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HOT SEMIWORKS SUMMARY

RUN PX-13

By

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August 24, 1956

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1.0 INTRODUCTION

During Run PX-12 acceptable HA Column decontamination was demonstrated; however, generally under unstable column operating conditions. The principal objectives of PX-13 were:

- a. To duplicate the high PX-12 decontamination factors under stable HA Column operating conditions.
- b. To study the effects of fresh solvent, IO Column CP carbonate washed solvent, and IO Column Technical grade carbonate washed solvent on A Column decontamination factors.
- c. To study the effect of increasing the HA, HC, and IA uranium processing rates to 14 tons per day (plant equivalent).
- d. To study the effect of an intermediate 2A Column scrub on UX₁ decontamination.
- e. To study the effect of sulfamic acid addition to the HAF, HAS, IAF, and IAS and NaNO₂ omission from the HAF and IAF.

2.0 SUMMARY

Changing from fresh solvent (30% TBP in Shell E-2342) to normal IO Column carbonate washed recycled solvent, decreased the HA Column laboratory decontamination factors from approximately 2000 to 600-1000. HA and IA Column laboratory decontamination factors (600-1000 and 60-180 respectively) remained essentially constant for recycled solvent contacted in the IO Column with either CP or Technical grade sodium carbonate. Intermediate scrub introduction to the 2A Column resulted in 8-23% 2AW plutonium losses (based on 2AF) due to the short (8 ft.) 2A Column extraction section.

Increasing the HA Column plant equivalent operating rate from 10 to 14 tons U/day permitted the scrub section to operate closer to its flooding amplitude-frequency product and improved the decontamination by a factor of approximately 2.

No significant plutonium loss can be attributed to the omission of sodium nitrite from the HAF and the addition of sulfamic acid to the HAF and HAS. However, high IAW plutonium losses (7-30 times the Purex Flowsheet HW #3 value of <0.2%) may quite possibly be attributed to the omission of sodium nitrite from the IAF and the addition of sulfamic acid to the IAF and IAS streams.

The principal solvent extraction data are summarized in Table 2.1. More detailed results appear in Tables 5.1 to 5.3 and Figures 5.1 to 5.4.

TABLE 2.1
SOLVENT EXTRACTION SUMMARY DATA(1)

<u>Solvent Treatment</u>	<u>HA Freq.</u>	<u>Hours of Oper.</u>	<u>PRECYCLE(2)</u>		<u>Losses</u>			
			<u>dF (HAF = 0.0)</u>		<u>HAW</u>		<u>HCW</u>	
			<u>HAF(3)</u>	<u>HCP</u>	<u>% U</u>	<u>% Pu</u>	<u>% U</u>	<u>% Pu</u>
Fresh HAX - Batch CP Na ₂ CO ₃ Wash	70	16	3.5	3.5	0.6	0.3	0.003	0.02
IO Column Contact - CP 0.25 M Na ₂ CO ₃	60	3.5	2.9	2.9	0.7		0.01	0.004
" " " "	70	9.5	3.0		0.1	0.8		
" " " "	76	5.0(4)	2.8	2.9	4.6	4.7		0.007
IO Column Contact - Tech. 0.25M Na ₂ CO ₃	76	7.0(4)	2.8	2.8	0.3	2.6	0.008	0.006

<u>Solvent Treatment</u>	<u>IA Freq.</u>	<u>Hours of Oper.</u>	<u>PARTITION CYCLE(5)(6)</u>				<u>IAW Losses</u>	
			<u>dF (IAF = 0.0)</u>				<u>IAW Losses</u>	
			<u>IAP(7)</u>	<u>IBP</u>	<u>IBU</u>	<u>ICU</u>	<u>% U</u>	<u>% Pu</u>
IO Column Contact - CP 0.25 M Na ₂ CO ₃	60-72	19	1.9-2.2	1.9	2.4	2.8	0.001	1.8-3.5
" " " "	62-66	10.5	1.3-2.0	2.3	2.6	2.7	0.002	1.5
IO Col. Contact - Tech. 0.25M Na ₂ CO ₃	66	5.0	1.9				0.0006	6.5
" " " "	70-80 ⁽⁸⁾	6.0(9)	2.2	2.0	2.3		0.05	10.1

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TABLE 2.1 (Continued)

FINAL PLUTONIUM CYCLE(10)

<u>2AIS(11) Flow (HAF = 100)</u>	<u>Hours of Oper.</u>	<u>dF (IAF = 0.0)(6)</u>			<u>2AW(12) % Pu</u>
		<u>2AF</u>	<u>2AP</u>	<u>2BP</u>	
0.0	28.5	1.9	3.9	4.4-5.4	0.2-1.2
5.0	20.0	2.0	4.0	5.3	2.2-11.0
7.0	16.5	2.0	4.1	5.3	12.6-23.0

