in the cans was 70%, but some pieces of fuel had enrichments as high as 93%.

1.4.3 Oversize Cans of Fuel. Five EBR-II subassemblies in 5-in. (OD) by 36-in.-long cans and four EBR-II Mark II subassemblies in 4.5 in. (OD) by 28-30-in.-long cans were processed at ICPP. This fuel was no more than 70% enriched in ^{235}U. These cans did not fit into the dissolver or in the fuel insert in the High Load Charging Cask. The cans were transported in the charger without the insert under special procedures. The fuel was removed from the cans and then charged to the dissolver.

1.4.4 EBR-II Mark II Fuel Subassemblies. EBR-II Mark II fuel subassembly sections are approximately 26-in.-long Type 304 stainless steel hexagonal shrouds containing 91 fuel elements. Fuel elements are 0.130-in.-diameter pins, 13.5 in. long, contained in 0.18-diameter Type 316 stainless steel tubes. The tubes are 24.2 in. long with end fittings. The composition of the fueled portion of Mark II subassembly section is:

4.47 kg U (66.67% enriched before burnup)
0.12 kg Na
0.23 kg fissium
2.39 kg stainless steel Type 316 (cladding)
155 kg stainless steel Type 304 (shroud)

1.4.5 Stainless Steel Scrap. Two sealed, 4-1/4 in. x 7 in. cans of stainless steel scrap were charged to the dissolver on April 28, 1976. The cans were:

1 steel can containing ss/UO_2 scrap
wire and pins (178 g U; 164 g ^{235}U)
1 steel can containing ss/clad scrap
plates and pins (66 g U; 63 g ^{235}U

The Safety Review Board (responsible for approving all operations with respect to plant and personnel safety) granted approval to process this material under the "400 gram" rule rather than under a formal safety package. The fuel was charged to the dissolution process cell after the uranium was flushed from the equipment according to standard operating procedures. The normal dissolvent contained a minimum of 2.6 g/L Gd; the uranium was accounted for (defined as measuring at least 200 g of the total 227 g ^{235}U) in downstream process cells before more fuel was charged to the dissolution process cell.

1.4.6 APPR Cold Fuel Scrap. A small amount of APPR fuel (a stainless steel UO_2 cermet type fuel, 93% enriched in ^{235}U) was charged to the dissolver. The APPR fuel charged to the dissolver consisted of loose steel plates. The outside dimensions are 2.86 in. x 27 in. long; the fuel cladding is Type 304 stainless steel with Type 302B stainless steel/UO_2 cermet fuel meat.

2. Dissolution

2.1 Production Rates and Compositions

2.1.1 Mark IA Fuel Subassemblies. During the campaign, 314 Mark IA fuel subassembly sections, 84 of which were control subassemblies, were dissolved. The dissolution time for 174 subassemblies was 397 h, almost 32 kg ^{235}U/d. The total uranium dissolution rate was 60.9 kg/d, 122% of flowsheet value. Table I shows the composition of the dissolver product as indicated by routine samples taken during February and April 1975. Table II shows the composition of the dissolver product as indicated by routine samples taken during December 1975.

2.1.2 Clad Pins in Cans. Throughout the campaign, 231 cans of clad pins were processed. All of the clad pins were Mark IA fuel elements. The dissolution time for 83 cans of clad pins was 315 h, almost 19 kg ^{235}U/d. The total uranium dissolution rate was 35.7 kg U/d, 71.3% of flowsheet value. A total of 650 kg of ^{235}U, and 554 kg of Al was processed in the form of clad pins in aluminum cans. The acid dissolvent contained 0.01 $\underline{M}$ $Hg(NO_3)_2$ as a catalyst for the dissolution of aluminum. The aluminum alloy used in can fabrication was Type 6061-T6. Previous studies[2] indicated that a concentration of 0.01 $\underline{M}$ $Hg(NO_3)_2$ was necessary for the transition from the passive to the active condition for the 6061 alloy.

2.1.3 Skull Oxide in Cans. Ninety cans of skull oxide were processed: 78 between February 4 and 8, 1975; twelve during December 1975. Approximately 60 kg of 52 to 70 percent enriched uranium and 171 kg of aluminum cans were dissolved. The dissolution rate of the 78 cans processed in February was 9.98 kg ^{235}U/d, 58% of flowsheet value. Typical dissolver product composition is given in Table III.

TABLE I

AVERAGE DISSOLVER PRODUCT COMPOSITION - FUEL ASSEMBLIES[a]

Specific Gravity	1.41	±	0.04
HNO_3, M	3.33	±	1.09
Uranium, g/L	80.56	±	24.7
Stainless Steel g/L	42.5	±	17.1
Al, M	0.35	±	0.23
Conductivity, mhos/cm	0.234	±	0.08

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[a] The data were averaged and the results presented above are $\bar{X} \pm \sigma$

TABLE II

AVERAGE DISSOLVER PRODUCT COMPOSITION - CLAD PINS IN ALUMINUM CANS[a]

Specific Gravity	1.37	±	0.004
HNO_3, M	3.78	±	1.43
Uranium, g/L	44.2	±	20.0
Stainless Steel g/L	13.45	±	7.58
Al, M	0.85	±	0.25
Conductivity, mhos/cm	0.22	±	0.12

---

[a] The data were averaged and the results presented above are $\bar{X} \pm \sigma$

TABLE III

AVERAGE DISSOLVER PRODUCT COMPOSITION - SKULL OXIDE[a]

Specific Gravity	1.35	±	0.01
HNO_3, $\underline{M}$	3.14	±	0.47
Uranium, g/L	24.9	±	4.2
Al, $\underline{M}$	1.35	±	0.10
Conductivity, mhos/cm	0.17	±	0.02

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[a] The data were averaged and the results are presented as $\overline{\chi} \pm \sigma$.

2.2 Product Clarification

Clarification of the electrolytic dissolver product in the first electrolytic dissolution campaign of EBR-II fuel at ICPP[3] was accomplished by passing the solution through 8-in.-diameter settling tanks and the ICPP headend centrifuge. After this campaign the settling tanks were removed because their solids removal efficiency was less than expected and the presence of solids in the dissolver product appeared to cause no processing problems in downstream equipment. It was expected that the centrifuge would be required to remove the entire suspended-solids load in future campaigns. However, the centrifuge broke down during the 1974 coprocessing campaign, and it was decided to accelerate installation of new centrifuges rather than repair the old.

Pilot plant experience shows the electrolytic dissolver product contains approximately 3 wt.% suspended solids.[4] In the first EBR-II processing campaign, the suspended solids concentration in the sludge tank was 3.1 wt.%[3]. Based on previous experience, the suspended solids were assumed to be about 3 wt.% for similar fuel processed in this campaign.

2.3 Soluble Nuclear Poison

Since product streams enter several nongeometrically safe vessels during the process, a soluble nuclear poison (gadolinium) is required in the fresh acid feed to the dissolver. Gadolinium concentrations ranged from 2.6 to 4.3 g/L (see Figures 2 through 6).

A nuclear poison detection system is used to monitor the nuclear poison. The instrument determined poison concentrations in the region of 2.0 g/L Gd and has an error of less than 1%. The neutron count rate for neutrons passing through the soluble nuclear poison from a neutron source is measured to determine the poison concentration. A functional description of the instrument has been reported elsewhere.[1]

2.4 Off-Gas Analysis

The electrolytic dissolution process off-gas was sampled for tritium concentrations during this campaign. From April 14 to April 16, 1975 three off-gas samples were taken from the decontamination line on the demister. The sample was pumped from the decontamination line through a sampling system consisting of two 150 cc sample bombs connected in series and separated by a Drierite bed. After purging the system for about five minutes, the entrance and exhaust valves to the bombs were closed and the system disassembled. The tritium sample in the first bomb (upstream of the drying bed) was considered to contain total tritium (water vapor, HTO and hydrogen gas, HT). The bomb located downstream of the drying bed contained only HT. Tritium in the vapor phase (HTO) was calculated by difference.

The tritium sampling in the off-gas indicated about 99 percent of the tritium occurred as HTO. The tritium activity during this sample period was about 1.7×10^8 disintegrations per second in the dissolver off-gas. Using this number to project the stack release, it means that approximately 1.4×10^{-6} μCi/mL was leaving the ICPP stack.

During dissolution about 90 percent of the total tritium was released to the off-gas (using a calculated material balance between off-gas flow and the adjusted dissolver product feed rate). The 10 percent entering the extraction system was carried to high-level liquid waste storage with the fission products in the raffinate. The organic recycle stream and the product showed no tritium.

A contamination problem in the off-gas sample line necessitated the termination of sampling during this campaign. A new sampling system should be developed to eliminate problems caused by high relative humidity when sampling from the demister.