Notes:

1. Based on laboratory results.
2. HA (ex) total flow = 691 gal./ (hr.)(sq.ft.). (14 tons of uranium per day plant equivalent.)
3. For 3.5 dF the HAF fission product distribution was 60-65% ZrNb and 35-40% Ru. For 2.8-3.0 dF the HAF fission product distribution was 75-90% ZrNb and 10-25% Ru.
4. Uncentrifuged HAF used during this portion of run. HAF temperature = 18°C.
5. IA (ex) total flow = 691 gal./ (hr.)(sq.ft.). (14 tons of uranium per day plant equivalent.) All partition cycle columns operated at 14 tons of uranium per day plant equivalent. IA Column amplitude = 1.2 in. (ext. section) except as noted.
6. Average HAF/IAF dF = 2.9.
7. IAP fission product distribution = 80-90% ZrNb and 10-20% Ru.
8. IA Column extraction section amplitude = 1.07-inch.
9. IA Column cyclic flooding during this entire portion.
10. 2A (ex) total flow = 265 gal./ (hr.)(sq.ft.) for 0 ml./min. 2AIS flow. (10 tons of uranium per day plant equivalent.)
11. 2AIS = water. 2AIS introduced as an intermediate scrub stream.
12. 2AW losses are based on 2AF plutonium concentration.

TABLE 2.2
RUN CHRONOLOGY

<u>Date</u>	<u>Time</u>	
1-11-56		Charged 124 8-inch slugs.
1-15-56	0610	Jacket removal and dissolution.
to 1-18-56	0910	
1-18-56	0715	HA-HC Column started at 14 T/D with fresh HAX.
	2350	Switched to IO Column CP Na ₂ CO ₃ washed HAX.
1-19-56	1440	Started using uncentrifuged HAF.
	1945	Switched to IO Column Technical Grade Na ₂ CO ₃ washed HAX.
1-20-56	0240	Shut down HA-HC cycle. HAF temperature has been 18°C since 1600 1-19-56.
	0640	IA Column started (a = 1.2") at 14 T/D with IO Column CP Na ₂ CO ₃ washed IAX.
	0910	Started IBX, IBS, and IC Columns.
	0910-1400	IBS Column flooding out bottom.
	1235-1705	IA Column cyclic flooding.
	1420	Started 2A-2B Columns.
	2320	IA top DP cell increasing.
1-21-56	0115	IA Column flooding in scrub section. Reduced IA frequency to 70.
	0330	IA Column stable at frequency = 62.
	0820	Shut down IBX, IBS, and IC Columns.
	1205	Switched to IO Column Technical Grade Na ₂ CO ₃ washed IAX and 2AX.

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<u>Date</u>	<u>Time</u>	<u>TABLE 2.2 (Continued)</u>
1-21-56	1230	Started IBX, IBS, and IC Columns.
	1645	Reduced IA Column amplitude from 1.2 in. to 1.07 in.
	1735	IA Column flooding.
	1840	IBS Column flooding out bottom. Reduced IBS Column frequency to 40.
	1905	Shut down IBX, IBS, and IC Columns.
	1910	Started 2AIS at 15 ml./min.
	2045	Started IBX, IBS, and IC Columns.
	2210	IBS Column flooding. Reduced IBS frequency to 30.
	2250	Shut down IA Column.
1-22-56	0200	Shut down IBX, IBS, and IC Columns.
	0640	Started IBX, IBS, and IC Columns.
	1115	Shut down IBX, IBS, and IC Columns.
	1500	Increased 2AIS to 21 ml./min.
1-23-56	0740	Shut down 2A-2B cycle.

3.0 EQUIPMENT AND FLOWSHEET

No equipment changes were made between runs PX-10 and PX-13. The column dimensions and operating conditions are given in Table 3.1 for 14 T/D rates in the Precycle and Partition cycle columns and 10 T/D rates for the Second Plutonium cycle and Solvent Treatment columns. Due to the feed centrifuge failure, the last 12 hours of the run was operated with uncentrifuged HAF.

Purex Flowsheet - HW #3 was used with the following modifications:

- (1) HAS and IAS = 3.0 M HNO_3 - 0.5% $\text{NH}_2\text{SO}_3\text{H}$.
- (2) HAF and IAF = 1.35 M U, 2.0 M HNO_3 and 0.5% $\text{NH}_2\text{SO}_3\text{H}$.
- (3) HA Column single scrub operation; HAF:HAS:HAIS = 100:67:0.
- (4) No H_2O or NaNO_2 was added to the No. 1 waste concentrator.
- (5) NaNO_2 addition to the 2AF only.
- (6) IOF:IOS:IOR = 100:56:44.
- (7) 2AIS flow (H_2O) was 0 during the first portion of the run, 5.0 during second portion of run, and 7.0 during last portion of run. (HAF flow = 100).

Flow rates and composition of streams entering the columns are listed in Table 3.2. A schematic equipment flowsheet for the Hot Semiworks is shown as Figure 3.1.

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TABLE 3.1
COLUMN OPERATING DATA

Column	Packed Height ft.	Inside Diam. In.	Packing	Hole Diam. In.	% Free Area	Plate Spacing In.	Interface Location	Amplitude Inches ⁽¹⁾	Freq- uency CPM	Total Vol. Vel. Gal/hr/ft ²
HA (e)	8	3.07	S.S. Plates	(2)	(2)	(2)	Top	1.07	60-76	700
(s)	13	4.03	S.S. Plates (3)	0.125	23	2		0.60		330
HC	10	4.03	S.S. Nozzle Plates (4)	0.125	10	4	Bottom	0.50	100	800
IA (e)	8	3.07	S.S. Plates	(2)	(2)	(2)	Top	1.07-1.2	60-80	690-730
(s)	13	4.03	S.S. Plates (3)	0.125	23	2		0.60-0.70		330
IBX	18	4.03	S.S. Plates	0.125	23	2	Top	0.8	40	330
IBS	12	1.05	S.S. Plates	0.125	23	2	Top	0.8	30-50	920-1110
IC	10	4.03	S.S. Nozzle Plates (4)	0.125	10	4	Bottom	0.5	100	825
2A (e)	8	1.61	3/8" Fluorothene Raschig Rings				Bottom	1.0	50	265-290 ⁽⁵⁾
(s)	8	1.61	" "	" "	" "					
2B	10	1.05	S.S. Plates	0.06	21	2	Top	1.0	40	175 ⁽⁵⁾
IO	16	5.05	S.S. Nozzle Plates (4)	0.125	10	4	Bottom	0.5	70	560-620 ⁽⁵⁾

(1) Amplitudes are based on total cross sectional area of the columns. The Raschig Ring packing in the 2A Column increases the actual column amplitude to approximately 1.5 inches.

(2) "Graded Cartridge", see Figure 3.2 (HW-43074 RD) for details of HA and IA cartridges.

(3) Anti-backmixing plate at feed tee (See HW-39517 RD for details of construction).

(4) Nozzle depth = 0.035 inches.

(5) 2A, 2B, and IO Columns operated at 10 T/D. All other columns operated at 14 T/D.

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TABLE 3.2

FLOW RATES AND COMPOSITIONS
OF ENTERING STREAMS

Stream	Composition, G/L	Flow, ML/Min.	
		FX-13(1)	HW#3(2)
HAF	U = 337; HNO ₃ = 130; Pu = 0.07; NH ₂ SO ₃ H = 7.5	435*	420
HAIS		0 *	140
HAS	HNO ₃ = 194; NH ₂ SO ₃ H = 5.5	290*	140
HAX	30% TBP - 70% Shell E-2342	1540	1540
HGX	HNO ₃ = 0.63	2870*	2800
IAF	U = 330; HNO ₃ = 125; Pu = 0.077; NH ₂ SO ₃ H = 7.5	440	420
IAS	HNO ₃ = 194; NH ₂ SO ₃ H = 5.5	280*	280
IAIS		0	0
IAX	30% TBP - 70% Shell E-2342	1580	1540
IBS	30% TBP - 70% Shell E-2342	240	250
IBX	HNO ₃ = 6.3; Fe ⁺⁺ = 1.7	140±20*	126
IBXF	U = 76; HNO ₃ = 13; Pu = 0.018	1700	1920
ICX	HNO ₃ = 0.63	2900*	2800
2AF	HNO ₃ = 330; Pu = 0.06; NaNO ₂ = 5.8	168	172
2AS (2)	HNO ₃ = 30	23 *	21.6
2AX	30% TBP - 70% Shell E-2342	44	43
2BX	HNO ₃ = 0.63	23 *	21.6
IOF	30% TBP - 70% Shell E-2342	2400-3000	2420
IOR	Na ₂ CO ₃ = 26	ca. 1050	700
IOS	Na ₂ CO ₃ = 26	1300 *	110

* Calculated from tank depletion rates; others are by rotameter readings.