2.5 Dissolver Heel Out

The dissolver was heeled-out on a number of occasions during the campaign. The first heelout was started February 17 and completed February 25, 1975. The dissolver process was shut down for recirculation pump repair due to the increasing severity of pump vibration. Due to the pump vibration, flush connections to two valves failed during the heelout. Therefore, the pump could be operated only sporadically for the remainder of the heel-out.

The second heelout took place during the period April 27 to May 26, 1975. During this time, the N-cell inventory of uranium was processed through the second- and third-extraction cycles and denitration systems. The first-cycle extraction and hexone systems were cleaned out prior to shutdown. The plant was down for 10 weeks due to the Atmospheric Protection System (APS) tie-in. Operation of the dissolver resumed on September 30, 1975.

On two occasions (October 10 and 31, 1975) the electrolytic dissolution system was shut down because the extraction system feed tankage was full. At the end of the campaign the dissolver was again heeled-out. The heelout was completed on May 23, 1976; the dissolver was given one water rinse and shut down.

2.6 Dissolution Problems

During startup of the campaign, in February 1975, various minor problems occurred. The most significant of these was severe vibration of the recirculation pumps, which caused the system to be shut down; the campaign was resumed with use of the spare pump. On February 15 the second recirculation pump began vibrating slightly and the system was shut down on February 17. Subsequent to this a leak developed in a decontamination line that connects to the bottom of the recirculation loop control valve and eventually the valve broke off completely. It was obvious that the vibration of the pump caused the problem.

To eliminate vibration problems, the pumps were modified. The modifications included a change in the liquid takeoff system from the impeller and a recirculation system with a manual valve. This recirculation system allowed the outlet pressure, which has an effect on vibration, to be adjusted. The pumps performed adequately during the remainder of the run.

During February 1975, it was recognized that dissolution in the center of the dissolver was not as good as at the ends of the dissolver. The basket was found to be bowed and in contact with the dissolver side. A Kynar spacer inserted between the basket and the dissolver increased the dissolution rate in the basket center comparable to that in the basket ends.

Other minor problems, not directly related to the dissolution process were:

(1) The dissolution of oversized cans in February 1976 slowed down the processing rate. The lids to the oversize cans were removed in the dissolver process cell and the subassemblies were charged to the dissolver.

(2) Poor visibility in the fuel storage basin also slowed down the processing rate in March 1976. At that time, 40 Mark II subassemblies were scheduled for processing. Before being processed, the subassemblies had to be canned in the basin; this required excellent underwater visibility.

(3) Leaks developed in the heat exchanger for the new 4000 amp rectifier and the rectifier was shut off for brief periods to allow repair.

(4) The secondary heat exchanger for the closed gadolinium cooling loop was fouled with mineral deposits from the cooling water. These were removed by flushing with chemicals.

3. First-Cycle Extraction

After leaving the dissolver, the product solution entered uranium accountability tanks (G-105 and G-155) for feed adjustment and uranium accountability. From these tanks it was sent to a feed tank (G-106) and then air-lifted to the first-cycle extraction column (G-111). The dissolved uranium was partitioned from the associated metals and fission products in the column using ten percent tributyl phosphate (TBP) in a normal paraffin hydrocarbon (NPH) solvent. Nitric acid carried out of the column in the organic stream was neutralized and removed in the scrubbing column (H-100) by contact with 0.20 $\underline{M}$ NH_4OH and 0.5 $\underline{M}$ $Al(NO_3)_3$. The uranium was stripped from the organic phase by contact with an aqueous stream containing 0.001 $\underline{M}$ HNO_3. Any residual TBP was removed from the uranium product stream leaving the stripping column by contacting it with Amsco (125-90W hydrocarbon) in the wash column (H-134). The aqueous product stream was then concentrated to 300 g/L U in the product evaporator (H-130) and stored in the intercycle storage tanks (N-cell) for later processing through two more cycles of extraction.

Typical flowrates and compositions during first-cycle extraction are shown in Figures 3 through 7. Performance of important components of the first-cycle extraction system are discussed below.

3.1 Waste Generation

The high-level waste volume (IAR stream) generated during this fuel processing campaign was approximately 396,000 liters. The total amount of uranium lost, including estimated losses due to undissolved solids, was 2,160 grams. The total amount of ^{235}U processed (according to accountability figures) was 1690 kg. Since the major portion of the fuel was 50 to 70 percent enriched in ^{235}U, at most (considering the lost uranium to be 70 percent enriched) 0.091 percent of the ^{235}U processed was lost to the waste stream.

3.2 Extraction Efficiency

The uranium (^{235}U) recovery in the first-cycle extraction column was at least 99.9 percent. The number of theoretical stages in the column was determined using a constant distribution coefficient (E_A) of 10. The number of theoretical stages was at least 4.1.

3.3 Tritium in the First-Cycle Extraction System

From April 10 to April 22, 1975 three sets of liquid samples were taken from the first cycle extraction system . Each set consisted of samples from the adjusted dissolver product (IFu), the raffinate (IAR), an organic recycle stream (ICR), and the product stream (ICP). Only 10% of the total tritium released during dissolution entered the first-cycle extraction system. Off-gas sampling indicated that 99 percent of the tritium in the vapor occurred as HTO. At least two-thirds of the tritium entering with the adjusted dissolver product eventually leaves with the raffinate stream. No more than 0.039 percent (via the ICP) leaves with the first-cycle product.

3.4 Undissolved Solids in the First-Cycle Extraction System

In this fuel processing campaign the centrifuge was inoperable. As a result, large amounts of solids were deposited throughout the first-cycle extraction system. These solids (mainly siliceous fissium solids) appeared to plug the packed columns and reduce their efficiency. The second- and third-cycle columns and the K-cell stream stripper, as well as the first-cycle columns, were detrimentally affected.

Various tanks in the electrolytic process were sampled for solids. The samples are listed in Table IV.

4. Second- and Third-Cycle Extraction Systems

4.1 Dual Scrub Operation

The flowsheet for the second- and third-cycles of extraction is shown in Figure 8. A demineralized water secondary scrub in III-A column (Q-102) minimized the carry-over of metallic ions and thus increased the chemical purity of the product. When water was used for the secondary scrub solution, the aluminum nitrate primary scrub flowrate had to be increased to maintain adequate salting strength in the extraction section of the column. Use of the dual scrub operating mode results in an average aluminum contamination of 169 ± 157 ppm (based on U and averaged over 58 cans of denitrator product). The concentration of contaminants, other than aluminum, that form nonvolatile-fluorides (Cr, Cu, Fe, Mn, Mg, and Na) averaged approximately 156 ± 94 ppm ($\chi \pm \sigma$), as compared to the first campaign, where the concentration of these contaminants was approximately 200 ppm.

4.2 Plutonium Decontamination

Since the final uranium product from the campaign was to be sent to Portsmouth and/or Oak Ridge, where further processing of the UO_3 would involve contact operations, a very low transuranic alpha activity was required (less than 5000 disintegrations per minute per gram of uranium), as compared to product produced in previous campaigns (2.5×10^5 d/m/g U). In view of the high plutonium extraction efficiency (greater than 90%) expected in the first extraction cycle (i.e., low decontamination), meeting this specification required good Pu decontamination in both the second- and third-extraction cycles. Ferrous sulfamate has been routinely used in the second-cycle scrub to reduce

TABLE IV

UNDISSOLVED SOLIDS IN THE FIRST-CYCLE EXTRACTION SYSTEM

Vessel[a]	UDS (g/L)	Comments
G-106	6.5	All samples were black with exceptions of G-113 samples and the H-126 sample.
G-106 sparged	7.9	
G-105	2.9	
G-105 sparged	1.612	The G-113 samples were clear with no solids visible.
	10.0	
G-113	11.3	
After G-106 sparging		
H-108	none	The H-126 sample had a few solids floating in it.
H-126	0.52	
H-131	27.1	

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[a] See Figure 6 for identification of each vessel.

The undissolved solids content (when present) ranged from 0.52 g/L to 11.3 g/L.

the plutonium to inextractable Pu(III), thus diverting it to the aqueous raffinate, but has not been used in third-cycle because of the undesirable iron contamination that would result in the final product. In this campaign, however, the aforementioned water scrub entering the IIIA column above the aluminum nitrate scrub (III AS) allowed the use of ferrous sulfamate in the III AS, greatly enhancing Pu decontamination in the third extraction cycle.

Plutonium decontamination factors[a] (df's) were determined from analysis of process samples for each of the extraction systems. The decontamination factor for the first-cycle extraction system was an average 2.79 ± 0.55 (data taken in November and December 1975). The decontamination factor for the second-cycle extraction ranged from 1 to 55,400, with an average (discarding those values indicating a column upset or possible analytical error) of 135 ± 48.3. The decontamination factor in the third-cycle extraction system rangedfrom 0.19 to 2920, with an average of 1630 ± 640. The plutonium decontamination provided by the second- and third-cycle extraction systems resulted in acceptable product quality.