- (1) 2A, 2B, and IO Columns operated at 10 T/D plant equivalent rates. All other columns operated at 14 T/D.
- (2) 2A Column operated with 2AIS (H₂O) during last two portions of run. 2AIS rate was 15 ml/min for second portion and 21 ml/min for third portion of 2A-2B run.

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4.0 FEED PREPARATION

4.1 Slug Charging

For Run PX-13, 985 pounds of uranium metal (246 MWD/T cooled 133 days at the time of dissolving) were added to an empty dissolver.

4.2 Jacket Removal

The slug jackets were removed by the standard procedure of boiling for three hours in a solution initially containing 600 pounds of 26 weight per cent NaNO_3 and 164 pounds of 50 weight per cent NaOH . Operation was normal.

4.3 Dissolution

Data obtained during the dissolution are reported in Table 4.1. A total of 875 pounds of uranium was dissolved in four downdraft cuts with an average acid consumption of approximately 2.75 mols of HNO_3 per mol of uranium dissolved. Operation was normal.

4.4 Centrifugation and Feed Adjustment

Due to seal water dilution, cuts 1 and 2 were concentrated prior to centrifugation. The first three cuts were combined after centrifugation and adjusted to HAF specifications by addition of nitric acid, sulfamic acid, and water. No NaNO_2 addition was made. The feed was digested for two hours at 70°C . The fourth dissolver cut was not centrifuged due to mechanical failure of the centrifuge; otherwise feed preparation was the same as Cuts 1 and 2.

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TABLE 4.1
DISSOLUTION DATA

	Cut Number			
	1	2	3	4
Source of acid	Fresh	Fresh	Fresh	Fresh
Initial acid concentration, % (1)	48	48	48	48
Weight of 100% HNO ₃ , Lb.	237	237	237	158
Uranium charged, Lb.	985			
Reflux water addition, Lb.	50	50	50	33
Reflux addition duration, Hr.	1	1	1	0.5
Air addition rate, SCFM	4.9	4.5	4.9	4.7
Time at boiling, Hr.	5.5	6.0	8.5	9.0
Final specific gravity (boiling)	1.71	1.71	1.71	1.76
Final vapor temperature, °C.	--	--	--	--
Uranium dissolved, Lb.	454		231	191
Dissolver heel, Lb.	531		300	109
Acid consumed (2)	2.85		2.80	2.50
Dissolver solution, U M	2.6		2.3	2.8
Dissolver solution, HNO ₃ M	2.8		2.5	1.7

- (1) Includes reflux water addition.
- (2) Moles of HNO₃ per mole of uranium dissolved.

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5.0 SOLVENT EXTRACTION

5.1 Operation

Prior to the start of Run PX-13 the solvent remaining in the columns was displaced and the solvent in the various solvent tanks was removed from the system. All columns and tanks were then emptied and the entire system was flushed with 5-10% HNO_3 to remove all high MWD/T material remaining from Run PX-10. Prior to column startup, all columns were filled to the normal interface level with cold aqueous streams. The startup procedure entailed simultaneous start of all cold and hot streams.

HA Column operation was divided into three main portions, each using a different type of HAX organic treatment, as indicated on Figure 5.1. The first 15 hours of operation utilized fresh solvent as HAX; the next 20 hours, CP Na_2CO_3 washed IOF and the last seven hours, Technical Grade Na_2CO_3 washed IOF. In addition to these solvent changes, several frequency changes were made. Due to the mechanical failure of the feed centrifuge, the last 12 hours of HAF was uncentrifuged. All processing in the HA Column was done at 14 T/D (Plant equivalent) at 1.07 in. extraction section amplitude. The HA-HC Column operation was stable for the entire run.

The IA Column was operated at 14 T/D plant equivalent rates and a number of different frequencies as well as two different amplitudes in an attempt to achieve the optimum pulsing conditions. For the first 34 hours of operation, the extraction section amplitude was 1.2-inch. This was decreased to 1.07-inch amplitude for the last 6 hours of operation. The principal operating condition changes are indicated on Figure 5.2. The column flooded at a frequency of 68 and extraction section amplitude of 1.2-inch approximately six hours after column startup. The flooding was cyclic in nature and persisted for four hours but was alleviated by increasing the interface control sensitivity. The column flooded again several hours after the frequency was increased from 68 to 72. Successive reductions in frequency from 72 to 66 to 62 produced stable operation. The column flooded for the third time immediately after the amplitude was decreased to 1.07-inch (extraction section) and the frequency increased to 80. The unstable condition continued for the remainder of the run despite frequency decreases to 66 cycles/min.

In addition to the above changes in operating conditions, two differently treated solvents were used as IAX. For the first 30 hours of operation, CP Na_2CO_3 washed IOF was used and for the last eleven hours a change was made to Technical Grade Na_2CO_3 washed IOF.

The IBX, IBS, and IC Columns were operated at 14 T/D plant equivalent rates for the entire run. However, since the 2A and 2B Columns were operated at 10 T/D plant equivalent rates, the IBX, IBS, and IC Columns were shut down several

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times due to a shortage of volume in the IEP (2AF) tank. The IEX and IO Columns were operated without difficulty except for one to two hours of unstable operation each time these columns were started. The IES Column flooded the first few hours after each column startup. Generally the IES frequency was reduced to 30-32 cycles per minute to break the flood. The IES Column was normally operated at a frequency of 50 except for the periods of lower frequency required to break the floods. See Figure 5.3.

The 2A-2B Column operation was divided into three portions as indicated in Figure 5.4. For the first 28 hours, the 2A-2B Columns were operated at 10 T/D plant equivalent processing rates using Purex Flowsheet - HW #3 conditions. During the next 20 hours of operation, 15 ml./min. of 2AIS (2AIS = H₂O - 2AF:2AS:2AIS = 100:12.6:8.7) was added as an intermediate scrub stream in addition to normal Purex Flowsheet rates. For the last 17 hours of the run, the 2AIS flow was increased to 21 ml./min. (2AIS = H₂O - 2AF:2AS:2AIS = 100:12.6:12.6). The 2A Column was operated without difficulty throughout the entire run. Due to a partially plugged 2BP valve and/or rotameter, the 2B Column interface position was maintained by manual adjustment of the 2BP rate to flowsheet conditions.

5.2 Data

With the exception of the polarograph and the IOF gamma monitor, all of the in-line instruments were in working order during the entire run. The principal data are plotted in Figures 5.2 to 5.4. Tables 5.1 to 5.3 summarize the laboratory analyses.

5.3 Discussion

HAP in-line gamma monitor results agreed quite well with laboratory results. The HAP uranium photometer results, however, were as much as 10 g./l. higher than the laboratory results, but part of this difference may be attributable to instrument standardization problems. After restandardization, the difference between laboratory and in-line results decreased to 2-5 grams/liter.

The HAF/HAP in-line decontamination factors averaged 2000 during the fresh solvent portion of the run but decreased by a factor of 2-3 when the HAX was switched to CP Na₂CO₃ washed IOF. For the remainder of the run, the in-line decontamination factors remained in the range of 440-850 with an average value of approximately 600. Little or no difference in HA Column decontamination can be attributed to use of uncentrifuged HAF. In a similar manner, switching from CP to Technical grade Na₂CO₃ in the IO Column produced no significant change in the HAF gamma activity. As indicated in Figure 5.1, essentially no DF was obtained in the HC Column throughout the entire run. The HAF gamma monitor cell was changed twice during the run because of a gradual buildup of background activity on the cell walls. For the six hour period between 0300 and 0900 on 1/19/56, the HAF gamma monitor full and empty cell readings were essentially identical; consequently no DF values were plotted for this period.

The HAW uranium and plutonium losses ranged from a flowsheet value of $< 0.2\%$ to 10-20 times this value. No explanation can be given for these variations. No significant plutonium loss can be attributed to the omission of sodium nitrite from the HAF and the addition of sulfamic acid to the HAF and EAS, since in general the uranium and plutonium losses were of the same order of magnitude.

Because the IAF in-line gamma monitor results were consistently lower than the IAF laboratory gamma results, both the in-line and the laboratory decontamination factors relative to IAF were based on the average IAF laboratory total gamma activity (2.91×10^4 $\mu\text{c/l.}$). Using this basis, the IAF/IAF in-line and laboratory decontamination factors check quite well. The IAF uranium photometer results were 5-10 g./l. lower than the laboratory results until the photometer was standardized. After standardization, the photometer and laboratory results agreed within 1 g./l. for the remainder of the run. In spite of a number of frequency changes, the IAF/IAF in-line DF remained in the range of 50-140 during periods of steady operation with an average DF of approximately 60. The IAF/IAF DF was essentially the same for both CP and Technical grade Na_2CO_3 washed recycled solvent. Because the IA Column flooded during the entire 1.07-in. amplitude period, the effect of amplitude change on IAF/IAF DF cannot be determined. The IAW Pu losses were 7-30 times the Purex Flowsheet - HW #3 values of $< 0.2\%$, whereas the IAW U losses ranged from 1/100 to 1/300 of the flowsheet value of $< 0.2\%$ during the periods of steady column operation. Since the IAW was not analyzed for SO_4^{2-} or Pu^{+3} , these high IAW Pu losses may possibly be attributed to any or all of the following factors:

- a) NaNO_2 omission from the IAF.
- b) Addition of sulfamic acid to the IAF and IAS.
- c) Excessive storage time of IAF prior to processing (IAF was stored up to 100 hours).
- d) Reduction in Pu E_a^0 due to sulfate formation.