5. Fluidized-Bed Denitration

The denitrator was operated three times during this campaign to convert the uranyl nitrate product from the hexone extraction system into UO_3. The operating periods were November 13 to November 29, 1975; February 26 to March 7, 1976; and all of November 1976. Approximately 1700 kg of ^{235}U were packaged in this campaign. The overall processing rate was 1.03 kg ^{235}U packaged per hour of denitrator operating time.

The concentrated uranium product stream from the extractive purification systems is processed through the denitrator to form a solid uranium oxide. In the fluidized denitrator, the uranyl nitrate is converted to uranium trioxide by the following reaction:

$$UO_2(NO_3)_2 \xrightarrow[\text{heat}]{} UO_3 + NO_2 + NO + O_2.$$

The solid UO_3 is then packaged and stored to await shipment to fuel fabrication facilities.

5.1 System Performance

All of the denitrator runs associated with the second electrolytic dissolution campaign had instrument, pressure probe, nozzle restriction, thermocouple and particle size control problems. The denitrator off-gas vacuum pump performed at only one-third capacity. One of the three bed heaters failed during the run.

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[a] The decontamination factor for a contaminant is the ratio of the contaminant weight per unit weight of U in the feed to the contaminant weight per unit weight of U in the product for a particular process.

5.2 Product Characteristics

The denitrator product normally contains trace amounts of Al and other contaminated salts that form nonvolatile fluorides. The average concentration of these contaminant and the transuranic levels are shown in Table V. The transuranic levels cannot exceed 5000 d/m/g U for packaging and shipping.

TABLE V

DENITRATOR PRODUCT IMPURITIES

Contaminant	Concentration ($\mu g/g$ U)
Al	169 ± 157
Cr	10.1 ± 5.7
Cu	6.6 ± 5.0
Fe	68.4 ± 57.8
Mg	2.2 ± 3.2
Mn	<5
Na	64 ± 28.7
Ni	11.4 ± 10.8
Ca	12.5 ± 7.5
Total Transuranics	1820 ± 1300 d/m/g U[a]

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[a] Disintegrations/minute/g of uranium.

IV. RECOMMENDATIONS

1. Past processing campaigns of EBR-II fuel without the plant centrifuge have resulted in fissium solid deposition throughout the extraction system. This deposition has resulted in poor operation of the first-cycle extraction system and the K-cell steam stripper and allowed higher than normal uranium losses to the raffinate streams from the second- and third-cycle extraction systems. Laboratory studies indicate aging the dissolver product at high temperatures and high concentrations of HNO_3 reduces the amount of fissium solids transferred to the organic phase in the first-cycle solvent extraction; this procedure could be used in the plant if the certrifuge is not available for the next campaign.

2. Oversize cans are inconvenient and slow the processing rate of the dissolver; the use of such cans should be avoided.

3. There is insufficient vacuum on Cell 5. This should be improved to give added protection against airborne contamination into the plant from that cell.

4. During the next decontamination of Cell 5, the spare slave manipulators without boots should be installed in that cell. One of the slave manipulators (with boots) is broken and cannot be removed from the cell because of the boots.

5. The electromagnetic (EM) flowmeter failed for the second time in the history of electrolytic processing. The EM flowmeter should be removed or replaced with something more reliable.

6. The possibility of installing spray nozzles in strategic locations in the system to aid in decontamination efforts should be investigated.

7. A remote, moveable TV camera in the electrolytic dissolution cell is recommended for use in closeup viewing of fuels and equipment.

8. A moveable charging area shield with a winch and manipulator would be helpful in removing materials from the electrolytic dissolution cell.

9. The nuclear poison detection system (NPDS) should be removed from inside the acid feed tank to the acid feed line.

10. The denitration system should be upgraded to alleviate persistent problems with bed-heating, particle growth, off-gas vacuum and pressure tap restrictions.

11. An automatic, constant monitor for the electrolytic dissolver product composition would be extremely helpful in operating the electrolytic dissolution system. Benefits from the constant monitor would be: (a) drastic reduction in sampling frequency and, thus, a reduction in analytical personnel radiation exposure; (b) a more accurate or automatic feed makeup for the first-cycle extraction system; and (c) probable elimination of many of the administrative safety controls now imposed on the electrolytic dissolution and extraction systems.

12. Radiation monitors should be installed in various parts of the electrolytic cell to reduce radiation exposure to health physics personnel during survey.

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