Despite the fact that both in-line and laboratory IAF/IBU decontamination factors were based on the average laboratory IAF analysis, laboratory factors ranged up to twice the in-line values. Part of this discrepancy may possibly be attributed to the small difference between full and empty IBU gamma monitor cell readings. Laboratory IBXF, IBU, and ICU total gamma analysis indicate uranium stream decontamination factors of 4-10 across the IB Column with a corresponding value of approximately 1.5 to 3.0 across the IC Column. The overall HAF/ICU laboratory DF was $5-6 \times 10^5$ ($\text{DF} = 5.7-5.8$). The ICW total gamma activity varied between 12 and 18 $\mu\text{c/l.}$ If it is assumed that this ICW activity is a fixed solvent activity, the decontamination factors would be affected in the following manner:

- a) IAF/IAF DF would be approximately 10-30% higher.
- b) IBXF/IBU DF would be higher by a factor of approximately 2-3.
- c) IBU/ICU DF would be essentially 1.0.

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The IBU plutonium losses ranged from the flowsheet value of 0.2% to 2 times flowsheet value. The IBP U losses, however, were as much as 300 times the flowsheet value of $1 \times 10^{-5}\%$. The ICW uranium losses were approximately 10 times the flowsheet value of $< 0.2\%$ throughout the run.

When the IAF laboratory gamma analysis of 2.91×10^4 $\mu\text{c}/\text{l.}$ was used as a basis, both the in-line and laboratory IAF/2AF decontamination factors agreed within 30%. However, the in-line 2AF/2AP decontamination factors were as much as twice the corresponding laboratory values. The 2AP/2BP laboratory decontamination factors varied from 7 to 38, however, for decontamination factors above 10, the 2BP total gamma analyses were at the lower limit of laboratory detection. Consequently, total gamma variations of a factor of two may be attributed to unreliability of laboratory analyses at these low activity levels. The high 2AP/2BP decontamination factors may be due in part to lack of correction for fixed activity in the solvent. Correction for fixed activity was not made, however, since the 2BW stream was not sampled. The general effect of this fixed activity correction would be an increased decontamination factor in the 2A Column with a corresponding decreased decontamination factor in the 2B Column. Overall HAF/2BP laboratory decontamination factors ranged from 2.1×10^7 to 2.2×10^8 ($\bar{d} = 7.3 - 8.3$). Due to initial 2B Column plutonium buildup and refluxing of plutonium in the 2A Column, the 2BP plutonium analyses were considerably lower than flowsheet values. With the exception of the first two 2BP samples (3 and 11 hours after column startup), the 2BP gamma/AT ratio remained in the range of $1 - 2 \times 10^{-10}$ $\mu\text{c}/(\text{c}/\text{m})$ with an average value of 1.2×10^{-10} $\mu\text{c}/(\text{c}/\text{m})$.

The 2A Column operation was divided into three portions in order to study the effect of an intermediate water scrub stream on UX_1 decontamination. Table 5.3 lists the 2AP and 2AW UX_1 concentrations. Since the 2AF UX_1 activity (calculated from material balance, 2AW, and 2AP UX_1 activities) varied by as much as a factor of ten during the run, the data obtained do not warrant any definite conclusions. Some of the discrepancy in 2AF UX_1 concentration may possibly be attributed to IBS Column flooding. In addition to this, the 2AP UX_1 concentrations were generally at the lower limit of laboratory detection. The intermediate-scrub 2A flowsheet gave excessive 2AW plutonium losses as indicated in Figure 5.4. The 2AW plutonium losses increased from 0.2-1.2% for zero 2AIS flow, to 2.2-11.0% for 15 ml./min. 2AIS flow, to 12.6-23.0% for 21 ml./min. 2AIS flow. These high product losses were aggravated by the short (8 ft.) Hot Semiworks 2A Column extraction section. A considerable increase in 2A Column plutonium reflux occurred during the latter two portions of the run as indicated by decreasing 2AP plutonium concentrations. The 2AW plutonium losses were based on 2AF plutonium concentrations. These same losses based on HAF plutonium concentrations would be lower by a factor of two due to the low 2AF plutonium concentration.

TABLE 5.1
PRECYCLE LABORATORY RESULTS(1)

	Fresh HAX(2) HA f = 70	0.25 M CP Na ₂ CO ₃ Contact in IO Column			0.25M Technical Na ₂ CO ₃ Contact in IO Column HA f = 76
		HA f = 60	HA f = 70	HA f = 76	
<u>HAP:</u>					
U-g/l	86	92	89	89	90
Pu-g/l	0.020	0.021	0.019	0.020	0.016
HNO ₃ -g/l	9.9	8.5	10.7	9.3	9.8
Total γ-μc/l	2.1 x 10 ³	7.9 x 10 ³	7.3 x 10 ³	9.1 x 10 ³	1.0 x 10 ⁴
% ZrNb	60	90	80	85	85
% Ru	40	10	20	15	15
<u>HAW:</u>					
U-g/l	1.3	1.5	0.3	9.9	0.7
Pu-g/l	1 x 10 ⁻⁴	(3)	4 x 10 ⁻⁴	2 x 10 ⁻³	1 x 10 ⁻³
HNO ₃ -g/l	145	(3)	153	150	150
<u>HCP:</u>					
U-g/l	49	49	(3)	48	54
Pu-g/l	0.013	0.013	(3)	(3)	0.010
HNO ₃ -g/l	3.6	2.4	(3)	5.3	4.8
Total γ-μc/l	1.19 x 10 ³	5.0 x 10 ³	(3)	4.9 x 10 ³	6.1 x 10 ³
<u>HCW:</u>					
U-g/l	2.4 x 10 ⁻³	1.2 x 10 ⁻²	(3)	(3)	7.4 x 10 ⁻³
Pu-g/l	2.8 x 10 ⁻⁶	8.3 x 10 ⁻⁷	(3)	1.3 x 10 ⁻⁶	1.1 x 10 ⁻⁶
Total γ - μc/l	1.3 x 10 ²	1.5 x 10 ²	(3)	1.4 x 10 ²	2.0 x 10 ²

Notes:

- (1) HAF composition: U = 332 - 342 g/l; Pu = 0.073 g/l; HNO₃ = 130 g/l; Total γ = 2.6 x 10⁷ μc/l.
 (2) Batch washed with 0.25 M CP Na₂CO₃.
 (3) No sample.

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TABLE 5.2

PARTITION CYCLE LABORATORY RESULTS(1)

	0.25 M CP Na ₂ CO ₃ Contact in 10 Column a = 1.2 in. f = 60-72 f = 62-66		0.25 M Technical Na ₂ CO ₃ Contact in 10 Column a = 1.2 in. a = 1.07 in. f = 66 f = 70-80		
<u>IAP:</u>	U - g/l	86	87	87	89
	Pu - g/l	0.017	0.018	0.020	0.01
	HNO ₃ - g/l	9.5	10.0	10.0	11.0
	Total γ - μc/l	74	130	103	51
	% Zr-Nb	80	90	85	85
	% Ru	20	10	15	15
<u>IAW:</u>	U - g/l	0.003	0.004	0.001	0.1
	Pu - g/l	0.001	0.001	0.003	0.005
	HNO ₃ - g/l	116	146	152	163
<u>IBXF:</u>	U - g/l	75	77	(2)	(2)
	Pu - g/l	0.018	0.019	(2)	(2)
	HNO ₃ - g/l	15	12	(2)	(2)
	Total γ - μc/l	127	206	(2)	(2)
<u>IBU:</u>	U - g/l	76.1	--	(2)	63
	Pu - g/l	4 x 10 ⁻⁵	6 x 10 ⁻⁵	(2)	7 x 10 ⁻⁵
	Total γ - μc/l	28	17	(2)	34
	% Zr-Nb	65	65	(2)	55
	% Ru	35	35	(2)	45
<u>IBP:</u>	U - g/l	0.02	0.008	(2)	0.033
	Pu - g/l	0.16	0.19	(2)	0.16
	HNO ₃ - g/l	102	95	(2)	101
	Total γ - μc/l	1210	420	(2)	870
<u>ICU:</u>	U - g/l	47	48	(2)	(2)
	HNO ₃ - g/l	7	--	(2)	(2)
	Total γ - μc/l	7	8	(2)	(2)
<u>ICW:</u>	U - g/l	1.2	2.0	(2)	0.9
	Total γ - μc/l	18	12	(2)	13

Notes:

(1) IAF Composition: U = 330 g/l; Pu = 0.077 g/l; HNO₃ = 125 g/l;
Total γ = 2.9 x 10⁴ μc/l; Zr-Nb = 85-90%; Ru = 10-15%.

(2) No Sample.

TABLE 5.3FINAL PLUTONIUM CYCLE LABORATORY RESULTS(1)

	<u>0 ml/min 2AIS(2)</u>	<u>15 ml/min 2AIS(2)</u>	<u>21 ml/min 2AIS(2)</u>
<u>2AP:</u> Pu - g/l	0.18	0.043	0.16
HNO ₃ - g/l	7.0	4.4	7.0
Total γ - μ c/l	23	19	17
% Zr-Nb	0	67	0
% Ru	>95	33	>95
UX ₁ - c/m/ml	30-70	10-20	10-40
<u>2AW:</u> Pu - g/l	$(1.2-4.9) \times 10^{-4}$	$(1.2-5.9) \times 10^{-3}$	$(7.1-13.0) \times 10^{-3}$
HNO ₃ - g/l	310	270	266
UX ₁ - c/m/ml	10-120	10-40	30-100
<u>2BP:</u> Pu - g/l	0.04 - 0.4	0.14 - 0.33	0.28
Total γ - μ c/l	1.6 - 17	2.2	2.1

Notes:

- (1) 2AF Composition: Pu = 0.06 g/l; U = 0.03 g/l; HNO₃ = 330 g/l;
Total γ = 480 μ c/l; ZrNb = 95%; Ru = 5%. Calculated UX₁ = 15-150 c/m/ml.
- (2) Purex Flowsheet - HW #3 10 T/D plant equivalent 2AS flow = 21.6 ml/min.

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FIGURE 5.1

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FIGURE 5.4

Note: Both the In-Line and Lab. 1AF/2AF DF's are based on an average Lab. 1AF Gamma analysis of 2.91×10^4 $\mu\text{C/L}$.

FINAL PLUTONIUM CYCLE DATA

	In-Line	Lab
1AF/2AF DF	□	■
2AF/2AP DF	○	●
2AF/2BP DF		▲

DF BASIS: $1AF \gamma = 2.91 \times 10^4$ $\mu\text{C/L}$

$$\frac{HAF}{1AF} \text{ DF} = 870$$

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6.0 SOLVENT RECOVERY

At the completion of PX-12 all the solvent was jettied out of the system and the solvent tanks and IO Column were given two 5-10% HNO_3 flushes. PX-13 was started with fresh solvent which had been batch washed with 0.25 M CP Na_2CO_3 . The Shell E-2342 diluent for this solvent was obtained from the Furex Plant. Fresh solvent was used for the first sixteen hours of HA-EC operation. The HCW from this portion of the run was processed through the IO Column at 10 T/D rates using 0.25 M CP Na_2CO_3 as scrub. The IO Column was operated on an intermittent basis during the entire run at 10 T/D rates (plant equivalent). The HAX used for the last 7 hours of HA-EC operation was 0.25 M Technical Na_2CO_3 washed IOF. The solvent used for the first 30 hours of IA Column operation was CP Na_2CO_3 washed IOF. The solvent used for the last 12 hours of IA Column operation was 0.25 M Technical Grade Na_2CO_3 washed IOF. The HCW varied in activity from 130 $\mu\text{c}/\text{l}$ to 200 $\mu\text{c}/\text{l}$ with 0.004-0.02% Pu losses and 0.003-0.01% U losses. The ICW activity varied from 13 $\mu\text{c}/\text{l}$ to 18 $\mu\text{c}/\text{l}$ with 1-3% U losses. The IO Column was operated at the following conditions:

a = 0.5-inch
f = 70
IOF:IOS:IOR = 100:56:44
VV = 600 gal/hr/ft²

The IOF in-line gamma monitor was inoperative during the entire run. IOO-L in-line gamma readings varied from 120 $\mu\text{c}/\text{l}$ to 180 $\mu\text{c}/\text{l}$. However, these in-line gamma monitor results are of doubtful value since the ICW lab gamma analysis were consistently lower than the IOO-L in-line gamma readings by a factor of 10.

7.0 ACID RECOVERY

Both of the Waste Concentrators were started with recovered acid from FX-12 in the pots. The No. 1 Waste Concentrator vapor line plugged early in the run, thus necessitating shutdown of this concentrator. For the remainder of the run the HAW was collected in the No. 1 Waste Concentrator and batch jettied to the service concentrator. For this reason and because of emphasis on other portions of the process, no major effort was made to accumulate acid recovery data during PX-13.

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